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## INDEX OF VOL. XIII.—1894.

### INDEX OF AUTHORS' NAMES.

N.B.—In this Index, (P) indicates that the matter referred to is an abstract of a Patent.

		PAGE
<b>A</b>		
Abbott, W., and Wilks, W. R. Rubber-coated Waterproof Fabrics (P).....		248
Abney, W. de W. See Hummel.....		803
Abou, J. W. Bleaching Paper Pulp, &c. (P).....		834
Abraham, K. Use of Sulphurous Acid in Sugar Manufacture.....		165
Abraham, E. See Fenn.....		896
Acheson, E. G. Carbons for Electric Lamps, &c. (P).....		1207
Ackerman, P. The Substantive Dyestuffs in the Dyeing of Half-Wool Goods.....		386
Aetion Gesellschaft für Anilin Fabrikation, Manufacture of 2:7-Amido-naphtholdisulpho Acid and Colouring Matters therefrom (P).....		876
Manufacture of a New Amidonaphtholdisulphonic Acid (P).....		32
Manufacture of Basic Colouring Matters (P).....		511
Manufacture of Colouring Matters (P).....	246, 724,	1189
Manufacture of Mordant-dyeing Colouring Matters (P).....		723
Manufacture of New Bases and their Sulpho Acids (P).....		1055
Photographic Developers (P).....		459
Aetion Maschinenbauanstalt vorm. Venueth and Ellenberger. Process and Apparatus for Drying and Treating Distillers' Washes (P).....		827
Adam, P. Sodium Boro-Salicylate.....		834
The Emetics.....		1216
Addie, J., and others. Production of Cyanides (P).....		635
Adeney, W. E., and Parry W. K. Purification of Sewage and other Liquids (P).....		831
Adkins, J. J. Treating Brewers' Spent Hops for Production of Fodder and Litter (P).....		168
Aguillon, M. Heat of Combustion of Coal-Gas, and its Relations to Lighting Power.....		26
Aitken, A. P. Rapid Method of Determining Moisture and Oil in Linseed Cakes, &c. (illus.).....		469
Aitken, H. Apparatus for obtaining Cyanogen, Cyanides, &c. (P) (illus.).....		517
Albright, G. S., and Hood, J. J. Manufacture of Manganese Peroxide (P).....		517
Alexander, H. Apparatus for Electro-Deposition of Metals (P).....		819
Allein and Gaud. Estimating Cupric Reducing Power.....		1229
Allen, A. H. Discussion on Commercial Analysis of Lard.....		708
Discussion on Estimation of Tanning Matter.....		495
Allen, C., and Davy, C. Apparatus for Manufacture of Cast Iron and Steel (P).....		815
Allen, C. M. Processes and Converters for Smelting and Refining Ores.....		1203
Allen, S. Filtering Apparatus (P).....		378
Allen, W. Apparatus for Heating by Creosote, &c. (P) (illus.).....		1050
Alliott, J. B., and Paton, J. McC. C. Furnaces for Burning Refuse (P) (illus.).....		898
Allison, T. R. Extract of Cocoa (P).....		56
Allison, W. O. See Pennington.....		725
Altschul, M., and Meyer, V. The Chlorination of Ethyl Alcohol.....		415
Amend, O. P. Improvements in Dyeing (P).....		151
Ammann, K. Washing Blue (P).....		1209
Anderlik. Mucilaginous Beet Juice.....		749
Anderson, G. R., and others. Artificial Asphaltum or Pitch, and Process of Making Same (P).....		740
Anderson, J., jun. Apparatus for Separating Foreign Matter from Waste Rubber, Bark, Grain, &c. (P).....		974
Andersson, H. K., and Sjöström, D. Disinfecting Apparatus (P).....		900
Andiran, A. J. J. d'. See Durand.....	145, 146, 247, 802,	1055
Andreoli, E. Electrical Production of Ozone and Luminosity (P).....		162
Electrolytic Apparatus (P).....		190
Andresen, M. Development of Photographs (P).....		1249
Andrews, T. Effect of Stress on Corrosion of Metals.....		1266
Angele, W. Manufacture of Soluble Starch (P).....		765
Angell, A. Manufacture of Oil Paints and Compositions (P).....		803
Anning, J. J. Iodine Liniment (P).....		836
Antal, J. Cobalt Nitrate an Antidote for Cyanide Poisoning.....		1106
Antonchevitch, A. See Sahanejeff.....		1676
Appert, L. Engelmann's Process for Decoration of Glass.....		1197
Archbutt, L., and Deeley, R. M. Purification of Sewage (P).....		899
Armstrong, M. M. Method and Furnaces for Smokeless Combustion (P) (illus.).....		761
Arnaudon, J. J. Vegetable Silks, particularly the Kapok of India.....		247
Arnold, J. O. The Physical Influence of Elements on Iron.....		642
And Read, A. A. Conditions in which Carbon exists in Steel.....		740
Arnold, W. Apparatus for Washing Filtering Media (P) (illus.).....		16
Arquembourg, H. See Grouvelle.....		950
Arth, G. Experiments on Coals immersed in Water.....		1352
Artiques. A New Photographic Printing Paper.....		720
Artus, C. Treatment and Preparation of Alimentary Fats and Compounds (P).....		969
Aseough, J. Manufacture of Sodium Bifluoride (P).....		1297
Ash, C. A. Apparatus to Enable a Person to Breathe Fresh Air while Surrounded by a Noxious Atmosphere (P).....		733
Ash, T. M., and others. Coating Non-metallic Articles with Metal (P).....		501
Ashcroft, E. A. Process and Apparatus for Treating Refractory Ores (P).....		57
Ashworth, A., and Burger, J. Production of Colouring Matters (P).....	691, 723,	
Aspinall, F. B., and others. Bleaching, Sweetening, &c. Oils and Fats (P).....		1298
Atlass, E. See Lehl.....		51
Aubry, L. Composition of Hops, and their Influence on Wort.....		177
Composition of Hops, and Influence of Hops on Wort.....		411
The Composition of Hops, and their Relation to Wort.....		895
Auer, H. See Hurler.....		978
Autenrieth, W. Yellow Prussiate of Potash, and Detection of Hydrocyanic Acid.....		843
Axedorfer, G., and Sasse, C. F. H. Apparatus for Mixing Gas and Air (P).....		1620

	PAGE		PAGE
Barnheim, L., and Jernander, A. Electric Lignating Compound (P).....	561	Bartlett, G. See Peptine Maltine, Lim. ....	1083
Bach, A. Action of Light on Phenol.....	988	Baschmakoff, S. de. See Nalcin.....	58
Ba h. O. Valuation of Zinc Dust.....	765	Basle Chemical Works Rindschidler. Manufacture of a New Dioxynaphthalene-mono-sulpho Acid and Colouring Matters therefrom (P).....	632
Bachofen, F. See Lunge.....	36	Manufacture of a New Dioxynaphthoic-mono-sulpho Acid, and Colouring Matters therefrom (P).....	632
Baltische Anilin und Soda Fabrik. Manufacture of Alpha-beta-methyl-eurhodines (P).....	879	Bates, F. G. Baths for Treating Iron or Steel (P).....	645
Manufacture of Aurumine (P).....	217	Bateson, P. Discussion on Manufacture of Straw Cellulose.....	197
Manufacture of Basic Colouring Matters (P).....	723	Battut, Control of Beetroot Sugar Manufacture.....	759
Manufacture of Beta-methyl-eurhodine (P).....	878	Bau, A. Estimation of Succinic Acid formed in Fermentations.....	287
Manufacture of Colouring Matters (P).....	1189	Baum, H. Manufacture of Orthohalogen-phenol, and Pyrocatechine therefrom (P).....	658
Manufacture of Colouring Matters and Intermediate Products (P).....	1191	Baum, J. H. Production of Pyrocatechine (P).....	579
Manufacture of Dihydroxy-naphthalene-sulpho Acids and Corresponding Azo Dyes (P).....	942	Baumann, T. Report on Petzold's Paper on "A New Direct Grey".....	143
Manufacture of Mordant-dyeing Colouring Matters (P).....	943	Baumbach, O. A Disinfecting Oven (P).....	413
Manufacture of New Colouring Matters from Naphthalene (P).....	650	Baumert, P., and Peck, A. Artificial Stone and Marble (P).....	413
Manufacture of New Dyes of the Phenanthrene Series (P).....	942	Baur, A. Bye-Products of the Preparation of Butyltoluene.....	1213
Manufacture of New Sulpho-Acids of Phenylresinduline, &c. (P).....	510	Production of Artificial Musk (P).....	903, 903
Production of a New Amido-Sulpho-Acid and Azo Dyes therefrom (P).....	724	Bayley, T. Treating Grain, &c. for Brewing (P).....	1082
Production of New Acid Colouring Matters (P).....	145, 801	Bayrac, P. Volumetric Estimation of Lead in Tin Solutions or Platings.....	429
Production of New Azo Dyes (P).....	1054	Beacham, F. C. D. Colours for, and Colouring, Photographs, &c. (P).....	759
Production of New Azo-Dyes, and Component therefor (P).....	144	Beadle, C. Commercial Cellulose Derivatives.....	960
Production of New Colouring Matters and Leuco Compounds (P).....	802	Marking Indelibly on Paper and Fabrics (P).....	1194
Production of New Compounds for Preparation of Diazo Compounds (P).....	1189	Beardmore, W. See Fairfax.....	1067
Production of New Diazo Dyes (P).....	879	Beaudet, De-fecation and Saturation.....	965
Production of New Sulpho-Acids, and New Colouring Matters therefrom (P).....	144	Beaulis, A. Report on the Mexican Leather Industry.....	992
Baerlein, M. Manufacture of Soap Compositions (P).....	962	Beck, P. Carbonic Acid Pressure Apparatus (P).....	783
And Dreyfus, C. Cleansing Mixture or Paste (P).....	939	See Freund.....	339, 540
Bacard, H. Electrical Transport of Heat in Electrolytes (illus.).....	71	Becker, American and Russian Petroleum.....	872
Bady, F. G., and Guthrie, M. Apparatus for Electrolytical Decomposition of Brine and other Liquids (P).....	818	Becker, A. See Sachse.....	409
Baines, F. E. V. Treatment of Rice and other Grain for Brewing (P).....	268	Becker, W. Process or making Leather Soft, Durable, and Waterproof (P).....	164
Baird, M. B. See Murray.....	1054	Bedford, J. E., and C. S. Manufacture of Oil Varnish and Drying Medium for Paints (P).....	863
Baker, H. Breton. The Influence of Moisture on Chemical Action.....	767	Treatment of Linseed Oil (P).....	491
Baker, R. C. Apparatus for Cooling or Warming Liquids (P).....	713	Bedson, P. P. Discussion on Lunge-Rohrman Plate Towers. Note on Poisonous Action of Lead Salts.....	1037 610
Baker, T. W. Destroying Noxious Gases from Destructors (P).....	1084	Presentation to Dr. J. T. Dunn.....	1155
Bale, F. Production of Chlorine by Ammonia-Soda Process (illus.).....	200	Behal, A., and Choay, E. Composition of Beech and Oak Creosotes.....	1157
Bale, J. H. Wafer China Mosaic (P).....	519	Qualitative Composition of Official Beech and Oakwood Creosotes.....	1087
Balke, P. On the Xanthine Bases.....	284	The Ethyl Phenols.....	794
Balland, M. On the Internal Temperature of Bread as it Comes from the Oven.....	55	Bejerinck, Butylic Alcohol Fermentation, and the Butyl Ferment.....	969
Balland and Musson. Sterilisation of Bread and Biscuits after Withdrawal from the Oven.....	169	Belby, G. Discussion on Electric Furnace for Laboratory Work.....	375
Ballantyne, H. Studies in Oil Analysis.....	1106	Discussion on Paraffin Lamp for Laboratories.....	1167
Baly, F. C., and Chorley, J. C. Thermometer for High Temperatures.....	418	Discussion on Production of Oil-Gas.....	501
Bandler, H. K. The Analysis of Steel.....	665	Belby, G. T. Proceedings of Annual General Meeting.....	693, 694
Bamberger, E., and Chittaway, F. Chrysen.....	27	Bell, A. H., and Carter, C. Apparatus for Precipitating Gold and Silver from Cyanide Solutions (P).....	816
Bamberger, M. On Xanthorrhiza Resins.....	47	Bell, J. Carter. Acknowledgment of Presentation by Manchester Section.....	608
Barber, C. M. Electro-Plating and Apparatus therefor (P).....	959	Discussion on Modern Systems of Sewage Treatment.....	609
Barber, C. See Monnet.....	658	Discussion on Sampling of Materials.....	217
Barber, C. J. Electrical Accumulator Plate (P).....	1206	Note on Estimation of Moisture in Wood Pulp.....	117
Barber, A. C. Preparation of Active Material for Accumulators (P).....	1206	Notes on the "Fertilisers and Feeding Stuffs Act".....	116
Barber, P. Geraniol.....	60	Bell, Sir L. Evaporating Saline Solutions (P).....	731
Rhodinol. The Chemical Principle of Otto's Res. ....	171	Treatment of Bicarbonate of Soda (P).....	735
And Bouveault, L. A Natural Unsaturated Ketone in Lemon-Grass Oil.....	1086	Use of Caustic Lime in the Blast Furnace.....	951
And Bouveault, L. Geraniol from Oil of Andropogon Schoenanthus.....	1086	Belloe, D., and Benard, E. Process and Apparatus for Disintegrating Cements (P).....	1198
And Bouveault, L. The Aldehyde of Lemon Grass Oil.....	1086	Belmans, L. Reducing Sulphuretted Ore into Oxides and Sulphur (P).....	153
And Bouveault, L. The Rhodinol of the Essence of Peppermint.....	1085	Benard, E. See Belloe.....	1198
Bardy, Detection of the Higher Alcohols in Spirits of Wine.....	287	Bender, Adolf. Jahres-Rundschau über die Chemische Industrie und deren Wirtschaftliche Verhältnisse für das Jahr 1893, &c.....	671
Barff, F. S., and others. Effecting the Protection of Iron and Steel Surfaces (P).....	41	Benedikt, R. Ekenberg's Process of Refining Fats, Oils, &c.....	891
Barillot, E. A New Reaction of Colehema.....	1099	And Zikes, H. Estimation of Chlorine in Fats.....	954
Barnett, H. T. Carbon Electrodes (P).....	44	Berens, H. See Lach.....	821
Platinised Electrodes (P).....	44	Benker, F. Preparation of Ammonium Nitrate (P).....	515
Barnett, A. A., and Hill, A. J. H. Separating Gelatine Films from Celluloid Supports (P).....	1088	Bennett, R. G. Composition of Varnish (P).....	1209
Bartel, A. See Schroeder.....	649, 1191, 1227	Benneville, J. S. de. Analysis of Pig-Copper, Brasses, and Bronzes.....	667
Bartle, L. Estimation of Salicylic Acid and Salicylates.....	1105	Laboratory Notes.....	667



	PAGE		PAGE
Berthelot, M. Composition of Newly-discovered Ancient Copper.....	1198	Bird, T. Inversion of Sugar by Glycerol.....	1211
Heating and Spontaneous Combustion of Hay.....	1196	Borecki, S., and Kohn's stick, J. Boiler Incrustation Fluid-P.....	711
Observations on Le Blanc's Paper on Minimum E.M.F. required to Decompose Electrolytes.....	742	Borg, C. See Valters.....	78
Bertram, J. Manufacture of Geraniol from Citronellal-ol (P).....	758	Borland, W. Apparatus for Mixing and Regulating Paper Stuff (P).....	754
And Gildenraster, E. Oil of Roses.....	834	Borntreger, A. Behaviour of Lead Acetate towards Sodium Carbonate, Sulphate, and Phosphate.....	1249
And Walbaum, H. Isoborneol.....	756	Influence of Lead Acetate on Cupric Reducing Power of Invert Sugar.....	1241
And Walbaum, H. Occurrence of Camphene in Etheral Oils.....	753	Influence of Volume of Lead Acetate Precipitate on Filtration of Sugar in Wine Analysis.....	1233
Bertrand, C. K., and Renault, B. General Characters of Bog-head Cannel.....	241	Removal of Lead from Musts and Wines.....	1250
Bertrand, E., and Thiel, O. Manufacture of Steel (P).....	846	The Volumetric Determination of Copper.....	67
Bertrand, G. The Latex of the Lacquer Tree.....	1071	Botcher, O. Detection of Adulterants in Basic Slaz.....	978
Besemfelder, E. R. Separating Metals from Ores, &c. (P).....	815	Bouchardat, G. Oil of Spike (Lavandula Spica).....	751
Bettel, Sweep Smelting and Refining Gold, Silver, and Platinum.....	953	Presence of Camphene in Essence of Lavender.....	753
Bevan, E. J. Discussion on Colorimetric Analysis.....	317, 318	Presence of Camphene in Spike Oil.....	658
Note on an Abnormal Melting Point.....	69	And Lafont, J. Synthetic Borneols.....	658
See Cross.....	334	Bouffard, A. On the "Cassage" of Wine.....	654
Beveridge, J. Discussion on Manufacture of Straw Cellulose.....	107	Bourne, See Grimoux.....	1086
The Manufacture of Straw Cellulose.....	101, 213	Bouvenot, L. See Barbier.....	1085, 1086
Beyer, A. F., and A. G. Apparatus for Treating Minerals (P).....	621	Bouvier, Filter for Diffusion Juice.....	167
Beyersack, M. W. Butyl Alcohol Fermentation and its Causes.....	167	Purification of Beetroot Juice.....	1210
Bibby, J. Machinery for Cleaning Cotton Seed (P).....	1070	Bower, G. and A. S. See Barff.....	41
Bibby, J., and J. Manufacture of Printing Ink (P).....	115	Bowing, J. Coking Processes and Apparatus, and Recovery of Products of Distillation (P).....	873
Bidet, A. Some Laboratory Apparatus (illus.).....	631	Boyd, H. A New Explosive (P).....	1219
Biedermann, R. Chemiker-Kalendar, 1895.....	1107	Boyer, L. See Levy.....	1071
Technisch-Chemisches Jahrbuch.....	845	Bracey, B. See Worsley.....	841
Bigelow, W. D. See McElroy.....	284	Bradbury, J. Apparatus and Furnaces for Concentrating Sulphuric Acid (P).....	251
Bizot, C. A Recuperative Gas Furnace (illus.).....	171	Bradley, F. J. See Hardy.....	894
Billings, A. W. Apparatus for Manufacture of Beer (P).....	896	Brain, F. W. T. Electric Fuses (P).....	790
Billings, W. A. See Warden.....	169, 169	Brand, "Maltol," as obtained from Crystallized Malt.....	891
Biltz, F. On Explosives.....	173	Brand, A. Manufacture of Chlorine (P).....	635
Binder, F. Artificial Production of Woollen Craps.....	1192	Brand, H. O. Manufacture of Parchment (P).....	1085
Bindshadler, H. See Read, Holiday and Sons.....	399	Brand, J. Malted.....	1213
Bird, T. See Hargreaves.....	250, 255, 328, 528, 886, 886, 958	Presence of Boric Acid in Beer.....	54
Birnbaum, T. Manufacture of Coloured Waterproof Fabrics (P).....	728	Brandes, W. Rectified Wood Vinegar.....	285
Bischof, G. Treatment of Lead Fumes (P).....	711	Brauns, D. Artificial Refrigeration from Central Stations.....	787
Bittel and Co. Manufacture of Filters (P).....	872	Brante and Co. Condensing Plates (P).....	931
Blackmore, H. S. Dissociating Soluble Salts by Electrolysis (P).....	257	Bredel, F. Water-gas in America.....	715
Blair, T. H. Manufacture of Photographic Films (P).....	659	Brendel, Rate of Filtration of Sugar Solutions.....	749
Blake, A. Improvement in Malting Grain (P).....	268	Brestowski, A. Handwörterbuch der Pharmacie.....	182, 768, 969
Blake, J. W., and S. J. Preservative Paints or Compositions (P).....	618	Brett, R. D. See Hill.....	153
Blake, W. P. Separation of Blende from Pyrites.....	524	Briand, L. Detection of Abstruse in Wines.....	1090
Blass, J. T. Machine for Loosening and Drying Yarns (P).....	917	Briant, L. Aeration: its Effect in Brewing.....	654
Blenkinsop, W. E. R. See Hartley.....	617	And Meacham, C. S. Hops.....	594
Bloch, L., and Schwartz, C. Azo-Colour Resists on Steam-printed Indico.....	1193	And Walker, W. H. Utilisation of Flour Rich in Diastase (P).....	55
Bloede, V. Discussion on Detection of Soap in Lubricants.....	1179	Brier, H. See The Scotch and Irish Oxygen Co.....	789
Blot, G. R. Plates for Secondary Batteries (P).....	819	Briggs, H. B. Manufacture of Condensed Milk Preparations (P).....	828
Blount, B. Discussion on Testing Hydraulic Cements.....	463	Manufacture of Condensed Milk Preparations, Invalids and Children's Foods (P).....	828
See Stancer.....	155	Brin, A. See Juhn.....	55
Bloxam, W. P. Discussion of Smokeless Powder.....	583	Brin's Oxygen Co. and Murray, K. S. Production of Oxygen from Air and Apparatus therefor (P).....	836
Blücher, H. Mantles for Incandescent Gas Lights (P).....	1182	Brintons, Linn., and Hansom, L. Machinery for Washing and Scouring Fibres (P).....	883
Boa, P. Discussion on Electric Furnace for Laboratory Work.....	376	Bristowe, L. H. Filtering Apparatus (P).....	934
Boehring, C. F., and Soehne. Manufacture of Alimentary and Medicinal Compounds (P).....	759	Broadbent, H. Discussion on Estimation of Tanning Matter.....	495
Obtaining an Iron Albumen Derivative from Animal Organs (P).....	658	Broadwell, E. C. Coating Metals with Aluminium or its Alloys (P).....	915
Bogodanoff, P. Temperature of Fusion of Tin.....	1066	Brock, J., and Hurter, F. Manufacture of Permanganates of Soda or Potash (P).....	374
Böhm, E. Manufacture of Enamel Glass Letters (P).....	1198	And others. Manufacture of Sulphocarbides and By-products, and Apparatus therefor (P).....	1195
Böhm, L. K., and Crawford, T. C. Mantles for Incandescent Gas-Burners (P).....	627	Brothers, W. Baking Powder (P).....	752
Boissien, M. P. de. Present State of the Petroleum Industry.....	791	Detergent Compound for Textiles (P).....	264
Boissien, P. de. Improvements in the Sulphuric Acid Industry (illus.).....	1057	Improved Manure or Fertiliser (P).....	160
Bokorny, T. The Physiological Action of Tellurous Acid.....	289	Brougier, A. Improving the Quality of Damaged or other Coffee (P).....	757
Bolton, W. The Weathering Properties of Sandstones.....	40	Brousse and Gay. Mercury Gallate: A New Antisyphilitic.....	171
Bona, M. D. Salicylic Acid Azo Colouring Matters. A Study on the Influence of Constitution on Fastness.....	381	Brown, A. J. Specific Character of the Fermentative Functions of Yeast Cells.....	821
Substantive Azo Colouring Matters from 1,4-Naphthylamine Sulphonic Acid (P).....	945	Brown, Cmm. Proceedings of Annual General Meeting.....	704
Bonato, P. Drinking Water Obtained from Artesian Wells at Mantua.....	752	Speech at Annual Dinner.....	704
Bandonnean, Manufacture of Potato Starch by Continuous-Flow System.....	759	Speech at Perth Banquet.....	707
Bondzynski, S. Estimation of Fat in Cheese.....	766	Brown, H. T. Proceedings of Annual General Meeting.....	709
Bonnet, A. Some Properties of Lead Oxides.....	34	Brown, J. A. See Dunn.....	1053
Bonnet, J. See Placet.....	37, 646	Brown, J. Campbell. Discussion on Aluminium Alloys.....	11
Bonnet, M. New Dyeing Process.....	726	Discussion on Inventions.....	1021
Boutemps, E. Employment of Ammoniacal Salts as Resists.....	1193	Brown, J. H. Treatment of Cuprous Iron Pyrites in Portugal.....	172
Borchers, W. Apparatus for Electrolytic Production of Metals of the Alkalies and Alkaline Earths (P) (illus.).....	159	Brown, W. A. Paste or Size for Cap Bottoms and Cotton Goods (P).....	725

	PAGE		PAGE
B. Low, A. S. Hardy, and J. W. C. ...	894	Carpene, A. Supposed Occurrence of Free Tartaric Acid in Wine. ....	1080
B. W. C. Hardy, S. W. M. ...	1198	Estimating Free Tartaric Acid in Wines. ....	1088
B. J. W. Hardy, and J. W. C. ...	170	Discussion on Rectification of Oil of Vitriol. ....	210
B. J. W. Hardy, and J. W. C. ...	379	Discussion on Sulphuric Acid Pipe Columns. ....	1150
B. J. W. Hardy, and J. W. C. ...	108	Carpenter, F. See Sestegni. ....	906
B. J. W. Hardy, and J. W. C. ...	244	Carr, A. C. See Raby. ....	638, 948
B. J. W. Hardy, and J. W. C. ...	363	Carr, F. H. See Dunstan. ....	172, 172
B. J. W. Hardy, and J. W. C. ...	337	Carr, J. M. See Doulton. ....	948
B. J. W. Hardy, and J. W. C. ...	1098	Carriek, R. See Weinberg. ....	887
B. J. W. Hardy, and J. W. C. ...	1094	Cartier, C. See Bell. ....	816
B. J. W. Hardy, and J. W. C. ...	42	Cartier, J. Making Manure from Sewage and Sludge (P). ....	964
B. J. W. Hardy, and J. W. C. ...	322	Carulla, F. J. R. Discussion on Extinction of Flames by Gaseous Mixtures. ....	1156
B. J. W. Hardy, and J. W. C. ...	249	Discussion on Fermentation in the Leather Industry. ....	221
B. J. W. Hardy, and J. W. C. ...	285	Casasnovas, C. Resists with Tannin Mordants. ....	882
B. J. W. Hardy, and J. W. C. ...	70	Casse, W. F. E. Preserving Milk and Cream, and Treating it for Transit (P). ....	537
B. J. W. Hardy, and J. W. C. ...	527	Cassell Gold Extracting Co. See MacArthur. ....	526
B. J. W. Hardy, and J. W. C. ...	1020	Cassella and Co., Ltd. Manufacture of Disazo Colouring Matters (P). ....	246
B. J. W. Hardy, and J. W. C. ...	1068	Production of Azo Dyestuffs (P). ....	1191
B. J. W. Hardy, and J. W. C. ...	60	Production of Diamido-naphthalene-sulpho Acid and Amido-naphthol-sulpho Acid, and Colouring Matters from the Latter (P). ....	244
B. J. W. Hardy, and J. W. C. ...	1057	Production of Disazo Dyestuffs (P). ....	246
B. J. W. Hardy, and J. W. C. ...	261	Production of Dyestuffs (P). ....	1191
B. J. W. Hardy, and J. W. C. ...	52	Production of New Acetnaphthylene-diamine-sulpho Acids and Colouring Matters therefrom (P). ....	801
B. J. W. Hardy, and J. W. C. ...	359	Production on the Fibre of Fast Dyestuffs (P). ....	390
B. J. W. Hardy, and J. W. C. ...	427	Castenholz, A. Apparatus for Measuring Pressure of Gas in Guns (P). ....	929
B. J. W. Hardy, and J. W. C. ...	933	Castle, P. C. D. See Hanson. ....	410
B. J. W. Hardy, and J. W. C. ...	725	Castner, H. Y. Carbons for Electrodes (P). ....	1067
B. J. W. Hardy, and J. W. C. ...	1087	Electrolytic Apparatus (P). ....	529
B. J. W. Hardy, and J. W. C. ...	681, 723	Caulkins, H. J. See Land. ....	987
B. J. W. Hardy, and J. W. C. ...	832	Causse, H. Basic Bismuth Gallate (Dermatol). ....	171
B. J. W. Hardy, and J. W. C. ...	1090	Cavallo, W. See Hummel. ....	144
B. J. W. Hardy, and J. W. C. ...	1166	Cavazzani, G. Formation of Sugar in the Liver. ....	845
B. J. W. Hardy, and J. W. C. ...	21	Cave, G. G. Treatment of Yeast (P). ....	1214
B. J. W. Hardy, and J. W. C. ...	138	Cazenave, Dyeing with Lead Chromate from the Hygienic Standpoint. ....	970
B. J. W. Hardy, and J. W. C. ...	539	Cazenave, P. Blue Lakes derived from Dibromogallanilide, and some Blue Reactions of Polyphenols. ....	1098
B. J. W. Hardy, and J. W. C. ...	274	Germicidal Action of Gallanilide or Gallanol. ....	755
		Oxidophenolic Colouring Matters. ....	720
		Researches on the Homologues of Gallanilide: Galloparatolide. ....	755
		The Metallic Compounds of Gallanilide. ....	58
		Cerny, The Kilning of Malt in Relation to the Colour of the Malt Germ. ....	267
		Cerny, F. The Kiln-drying of Malt as judged by the Colour of the Combs. ....	895
		Chandelon, T. Preparation of Bicalcic Phosphate Soluble in Ammonium Citrate (P). ....	748
		Chandor, L. Composition for Making Incandescers (P). ....	733
		Chandel, F. Electric Furnaces (P). ....	936
		Production of Substances of Extreme Hardness (P). ....	956
		Chapman, A. Apparatus for Separating and Utilising Steam from Cane Juice (P). ....	1077
		Chapman, A. C., and Rolle, J. R. The Analytical Constants of Seal Oil. ....	843
		Chapman, E. T. See Wanklyn. ....	768
		Charleton, A. G. Nickel: its History, Uses, and Distribution. ....	640
		Charlier, A. C. J. Apparatus for Manufacture of Metallic Pigments (P). ....	407
		Process and Apparatus for Production of Metallic Pigments (P). ....	532
		Charpy, G. Changes Produced in Iron by Permanent Deformation in the Cold (Illus.). ....	40
		On the Allotropic Transformation of Iron under the Influence of Heat. ....	737
		Part Played by Transformations of Iron and of Carbon in Tempering. ....	1066
		Chassy, A. The Electrolysis of Copper Sulphate. ....	1204
		Chatfield, R. E. Utilising Acid Sulphates of Soda (P). ....	735
		Chattaway, F. See Bamberg. ....	27, 27
		Chassevant, A., and Richet, C. Influence of Mineral Antiseptics on Lactic Fermentation. ....	269
		Chem. Fab. Actien E. Schering. Crystalline Polymeric Base from Amido-acetic Aldehyde (P). ....	1218
		Manufacture of Aromatic Sulphuryl Derivatives (P). ....	1218
		Manufacture of Vanillin (P). ....	1218
		Choate, P. C. Preparing Solutions carrying Salts of Zinc (P). ....	395
		Producing Metallic Zinc (P). ....	741
		Choay, E. See Behal. ....	791, 1087, 1087
		Chorley, J. C. See Baly. ....	418
		Christomanos, A. C. Apparatus for Valuation of Manganese Ores, &c. (Illus.). ....	1221

## C

	PAGE		PAGE
Christy, T. Discussion on Oxidised Linseed Oil .....	1022	Coreoran, B. Discussion on Coloring Tree Analysis .....	317
India-rubber .....	1135	Cordewener, L. and Kniwald, A. de. Apparatus for Manu- facture of Margarine and Alimentary Fat (P) .....	279
Chuard, E., and Jaccard, M. Alteration of the Sulphuric Acid in Wines .....	980	Cornell, W. N. Paper Pulp Imitating Natural Wood (P) .....	146
Church, A. H. The Laboratory Guide: A Manual of Practical Chemistry .....	769	Cosham, T. Construction of Filtering Beds (P) .....	1181
Ciamician, G., and Silber, P. Alkaloids of the Bark of Pome- granate Root .....	1217	Cousin, H. Action of Sulphuric Acid on Pyrocatechol and Homopyrocatechol .....	60
A New Derivative from Cotoneblack .....	756	Cawen, E. Method and Apparatus for Producing Cyanide (P) (illus.) .....	110
The Alkaloids of the Bark of the Pomegranate Root .....	416	Cowper-Coles, S. O. Method and Apparatus for Pickling Metal (P) .....	883
Claasen, J. Yield of Sugar from Mascacuite .....	1210	And Walker, B. W. Coating Metal Plates, &c. (P) .....	1067
Amounts of Coal and Steam required in the Manufacture of Sugar .....	824	And Walker, B. W. Deposition of Metals and Compound (P) .....	1067
Clapham, J., and others. Improvements in Bleaching and Dis- coloration (P) .....	390	And Walker, B. W. Electro-Deposition of Zinc, &c. (P) .....	1067
Preparing Fibres and Fabrics for Reception of Aniline Black (P) .....	728	Cox, J. A Metallic Alloy (P) .....	158
Clapp, W. J., and Williams, S. D. Manufacture and Purifica- tion of Iron, and Means therefor (P) .....	1201	Craftree, J. W. Rotary Washing and Dyeing Machines (P) ..	881
Clapperton, G. Practical Paper-making .....	549	Cramer, E. Nitrocellulose in Filter-Paper .....	833
Clark, E. O. Electrodes for Arc Lamps (P) .....	617	Crane, W. E. A Crucible Furnace for Burning Petroleum (illus.) .....	21
Clark, F. W. Distillation and Breaking-up of Liquid Hydro- carbons (P) .....	381	Crane, F. Electrolytic Apparatus (P) .....	819
Extraction of Naphthalene and other Hydrocarbons from Coal-tar, &c. (P) .....	797	Evaporating Apparatus (P) .....	872
Clark, J. Discussion on Electric Furnace for Laboratory Work Discussion on Waters for Dietetic Purposes .....	376	Method and Apparatus for Manufacturing Caustic Soda (P) (illus.) .....	636
Proceedings of Annual General Meeting .....	500	Salt Evaporators (P) .....	1186
Clark, W. Fluid Meats, and Manufacture of same (P) .....	168	Crane, Y. Evaporating Apparatus (P) .....	872
Food Preparations, and their Manufacture (P) .....	169	Crawford, T. C. See Bohm .....	627
Clarke, P. See Peuchen .....	251	Cresswell, C. G. Discussion on Oxidised Linseed Oil .....	1022
Clarkson, T. Discussion on the Sampling of Materials .....	217	Discussion on Sterilisation of Liquids .....	1137
The Sampling of Materials (illus.) .....	214	Cripps, F. S. Valves for Tar, Ammoniacal Liquor, &c. (P) ..	598
Clasen, J. Binding Medium for Size Colours, and Manufacture of Same (P) .....	962	Washers for Extracting Tar, &c., from Coal-Gas (P) (illus.)	379
Classen, Loss of Heat in the Steam Engines of a Sugar Factory .....	748	Critchlow, J. Filter Presses (P) .....	920
Classen, A. Electrolytic Determination of Lead .....	547	Crobaugh, F. L. Estimation of Graphite in Pig Iron .....	655
Quantitative Electrolytic Analysis .....	1223	Crochetelle, J. See Dumont .....	652
Claus, H. Manufacture of Enamelled Goods (P) .....	33	Crompton and Bros. Spontaneous Combustion of Lamp- Shades .....	670
Claus, W. H., and Rée, A. Manufacture of Blue Colouring Matter containing Sulphur (P) .....	384	Crompton, R. E. B., and Dowsing, H. J. Heating of Surfaces for Treatment of Paints, &c., by Electricity (P) .....	133
Clermont, P. de. Oxidation of Nickel Sulphide .....	151	Means for Use in Application of Electricity for Heating Liquids (P) .....	256
Clough, S. Apparatus for Gassing Yarn and Threads (P) .....	880	Crookes, W. Glazing Bricks, &c. (P) .....	519
Clowes, F. Discussion on Commercial Analysis of Lard .....	709	Select Methods in Chemical Analysis, Chiefly Inorganic ..	846
Discussion on Extinction of Flames .....	1156	Crosby, G. G. Disinfecting Apparatus (P) .....	1084
Discussion on Fermentation in the Leather Industry .....	221	Cross, C. F. Discussion on Electrolysis of Fused Salts .....	153
And Feilmann, M. E. Minimum Proportions of Carbon Dioxide and Nitrogen in Air Extinctive of Different Flames .....	1155	And Bevan, E. J. The Theory of Dyeing .....	551
Clubbe, E. J., and Southey, A. W. Secondary Voltaic Batteries (P) .....	15	Crossman, J. G. Apparatus for Fining Beers, &c. (P) .....	1082
Class, A. Pure Cultivation Yeast, and the Use of Antiseptics, in the Distillery .....	535	Crowther, H. W. Treatment of Waste from Gas-Liquor (P) ..	635
Clutterbuck, M. C. Rendering Materials for Surgical Bandages Antiseptic (P) .....	833	And Rossiter, E. C. Manufacture of Sulphocyanides (P) ..	887
Cochenhause, F. von. Valuation of Wool Fat .....	1100	Crozier, W. R. See Fellowes .....	682
Cochran, C. B. Composition and Sp. Gr. of Milk, Skim Milk, and Whey .....	269	Cudell, C. L., and M. Galvanic Batteries (P) .....	617
Cochrane, J. B., and Taylor, T. H. Purification of Iron for Casting, and Production of Steel and Wrought Iron (P) .....	711	Cunmer, F. D. Process and Apparatus for Drying or Heating (P) .....	247
Cohn, A. Electrolytic Preparation of Oxygen and the Halogens, and Production of Electrodes (P) .....	1205	Cunningham, J. See Addie .....	635
Coffin, C. L. Method and Apparatus for Electrically Heating or Working Metals (P) .....	161	Currie, W. C., and Rowlands, W. E. Apparatus for Drying Animal and other Matters (P) .....	714
Cohen, E. Electrolytic Copper Deposition in the Stolberg Lead Works .....	817	Cushny, A. R. The Alkaloids of Gelsemium Sempervirens ..	79
Cohen, J. B. Discussion on Mordanting Wool with Chromium Cohn, G. See Ehrlich .....	367	Cutler, S. Gas Condensers (P) .....	626
Cohn, L. Manufacture of Leather for Gloves (P) .....	894		
Cohn, T. A Protective Paint (P) .....	46		
Collins, F. N. Apparatus for Regulating the Temperature of Fermenting Liquids (P) .....	806		
Collins, J. M. Machines for Dyeing and Seaming Yarn (P) ..	389		
Colls, W. B. See Peacock .....	260		
Columbian Pottery Company. Kilns (P) .....	1063		
Comer, H. Discussion on Detection of Soap in Lubricants ..	1179		
Compagnie du Sel Agglomere. Forming Salt into Blocks by Fusion (P) (illus.) .....	535		
Compagnie Electro-Chimique de St. Bérn. Treatment of Sodium Chloride and Utilising Products in Manufac- turing White Lead, &c. (P) .....	886		
CConnell, J. T. Treatment of Hops (P) .....	896		
Conroy, J. Laundry Glaze (P) .....	531		
Cook, E. H. Effect of Heat on Iodates and Bromates .....	734		
Cook, R. Continuous Drying Kiln (P) .....	1047		
Cooper, C. F. See Hermite .....	170		
Cooper, W. J. See Wanklyn .....	627		
Copeland, C. Saggars for Use in Pottery Kilns (P) .....	588		

## D

Dagger, J. H. J. Discussion on Aluminium Alloys .....	50
The Manufacture and Industrial Value of Aluminium Alloys .....	4
Dahl, G. A. Production of Dyes (P) .....	879
Dahmen, H. Ritter von. Manufacture of Explosives (P) .....	1219
Dairy Supply Co., The. See Woosnam .....	1060
Dammer, O. Handbuch der Anorganischen Chemie .....	1237
D'Andrea, M. N. Manufacture of Paints and Acids from Proto-sulphate of Iron (P) .....	163
Manufacture of Red Oxide Paints (P) .....	262
Dankelman, A. Sodium Polysulphide for Cleansing Purposes (P) .....	945
Dantzenberg, F. X., jun. Melting Furnaces (P) .....	846
Darier, G. Chrysin .....	181
Darling, J. D., and Forrest, H. C. Producing Nitric Acid and Metals from Nitrates (P) .....	647
Darmstadter. See Jaffé .....	259
Dasse, G., and Fettweiss, C. Drying and Carbonising Appa- ratus (P) .....	1192
Davie, J. See Wotherspoon .....	505
Davies, M. L. Discussion on Rectification of Oil of Vitriol ..	249
Davis, G. E. Construction of Ammonia Apparatus (P) .....	948
Discussion on Sulphuric Acid Pipe Columns .....	1150
Discussion on the Sampling of Materials .....	217
Davis, J. E. Rolls of Machinery for Treating India-Rubber (P)	378
Davy, C. See Allen .....	815

	PAGE		PAGE
Dawson, J. Apparatus for Washing Wool or other Fibres (P).....	802	Dressel, O., and Kothar, R. Some Sulphonations of Naphthalene Derivatives.....	875
Day, D. T. Mineral Resources of the United States.....	846	Dreyfus, C. Discussion on the Vulcanisation of India-rubber. On Resist and Discharge Styles with Aniline Black.....	17 485
Day, W. C., and Bryant, A. F. Note on Pemberton's Method of Phosphoric Acid Determination.....	657	Proceedings of Annual General Meeting.....	693, 693
Deeley, R. M. See Archbutt.....	869	Production of New Colouring Matters (P).....	1191
Deering, W. H., and Redwood, B. Report on Castor Oils from Indian Section of Imperial Institute.....	959	See Baerlein.....	939
DeGENER, J. Assays by Concentrating Beetroot Juice.....	1240	Statement on Opening of the Manchester Ship Canal.....	11
Dehmer, P. P. Preservation of Timber from Attacks of Insects.....	40	Drigfield, V. C. See Gaskell, jun.....	1186
Dehn, N. von. Comparative Value of Various Phosphatic Manures.....	263	See Muspratt.....	38
Delbrück, M. Drying Potato Pulp with Molasses for Cattle Food.....	365	Drucker, See Steffen.....	823
Delhotel, E., and Moride, F. Improvements in Filters (P).....	136	Drummond, J. Apparatus for Filtering and Separating Liquids (P).....	377
Demond, C. D. See Hofman.....	638	Dubers, F., and Mohrdieck, P. Electrical Battery (P).....	1068
D. Mesenthal, H. Discussion on Sterilisation of Liquids.....	1187	Dubets, See Mixer.....	546
Discussion on Treatment of Gold Ore in Transvaal.....	349	Dubois, H. See Muller.....	972
Treatment of Gold Ore in the Transvaal (illus.).....	326	Dudley, C. B., and Pease, F. N. Determination of Phosphorus in Steel.....	665
Dempster, R. An Improved Furnace (P).....	871	Dudley, W. L. Action of Gaseous Hydrochloric Acid and Oxygen on the Platinum Metals.....	255 255
Danges, G. Volumetric Determination of Silver in any Form.....	664	The Electro-deposition of Iridium.....	110
Dépierris, J. Production of Fast Reds in Calico without Mordants.....	1194	Duff, E. J. Gas Producers (P).....	633
Desesquelles, E. Mercuric Phenolates and certain of their Derivatives.....	835	Duffon, A. The Fading of Colours.....	53
Desimukh, M. G., and Gajjar, T. K. Rasa-Ramya-Rahasya.....	288	Dull, G. See Lintner.....	715
Desprez, E. M. New Elastic Material, and Application of same (P).....	1200	Dumas, A. Pipes for Heating, Cooling, or Evaporating Apparatus (P).....	530
Devi, J. S. Apparatus for Blending Powdered Materials.....	1181	Tubes for Heating, Refrigerating, &c. (P).....	652
Apparatus for Treating Powdered Materials (P).....	1181	Dumont, J., and Crochetelle, J. Influence of Potassium Salts on Nitridation.....	318
De Vries, Transparent Cement for Porcelain.....	888	Dunham, R. W. Discussion on Colorimetric Analysis.....	691
Dewald, Combustible Gases arising from the Diffusion Battery.....	749	Dunlop, Baillie. Speech at Annual General Meeting.....	1155
Dewan, J. Method and Means for Producing Vacuum (P).....	23	Dunn, W. G., and others. Means of using Baking Powder (P).....	1083
Dewan, Lord Provost. Speech at Perth Banquet.....	707	Dunstan, W. R. Aconitine.....	540
Dickson, C. See Stone.....	178	And Carr, E. H. On the Aconite Alkaloids, Part IX. The Action of Heat on Aconitine.....	172
Dickson, J. See Macnab.....	750, 929	And Carr, E. H. On the Aconite Alkaloids, Part X. On the Conversion of Aconitine into Isaconitine.....	172
Ditzmann, M. The German Chemical Trade since 1885.....	1110	And Harrison, E. F. On the Aconite Alkaloids, Part VIII. Picroconitine.....	172
Dugby, E. J. T. Material for Crucibles or Melting Pots (P).....	158	Dupont, See Vivien.....	902
Dillon, T. A. Preparing Peat for Heating, &c. (P).....	212	Dupre, A. Discussion on Dissolved Organic Matter in River Waters.....	321
Dismore, J. H. R. Gas Hydraulic Mains (P).....	626	Discussion on Sodium Peroxide Explosion.....	199
Manufacture of Gas (P) (illus.).....	934	Explosion Caused by Sodium Peroxide.....	198
Don, J. B. C. Thermo-Electric Batteries and Apparatus for Making Same (P).....	890	Spontaneous Combustion of a Lamp-Shade.....	670
Ditte, A., and Metzner, R. Action of Metals on Acid Solutions of their Chlorides.....	43	Durand, J. C. L., and others. Manufacture of Colouring Matters (P).....	247
Divers, E. The Manufacture of Camol in Japan (illus.).....	108	And others. Manufacture of New Colouring Matters derived from Dioxidiphenylmethane (P).....	145
Dixon, G. See Musgrave.....	1048	And others. Manufacture of New Condensation Products and Colouring Matters (P).....	1055
Dixon, J. W., and Skinner, W. Metallic Alloy (P).....	957	And others. Manufacture of New Substantive Colouring Matters for Dyeing Cotton (P).....	802
Dobbin, J. J., and Lauder, A. Corydaline.....	1217	And others. Manufacture of New Unsymmetric Diamido Bases and Tetrazo Colouring Matters derived therefrom (P).....	146
The Alkaloids of Corydalis Cava.....	1217	And others. Manufacture of Unsymmetrical Diamido Bases, and Colouring Matters therefrom (P).....	32
Dodgson, J. W. See Burch.....	525	Durio, J. Tanning Hides and Skins (P).....	1074
Dods, H. Influence of various Substances on Estimation of Potash by Tate's Process.....	1055	Dvorkovitz, P. Discussion on Distillation of Peat.....	607
Dodgner, O. The Volatile Oil of Rowan Berries, Parasorbic Acid, and its Isomerism with Sorbic Acid.....	539	Manufacture of Coke and Coal-Gas, &c. (P).....	626
Döerfling, P. Influence of Percentage of Fat in Sugar-beet Seed, and Size of Latter on Richness of Beets in Sugar.....	410	The Distillation of Peat (illus.).....	596
Dombrows, H. A. A. See Parent Syndicate Oil Extractor Co.....	240	Dyer, Bernard. Analytical Determination of "Available" Mineral Plant Food in Soils.....	288
Donaldson, W. Heating Gases by Electricity (P).....	529	Dyson, E. Apparatus for Concentrating Acids (P).....	1659
Dont, F. See Singer.....	645		
Donath, C., and Strasser, R. Estimation of Indigotine in Indigo (illus.).....	426		
Donath, E. Galvanised Iron Sugar Moulds with Internal Coating.....	823		
Logwood Extract.....	982		
The Inertive Action of Glycerol.....	823		
And Strasser, R. Note on Stearin Pitch.....	380		
Doolittle, O. S., and Eavenson, A. Determination of Phosphorus in Steel by Dudley's Method.....	666		
Dor, C. H. See Anderson.....	729		
Doulton, H., and Slater, J. Treating Vitreous Surfaces for Hermetically joining Same (P).....	697		
Doulton, H. L., and Carr, J. M. Moulds for Terra-cotta Blocks (P).....	948		
And Manger, A. W. Softening and Purifying Water, and Apparatus therefor (P).....	832		
And Rix, W. P. Ornamenting Pottery Ware (P).....	335		
Dowling, H. J. See Grompton.....	138, 256		
Drake, T. Electrolytic Production of Caustic Soda and Chlorine (P).....	714		
Producing Caustic Soda or Potash and Chlorine by Electrolysis (P).....	887		
Dreyson, W. B. H. Connecting Metal to Earthenware or China (P).....	811		
Dräger, W. P. Discussion on the Theory of Dyeing.....	100		
Theory of Dyeing. (Part I.) "Intram Colours".....	161		
Druckmann, Grey Colour of Raw Beetroot Sugars.....	1210		

## E

Eaton, G. O. See Storer.....	816
Eavenson, A. See Doolittle.....	666
Ebert, G. Derivatives of Toly-Antipyrine.....	59
Eckenroth, H., and Kock, K. Quantitative Estimation of Acetone and Salicylic Acid in "Sal acetol".....	288
Edmunds, K. C. Explosives (P).....	1090
Edney, W. See Morris.....	504
Edwards, J. See Rushford.....	271
Edwards, V. Still or Retort for Distillation Process (P).....	700
Elfront, J. Fermentation of Molasses.....	824
Influence of Chemical Conditions on Activity of Brewers' Yeast.....	267
Influence of the compounds of Fluorine on Beer-Yeast.....	968, 1079
Inurement of Ferments to Action of Antiseptics, and Effect thereof.....	1213
Note on Fermentation of Molasses.....	533
Preserving Food, &c., and Supplying Sterilised Air to Rooms (P).....	833

	PAGE		PAGE
Feenoff, J., Reichler's Artificial Diastase .....	1080	Farbenfabriken vorm. F. Bayer and Co., Manufacture of Ray-	1000
The Diastase of Cereals .....	1080	Compounds derived from Pyrazine (P) .....	240
Ehrenberg, A., Extract of the Root of Aspidium Filix .....	758	Manufacture of Colouring Matters (P) .....	383, 383, 541
Ehrlich, E., The Newly-formed Sugars of Malt .....	835	Manufacture of Colouring Matters and Dyestuffs and Pre- paring there-with (P) .....	541
Ehrlich, P., and Cohn, G., Action of Acid Chlorides on Nitroso- dimethylamine .....	50	Manufacture of Colouring Matters from Anthraquinone (P) .....	1190
And Einhorn, A., Physiological Action of Cocaine Com- pounds .....	1218	Manufacture of Condensation Products from Anines or Derivatives thereof (P) .....	804
Einhorn, A., See Ehrlich .....	1218	Manufacture of Dyestuffs (P) .....	631
Einhorn, E., and Willstätter, R., The Technical Preparation of Cocaine .....	1217	Manufacture of Leuco Compounds and Colouring Matters therefrom (P) .....	722
Eitner, W., Refined Fish Tallow .....	894	Manufacture of New Alizarin Dyestuffs (P) .....	783
Ekenberg, M., Method of Separating Fats (P) .....	46	Manufacture of New Sulphoacids and their Derivatives (P) .....	542
Production of an Improved Fat Emulsion (P) .....	46	Manufacture of Piperazine and its Salts (P) .....	275
Ekin, C., and Thew, W. H., Manufacture or Preservation of Condensed Milk (P) .....	828	Manufacture of Sulphoacids of the Naphthalene Series and Colouring Matters (P) .....	246
Ekman, C. D., Obtaining Products from Sulphite Process Liquors (P) .....	1085	Production of Amido-naphthol Sulpho Acids and Colouring Matters therefrom (P) .....	800
Elbers, A. D., Smelting Reactions of Blast Furnace Slag, con- sidered on Thermo-Chemical Principles .....	598	Production of Alkylated Hydroxynaphthalene Sulphone Acid and Colouring Matters therefrom (P) .....	745
The Utilisation of Blast Furnace Slag .....	633	Production of a Naphthylamine $\beta$ - $\alpha$ - $\beta$ -Trisulpho Acid and other Sulpho Acids therefrom (P) .....	878
Elektrochemische Werke Corporation. See Kiliani .....	1068	Production of a Trihydroxynaphthalene Monosulpho Acid (P) .....	1074
Eliot, H., Studies on Yeast .....	293	Production of Beta, Beta, Dihydroxynaphthalene Beta, Beta, Disulpho Acid (P) .....	1192
The Evolution of Carbon Dioxide and Hydrogen Sulphide in Brewing .....	267	Production of Colouring Matters (P) .....	1130
Ellermann, F. W., Electric Accumulators (P) .....	161	Production of Colouring Matters and Materials therefor (P) .....	245, 801, 942
Elliott, S., A Liquid Fertiliser, Insecticide and Disinfectant (P) .....	652	Production of Colouring Matters from Anthraquinone (P) .....	723
Elliott, T. W., Composition for Coating Iron and Steel (P) .....	962	Production of Colours on Fibres (P) .....	722
Ellis, C. J., See MacArthur .....	526	Production of Derivatives of Betanaphthoquinone or Beta- naphthohydroquinone and Colouring Matters there- from (P) .....	31
Emden, T. W. L., Dubbing or Dressing for Leather (P) .....	747	Production of Derivatives of Hydroxyanthraquinones (P) .....	144
Emery, H. C. W., Cells for Electric Batteries (P) .....	494	Production of Direct Dyestuff Colouring Matters (P) .....	245
Emmense Zinc Co., The, Treatment of Zinc-Lead-Sulphide Ores (P) .....	402	Production of Dyestuffs (P) .....	760
Endemann, H., Discussion on Detection of Soap in Lubricants .....	1178	Production of Esters of S-phosphoric Acid (P) .....	60
Enquist, E. W., Manufacture of Carbonic Acid and Bye- Products (P) .....	948	Production of Naphthylamine sulpho Acids (P) .....	800
Engel, M. R., Analytical Separation of Chlorine and Bromine .....	1004	Production of New Colouring Matters (P) .....	30, 943
Engler, C., and Singer, L., Detection of Paraffin and "Lubricat- ing Oil" in Distillate of Train Oil .....	45	Production of Sulpho Acids Derived from $\beta$ -Naphthol- $\beta$ - Monosulpho Acid (P) .....	877
English, A. B., See Richardson .....	401	Production of Sulpho Acids Derived from $\beta$ -Naphthyl- amide- $\beta$ - $\beta$ -Disulpho Acid (P) .....	877
English, F. H., Volumetric Estimation of the Bases in certain Metallic Salts .....	545	Production of Sulpho Acids Derived from Naphthalene $\alpha$ - $\beta$ - $\beta$ - $\alpha$ -Trisulpho Acid (P) .....	878
Epstein, L., Maintaining Positive Plates of Secondary Voltaic Batteries in good operative Condition (P) .....	743	Production of Sulpho Acids of the Naphthalene Series (P) .....	1191
Erban and Specht, Improvements in Turkey-Red Dyeing .....	150	The Manufacture of Dyestuffs (P) .....	500, 510
Dyeing Textiles with Mordant Dyes (P) .....	1194	Farbenfabriken vorm. Meister, Lucius und Brunner, Dyeing Wool with Fast Colours (P) .....	633
Erdmann, H., Oxidation of Chloroform by Chromic Acid, and Preparation of Carbonyl Chloride (illus.) .....	65	Dyeing Wool with Sulphonic Acids of Anthraquinone Colouring Matters (P) .....	884
Recent Developments in Pharmaceutical Chemistry .....	540	Manufacture of Acid Colouring Matters (P) .....	247
Ernst, O., Stop-cock for Vacuum Desiccators (illus.) .....	62	Manufacture of a Novel Colouring Matter (P) .....	878
Ernst, R., Conditions for Production of Generator Gas .....	379	Manufacture of a Novel Diamido Base and of Colouring Matters therefrom (P) .....	384
Etaix, L., New Extraction Apparatus (illus.) .....	839	Manufacture of Caffeine Sulphonates (P) .....	846
Etebells, J. C., Apparatus for Supplying Liquid Fuel (P) .....	936	Manufacture of Colouring Matters (P) .....	384, 722
Evans, E. P., See Worcester Porcelain Co. ....	1197	Manufacture of Diamido-dibenzimidazols and Colouring Matters therefrom (P) .....	941
Evans, R. E., Clarification and Preservation of Syrups, Ex- tracts, &c. (P) .....	827	Manufacture of Thionine Colouring Matters (P) .....	247
Evelyn-Liardet, W., Safety Explosive, and Manufacture there- of (P) .....	61	Producing Fast Colours from Direct Dyes on Cotton (P) .....	883
Evers, F., Compounds of Sugars with Iron .....	445	Producing Insoluble Azo Colours on Fibre (P) .....	1054
Evershed, F., Discussion on the Theory of Dyeing .....	1009	Production of Colouring Matters (P) .....	247
Ewan, Dr., Discussion on Action of Antiseptics on Yeast .....	1164	Production of Colouring Matters from Meta-amidophenol- phthalenes (P) .....	760
Ewdokimoff, B., See Nadiem .....	56	Production of Green and Bluish-Green Colouring Matters (P) .....	720
Ewing, R., Earthenware and other Pipe-Joints (P) (illus.) .....	788	Production of Iodo-derivatives of the Succinimide and of Substituted Succinimides (P) .....	800
		Production of Oxy-Anthraquinone (P) .....	878
		Production of Para-ethoxy- and of Para-methoxy-phenyl- succinimides (P) .....	800
		Farmer, J. S., and others, Manufacture of Linoleum and Similar Floor-cloths (P) .....	33
		Farr, E. H., and Wright, R., Methods for Determination of Alkaloids in Alkaloidal Tinctures .....	983
		Faulkner, E., and Johnson, J. E. J., Production of Extracts from Malted Grain (P) .....	1214
		Fawcitt, C. A., Discussion on Production of Oil-Gas .....	501
		Fayolle, M., See Villiers .....	1002, 1014, 1022, 1093, 1098
		Fehrenbaen, G., and Prud'hon, L., Producing Artificial Whal- bone, and forming same into Strips (P) .....	726
		Feilmann, F. E., See Clowes .....	1155
		Feilmann, M. E., Discussion on Extinction of Flames .....	1156
		Feldmann, W. R., The Cyanide Process in the Transvaal Mines .....	951, 952
		Felmann, J., Loss of Sugar in Manufacture .....	105
		And Herles, F., Estimation of Amount of Sugar lost during Boiling of the Juice .....	165
		Fell, A. G., Methods of Treating Lead Ores (P) .....	880
		Fellowes, F. W., Some Micro-Organisms causing Diseases of Beer .....	1080
		Fellowes, H. C., and others, Extraction of Rhea and other Fibres (P) .....	632
		Fentley, A. V. C., and J. B., and Moore, G., Coating Iron and Steel with Brass and other Alloys (P) .....	409



	PAGE		PAGE
Geyer, G. de. Process and Apparatus for Saccharification and Treatment of Wort in Brewing, &c. (P) (illus.)	539	Gredit, P. Treating Blast-Furnace Gases (P)	816
Gibbs, A. A., S. A., and W. T. Process for Ornamenting Glass (P)	888	Grée, P. D. de la. Evaporating Apparatus (P) (illus.)	1179
Gibson, J. M. Proceedings of Annual General Meeting	692, 693	Green, A. G. Discussion on the Theory of Hygroscopy	101
Gilchrist, J. See Spence	394	The Oxidation of Paracetamol	145
Gilchrist, P. S. Pipe Columns, in Manufacture of Sulphuric Acid (illus.)	1112	Translation of Schultz and Jolius' "Systematic Survey of the Organic Colouring Matters"	671
Gildermister, E. See Bertram	831	Green, L. See Ash	401
Giles, W. B. A Modification of the Lître Flask (illus.)	277	Green, W. A. Amalgamating Apparatus (P)	102
Gill, A. H. Determination of Nitrates in Potable Water	633, 634	Greening, E. W. and E. O. Treatment and Utilisation of Yeast (P)	1082
Gill, H. W. See Ash	101	Method and Means for Utilising Spent Hops (P)	655
Gill, J. C. Test for Free Sulphur	175	Greenwood and Batley, Ltd., and Lambert, H. Hydraulic Oil Press (P)	406
Gillibrand, R. T. Treating Leather, &c., with Fatty Matters (P)	651	Grezer, F. Augmentation of the Fermentable Capacity of Molasses	268
Gilmour, J. D. Manufacture of Cyanides and Cyanogen Compounds (P)	37	Gregg, J. A. S. Voltaic Batteries (P)	1207
Gilson, E. Crystallisation of Cellulose, and Chemical Composition of the Vegetable Cellular Membrane	1106	Gregor, G. The Estimation of Hydrocyanic Acid	176
Girard, A. The Temperature of Bread during Baking	269	Gregory, G. W. Thermostatic Instruments (P)	713
Girard, A. C., and Street, E. A. G. Electric Furnaces (P)	626	Gregory, H. R. Manufacture of Lead Pigments (P)	618
Manufacture of Carbon for Electrical Purposes (P)	647	Gréhan, N. Examination of Products of Combustion of Coal-Gas by Argand and by Auer Burners	1215
Giron, P. The Testing of Portland Cement	39	Experiments with Auer Incandescent Mantle	1059
Gittins, C. E. Apparatus for Softening Water (P)	271	Gröb, A. Execution of Masonry and Brickwork during Frost	713
Glaser, M. Sources of Error in Roesse's Method of Estimating Fuel Oil	982	Griggi, G. Detection of Mineral Acids in Vinegar	423
Glaser, C. Analysis of Pyrites	1095	Grillo, J., and Schroeder, M. Production of Ghee and Gelatin from Bones (P)	498
Glaser, F. Estimation of Phosphoric Acid by the Citrate Method	1225	Grimaux, E. Essence of Tarragon, its Transformation into Anethol	658
Glasgow, A. G. Water-Gas Apparatus (P)	935	And others. Physiological and Therapeutic Action of the Homologues of Quinine	1086
Glendinning, T. A. Discussion on Bacteriological Analysis of Water	1160	Grime, J. Oxidising Indigo Blues, and Apparatus therefor (P)	728
Glenk, R. Mucassar Oil	45	Vats for Dyeing Indigo Blues (P)	633
Goddard, R. J. Damp-resisting Materials (P)	1063	Grimshaw, H. Discussion on Modern Systems of Sewage Treatment	600
Godshall, L. D. The Chlorination of Gold Ores	155, 157	Resolution on Opening of the Manchester Ship Canal	11
Goebel, H. Manufacture of Paris Blue, &c., by Electrolysis (P)	876	Groche, E. Preservation of Wood (P)	949
Goez, G. A., and Fröh, J. C. Imitation of Marble (P)	638	Groeger, M. Estimation of Iodine in Presence of Bromine and Chlorine	420
Goehlich, W. The Alkaloids of the Papaveraceæ: Codeine	757	Groger, M. Preparation of Pure Potassium Iodate	419
Gohring, C. F. Prevention of Petroleum Spirit Fires in Chemical Cleaning	885	Gromier, G. Dressing Spathic Iron Ore at Allevard	1064
Goldberg, A. Paranitrophenol Sodium as an Acid Indicator	419	Grosche, B. Apparatus for Vaporising and Burning Mineral Oil (P)	139
Goldiner, F. See Harduck	966	Gross, A. Manufacture of a Detergent (P)	261
Goldmark, L. See Nienstaedt	511, 618	Grossmann, J. Manufacture of Hydrosulphurous Acid and Hydrosulphites (P)	1060
Goldthorp, H. and A. J. Metallic Tubes for Heating Liquids (P)	920	Manufacture of Hydrosulphurous Acid and Hydrosulphites	1196
Gomess, A. F. B. Treatment of Textile Vegetable Fibres (P)	248, 941	Grote, L. Manufacture of Artificial Stone, Preservative Paint, &c. (P)	255
Gooch, F. A., and Hodge, B. Detection and Separation of Arsenic	979	Grouvelle, J., and Arquebuz, H. Steam Superheaters (P) (illus.)	930
Goodwin, E. J. Manufacture of Sheet Material Suitable for Labels and Bookbinding (P)	148	Grumbacher, F. M. Preserving Vegetable Substances, and Obtaining Products of Distillation (P)	628
Treatment of Textiles for Stiffening same, and Production of Tracing Cloth (P)	803	Grunhut, L. Gravimetric Methods for Estimating Sugars	845
Goppelsroeder, F. The Reduction of Indigotine to Indigo White	358	Gruschwitz, A. Treatment of Textile Fibres (P)	1056
Gordon, A. and J. Process and Apparatus for Manufacture of Cyanides (P)	886	Gruy, T. Formation of Aluminium Sulphide	712
Gore, G. Decomposition of Liquids by Contact with Powdered Silica	428	Guerbet, Campholene	628
Gorlitz, A. Producing Intensely Brilliant Pictures on Glass (P)	811	Gui, G. Manufacture of Cement (P)	638
Gosling, A. Tariff Changes in Guatemala	989	Guichard, T. Composition and Analysis of Yeast	844
Göttig, Reliability of the Principal Methods of Determining total Carbon in Iron	421	Guignard, L. Localisation of the Active Principles of Mignonne	59
Various Methods of Estimating Carbon in Iron	1096	Guinard, Quinaseptol or Diaphthol	903
Göttig, C. Producing Metallic Deposits, especially on Aluminium	1200	Gulcher, R. J. Plates for Accumulators (P)	744
Gounelle, C. Oil and other Presses (P)	1048	Gunn, O. B. Modern Street Pavements	736
Gowland, W. A Japanese Pseudo-Spice (Shiromi) and its Relation to Purity of Japanese Copper and Presence of Arsenic in Japanese Shiromi (illus.)	463	Guntz, M. Preparation of Metallic Lithium	255
Discussion on Japanese Shiromi and Copper	471	Gurlings, P. The So-called Scum Fermentation in Japanese Sugar Factories	823
Goyder, G. A. Extraction of Gold from South Australian Ores by Potassium Cyanide	523	Gutensohn, A. Utilisation of Old Tin-Plate Articles (P)	401
Graebe, C. The Colour of Acenaphthylene	27	Guthrie, H. Electrolytic Apparatus (P)	1206
And Philips, A. Hydroxy-Derivatives of Anthraquinone-quinone	28	Guthrie, M. See Baily	818
Grand'Montagne, F. Manufacture of Artificial Marbles (P)	254	See Browne	594
Grandmougin, E. See Kopp	720, 726	Guttman, O. Discussion on Smokeless Powder	583
See Michel	37	Discussion on Sterilisation of Liquids	1137
Grandval, A. See Lajoux	58	The Manufacture of Smokeless Powder (illus.)	575
Grant, H. Report on Preserved Fruit Industry of Portugal	993	Guyot, E. G. See Latham	713
Gray, A. Manufacture of Zinc Oxide (P)	401		
Gray, G. Watson. Discussion on Methods for Estimation of Manganese	114		
Gray, J. Discussion on Paraffin Lamp for Laboratories	1167		
Retorts for Distilling Shale (P)	1054		
Gray, J. H., and Henderson, J. B. Effects of Mechanical Stress on Electrical Properties of Metals	957		

Haase, F. Insecticides (P)	830
Hadfield, R. A. Manufacture of Manganese Steel (P)	711
Results of Heat Treatment on Manganese Steel, and their bearing on Carbon Steel	641
Haeffley, E. Dyeing Yarns and Fabrics in Turkey Red (P)	513
Haen, E. de. Treatment of Cotton Cloths for Filtering Purposes (P)	34



	PAGE		PAGE
Hahnlein, F. H. Action of Salt on Putrefying Bacteria of Hairs Origin of Acid Fermentation in Tanning Liquors.....	49 650	Hawker, E. W. Report on the Bartlett Zinc-Lead Process....	41
Raackmann, C. T. L. New Fermentation Apparatus (illus.).....	64	Hawker, H. See Worcester Porcelain Co. ....	1197
Hahn, S. Manufacture of Faint Imitating Mother of Pearl (P).....	1072	Hay, A. B., and Park, J. M. Dyeing Cotton Fabrics and Yarns (P).....	391
Haines, R. Normal Chlorine in Spring Waters.....	828	Hedrick, M., and Goldiner, F. Hop-Tannin.....	906
Hairs, Estimation of Saccharine in Presence of Salicylic Acid.....	124	Hobbrand, The Chances Produced in Bread by Mould.....	412
Hakansson, P. Anti-septic and Preserving Agents (P).....	898	Hockel, L., and Schlagdenhauffen, Gardenia Resin.....	47
Hall, J. W. Treating Complex and Refractory Ores to obtain Metals and Pigments (P).....	741	Hoesler, F. See Seybold.....	748
Hall, T. G. Appliance for Breaking down Crusts in Gas-Pro- ducers (P).....	627	Hofmann, R. The Investigation of Saccharin.....	835
Process and Apparatus for Electrical Treatment and Pro- duction of Gaseous B. L. S. (P).....	528	Hofmann, F. Soap Tablets, Cakes, or Bars (P).....	902
Halske, See Semiers.....	31	Hömpel, A. See Morel.....	623
Hamilton, H. A New Compound of Soap (P).....	196	Hörmisch, W. Behaviour of the Calcium Salt of Veratric Acid on Dry Distillation.....	171
Hammond, J. B. S. Wright.....	1067	Hornelmann, G. Boiling Fermentations: A Criticism on Hesse's Results.....	968
Hampel, W. Determining Foreign Metals in Commercial Copper.....	421	Hornhering, C. Improvements in Tanning (P).....	651
Hanbury, C. Electrolytic Apparatus (P).....	45	Höbling, H. Manufacture of Emulsions (P).....	46
Handy, J. O. Determination of Phosphorus in Steel.....	664	And Passmore, F. W. Quantitative Estimation of Acetone and Salicylic Acid in "Sal Acetol".....	288
Hannay, J. B. Interrelation of Sulphide with Sulphate and Oxide of Lead.....	812	Hell, G. Occurrence of Copper in Extracts.....	902
The Metallurgy of Lead.....	639	Heller, O. Valuation of Crude Glycerol.....	908
Hanquiaux, J. B. Ovens for Firing Ceramic Ware (P).....	1048	Hellrich, A. Analysis of Potassium Nitrate.....	979
Hanriot, M., and Richey, C. Chlorolose.....	755	Helmers, L. O. Obtaining Aqueous Solutions of Phenols, &c. (P).....	900
Hanson, J. Report for Treating Town Refuse (P).....	899	Obtaining Sulphonic Acids and Sulphone-like Compounds (P).....	508
And others. Manufacture of Gum from a Waste Product (P).....	410	Hely, A. A. Preservation of Wood (P).....	154
Hanson, L. S. Brantons.....	883	Hemingway, H. W. Manufacture of Oxide of Iron (P).....	163
Hantke, See Wahl.....	805	Hempel, W. Determination of Carbon in Iron and Steel (illus.).....	421
Hantzsch, A., and Freese, H. Sulphur Compounds Accom- panying Aromatic Bases.....	1189	Henderson, G. G. Discussion on Waters for Dietetic Purposes.....	501
Haupt, F. Defecation of Beet-root Juice with Ammonia.....	293	Henderson, J. B. See Gray.....	957
Estimation of Quantity and Purity of Masecques.....	165	Hendrichs, V. Means for Producing Waterproof Ventilating Cloth (P).....	1192
New Tube for Polarimetric Observations.....	278	Hendrick, J. Discussion on Waters for Dietetic Purposes....	501
Hapke, L. Die Selbstentzündung von Schiffschadungen, &c.....	289	Hendfeldt, E. Valuation of Artificial Manures.....	1224
The Causes and Prevention of Spontaneous Combustion....	26	Henneberg, W. Apparatus for Condensing and Heating (P)....	713
Hardemann, G. G. M. See MacWhinnie.....	745	Henriques, H. Analysis of India-Rubber Substitutes.....	48
Hardy, E. Apparatus for notifying Presence of Gases in Air (P).....	1091	Henriques, R. Analysis of India-Rubber Articles.... 986, 987, 988 Analytical Control of Vulcanisation of India-Rubber.....	987
Hardy, J. T., and others. Fastening the Colours upon and in Coloured Leathers (P).....	894	Deductions from the Analysis of India-Rubber Articles... 70 India-Rubber Substitutes.....	47
Hargreaves, J., and Bird, T. Apparatus for Electrolysis of Chlorides and other Salts (P).....	958	Kapok Oil.....	258
Apparatus for Electrolysis of Iodides, &c. (P).....	886	Regenerated India-Rubber.....	407
Electrolysis of Iodides, Bromides, &c. (P).....	886	Henny, E. Estimation of Ammonia in Ammoniacal Liquor, &c. Estimation of Nitrates by Means of the Nitrometer (illus.)	764 764
Electrolytic Cells and Diaphragms (P).....	255	Hensel, J., and Spiethoff, F. Manufacture of Mineral Manure (P).....	1075
Making Electrolytic Apparatus (P).....	528	Henwood, E. N. Liquid Fuel.....	792
Manufacture of Alkali, and Apparatus therefor (P).....	250	Hepp, E. See Fischer.....	29, 29, 39
Method and Apparatus for Electrolytic Production of Alkali, &c. (P).....	528	Herbert, F. Lining Pipes with Enamel, Glass, &c. (P).....	154
Harkness, A. Report on the Cotton-Oil Industry in South Carolina.....	992	Herrbig, W. The Examination of Wool Fat.....	1068
Harlock, E. B. Apparatus for Continuous Evaporation of Liquids (P).....	23	Herbst, C. W. C. See Ortmann.....	137
Harper, J. R. See Kriens.....	614	Herrisse, E. Elastic Composition for Moulds (P).....	1211
Harris, D. Proceedings of Annual General Meeting.....	603	Heries, F. See Felemann.....	105
Harrison, E. F. See Dunstan.....	172	Hermite, E., and others. Improvements in Disinfecting (P)....	170
Hart, E. Apparatus for Condensing Nitric Acid and other Vapours (P) (illus.).....	1197	Method and Apparatus for Disinfecting (P).....	271
Hart, F. Cotton, Steam and Midge Oil.....	237	Heron, J. Manufacture of Compounds for Production of Ginger Beer, &c. (P).....	1082
Margarin rendered Consistent by Admixture of Phenol- phthalin.....	412	Preserving Yeast for Transport (P).....	168
Hart, P. Discussion on Oxidation and Corrosion of Iron and Steel.....	120	Herrmann, A. Colouring Tanned Hides by Means of Smoke (P).....	964
Hartley, J. W. Gas-making Apparatus (P).....	1050	Herrmann, C. See Nienstaedt.....	648
Hartley, W. N., and Blinkinsop, W. E. B. Treatment of Oils for Refining, Bleaching, &c. (P).....	647	Herrmann, P. Estimation of Calcium Sulphide in Animal Charcoal.....	546
Hartmann, F. Manufacture of a Substitute for Whalebone and Horn (P).....	1075	See Perl.....	1072
Hartidge, W. B. Manufacture of Fuel (P).....	873	Herscher, E. Continuous Sterilising Apparatus (P).....	1216
Hartung, M. Storage Batteries (P).....	256	Herz, W. Matches and Striking Surfaces therefor (P).....	1099
Haslam, J. G. Yarn-dyeing Machines (P).....	391	Herzfeldt, A. Comparative Defecation Experiments with Beet- root Juice.....	823
Hatch, A. I. Process and Apparatus for Utilising Producer- Gas for Calcining Limestone (P) (illus.).....	636	Precipitates formed during Heating of Beet Juice.....	749
See Koenig.....	42	Hertz, J. Brasilin and Haematocrylin.....	797
Havenschild, H. On the Manufacture of Hydraulic Mortar in France and Germany.....	1	And Smoluchowski, Th. v. Aurin.....	798
Haufl, J. Employment of Trinitroresorcin as Smokeless Powder (P).....	975	Hesekiel, A. Sensitised Paper for Photographic Purposes. (P).....	170
Hauser, G. C. F. See Reissert.....	973	Hess, C. See von der Linde.....	55
Hausermann, C. Occurrence of Perchlorate in Potassium Nitrate.....	947	Hess Storage Battery Co. Storage Batteries (P).....	1206
And Naschold, W. Electrolytic Production of Potassium Chlorate.....	885	Hesse, A. Treatment of Boiling (Foaming) Fermentations....	967
And Niehammer, F. Employment of Electrical Heat for Concentrating Sulphuric Acid.....	391	Hesse, O. Cinchonine.....	58
Hausermann, J. Sodium Salt of Perchromic Acid.....	286	Hydrocine.....	415
Hautefeuille, P., and Perrey, A. Contribution to the Study of Yeasts.....	654	Melting Point of Cocaine Hydrochloride.....	172
Hawdon, W. The Capacity and Form of Blast Furnaces.....	643	Parera Alkaloids.....	416
		The Atropine Alkaloids.....	416
		The Melting Points of Cocaine Hydrochloride, Cinchonine, &c.....	416
		Hetherington, A. E. See Brock.....	1195
		Heut, G. Concine and Nicotine.....	767



	PAGE		PAGE
Hewett, G. Electrolytes for Primary Batteries (P).....	820	Huguenn, D. E. See Durand.....	145, 146, 247, 802, 1055
Primary Galvanic Batteries and Liquids used therein (P).....	818	Hummel, J. J. Discussion on Determining Shade of Mazarin Pastes.....	496
Hewitt, P. C. Processes and Apparatus for Cooling and Forming Glue (P) (illus.).....	1075	Discussion on Dyeing Leather with Basic Colours.....	497
Removing Impurities, &c. from Glue (P).....	1075	Discussion on Indian Dyestuffs.....	553
Heyden, Nachfolger, F. von. Manufacture of Guaiacol (P).....	275	Discussion on Mordanting Wool with Chromium.....	567
Producing Esters from Guaiacol, Crocote or Crovon (P).....	275	Discussion on the Theory of Dyeing.....	555
Heyes, W. E. Discussion on Analysis of India-Rubber Articles.....	485	See Liechti.....	222, 356
Heyward, W. Recovery of Zinc or Spelter from Waste (P) ..	401	See Perkin.....	210, 211, 240, 1054
Hickisson, J. Manufacture of Coloured Marking Inks and Pencils (P).....	246	And Cavallo, W. The Colouring Matter of the Indian Dyestuff "Tesh".....	144
Hepe, W. L. Some Products of Starch Formation.....	267	And others. Report on Action of Light on Dyed Colours, And Perkin, A. G. Theoretical Properties of some Indian Dyestuffs.....	503
Hill, A. J. See Barratt.....	1088	And Procter, H. R. Improvement in Dyeing Leather with Basic Colours.....	496
Hill, F. R., and Brett, R. D. Method and Apparatus for Burning Hydrocarbons (P) (illus.).....	139	Hunt, E. W. See Jackson.....	218
Hill, J. Using Recovered Precipitated Carbonate of Lime in Manufacture of Portland Cement (P).....	255	Hunter, T. G. Removing Tin from Scrap, &c. (P).....	102
Hilliard, J. B. Preventing Gases from Intermingling in Liquids (P).....	1155	Huntington, A. K. Manufacture of Compounds of Iron with Fatty Acids (P).....	745
Hilsons, E. See Lezé.....	1069	Hurter, F. Apparatus for Manufacture of Chlorate of Potash by Electrolysis (P).....	810
Hingston, R., and Wernegreen, J. P. Antifouling Composition (P).....	1216	Discussion on Aluminium Alloys.....	10
Hirschsohn, E. The Distinction of Birch from Pine Tar.....	423	Discussion on Lunge-Kohrmann Plate Towers.....	1036
Hjorth, B. A., and Co. Heating Apparatus for Blow-Lamps (P).....	789	Discussion on Methods for Estimation of Manganese.....	115
Hear, R. W. See Aspinall.....	1208	See Brock.....	359, 1195
Hodan, C. Production of Enamelled Letters and Designs on Glass, &c. (P).....	811	And others. Electrolytic Cells (P).....	558
Hodge, B. See Gooch.....	979	And Omholt, J. Manufacture of Monocalcic Phosphate and Ammonia (P).....	469
Hoepfner, C. Electrolytic Production of Nickel and other Metals (P).....	744	And Omholt, J. Manufacture of Soda and Monocalcic Phosphate (P).....	394
Hoffmann, E. Dyeing Wool with Chromium Lakes (P).....	884	Husser, A. Sterilising Apparatus (P).....	1215
Hoffmann, F. Estimation of Free Acid in Solutions of Persalts of Heavy Metals.....	282	Hutchinson, R. Treating Gutta-Percha and Rubber (P).....	619
Hofman, H. O., and Demond, C. D. Determination of Refractivity of Fireclays.....	638	Treating Wool Greases (P).....	260
Hogg, T. W. Influence of Aluminium on the Carbon in Ferro-Carbon Alloys (illus.).....	955	Hyatt, W. H. Manufacture of Paint (P).....	648
Holand, R. The Estimation of Paraffins.....	286		
Holbrook. Discussion on Spontaneous Combustion of Oils.....	1156	Iantsch, E. Printing on Cotton Fabrics (P).....	514
Holde, D. Detection of Disguising and Perfuming Media in Oils and Fats.....	906	Ibbotson, W. Coating Paper with Aluminium (P).....	179
Properties of Sunflower Oil.....	892	Iles, M. W. The Curdling of Milk.....	538
Solubility Tests of Dark Mineral Lubricating Oils.....	668	Illingworth, C. R. A Superfatted Biniodide of Mercury Soap (P).....	531
The Analysis of India-Rubber Goods.....	425	Ilosva, L. L. de N. Estimation of Ammonia by Means of the Colorimeter.....	842
Holfert, J. Action of Formaldehyde on Vegetable Tissues.....	909	Estimation of Secondary Products containing Nitrogen formed during Combustion in Air.....	792
Holken, E., and Co. Dyeing Vegetable Yarns in Cops, &c. (P).....	391	Hydrogen Dioxide in Atmospheric Air and in Aqueous Deposits therefrom.....	755
Holland, G. Improved Cattle Food (P).....	168	Ingham, W. P. Construction of Blast-Furnaces, Shafts, &c. (P).....	625
Holleman, A. F. Volumetric Method of Estimating Phosphoric Acid.....	763, 843	Inston, A. J. Recovering Grease from Waste Suds (P).....	1070
Holler, A. See Ulzer.....	799	Irvine, R. Address to Scottish Section.....	1058
Holliday, R. See Read.....	511	Discussion on Electric Furnace for Laboratory Work.....	375
Hollrung. Phoma Betæ, the Beetroot-destroying Organism.....	264	Discussion on Paraffin Lamp for Laboratories.....	1167
Holt, W., and Sims, W. E. The Oxidation of the Alkali Metals.....	526	Discussion on Preparation of Carbon Blacks.....	131
Homatsch, A. See Sattmann.....	401	Discussion on the Oxidation of Aniline.....	238
Hood, J. J. See Albright.....	517	Manufacture of Lamp-black (P).....	582
And Salamon, A. G. Preparation of Cyanogen Compounds (P).....	1195	Note on Cabot's Paper on Preparation of Carbon Blacks.....	131
Hooper, E. F. Discussion on Colorimetric Analysis.....	318	Paraffin Lamp with Smokeless Flame (illus.).....	1167
Hooper, E. G. Discussion on Sterilisation of Liquids.....	1136	Proceedings of Annual General Meeting.....	700
Hope, A. P. Disinfection and Purification of Sewage, &c. (P).....	831	Speech at Annual Dinner.....	705
Horn, A. P. Furniture Polish (P).....	893	Vote of Thanks to Mr. C. A. Fawcett.....	128
Horn, W. J. Discussion on Oxidised Linseed Oil.....	1022	And Woodhead, G. Sims. On the Presence in Paper of Residual Chemicals.....	131
Hornby, J. The Gas Engineer's Laboratory Handbook.....	429	Ishii, N. Filters used in Manufacture of Alcohols, Oils, &c. (P).....	377
Horne, W. D. Automatic Extractor for Phosphates.....	278	Ivanoff, M. Disinfection of Town Sewage with Sulphuric Acid.....	537
Horton, W. H. Dry Soap, or Soap Powder (P).....	746	Ives. Discussion on Oxidised Linseed Oil.....	1022
Hotter, E. Estimating Pentosans in Vegetable Substances.....	424	Ives, E. C. Tanks for Precipitation of Sewage, &c. (P) (illus.).....	832
Hough, A. Plates for Secondary Batteries (P).....	14	Izarn. Novel Application of Bichromated Gelatin.....	1166
And March, O. Secondary Battery Plates or Elements (P).....	161		
Houllevigne, L. On the Electric Transference of Heat.....	72	J	
House, H. A., and others. Apparatus for Refining Metal by Electrolysis (P).....	1068	Jaccard, M. See Chuard.....	969
Howard, C. Vulcanising and Drying Wood (P).....	1063	Jackson, C. L., and Hunt, E. W. Open Soapings, &c., Machines for Textiles (P).....	248
Howard, D. Discussion on Colorimetric Analysis.....	317	Jackson, J. R. Poisonous Poddler Plants.....	897
Discussion on Dissolved Organic Matter in River Waters.....	321	Jackson, W. L. Softening and Cleansing Hides (P).....	1075
Discussion on Sodium Peroxide Explosion.....	199	Jackson, W. M. See Manchester Oxygen Co.....	2, 715, 1189
Discussion on Sterilisation of Liquids.....	1137	Jacob, H. Process and Apparatus for Imparting a Silky Appearance to Yarns (P).....	944
Proceedings of Annual General Meeting.....	692, 693	Jacobi. See Wolf.....	12
Speech at Annual Dinner.....	703	Jacobsen, Emil. Chemisch-Technisches Repertorium 182, 186, 1232	
Howarth, R. Manufacture of Metallic Alloys or Compounds (P).....	815	Jaeschin, H. Respiration of Malt on the Malting Floor (illus.).....	1078
Hübner, E. Manufacture of White Sugar from Molasses, and Centrifugal Apparatus therefor (P).....	824	Jaffe, B., and Darmstaedter. Distillation of Substances having a High Boiling-Point (P).....	259
Hughes, J. Discussion on Testing Hydraulic Cements.....	163	Jahn, E. Analgenc (Ortho-Ethoxy-Ara-Mono-Benzoylamido-Quinoline).....	1138
Hughes, W. H. and S. F. Scouring, Bleaching, and Disinfecting Wool and other Fibres (P).....	728		



	PAGE		PAGE
Koninek, L. L. New Method of Performing Schlosing's Process for Estimating Nitric Acid (illus.)	763	Lambert, G. Klem's Method of Compressing and Shaping Electro-Metals	1200
Kopp, K. The Glossing of Woollen Crapes	1193	Lange, H. Production of Mame-colored Fabrics by Mordanting the Yarn	387
And Grandmoulin, E. Acetine	720	Lange, E. Refining Sugar (P)	533
And others. Report on Bonnet's New Dyeing Process	726	Langebeck, K. Pottery Industries of the United States Chemically Considered	153
Koroff, R. Manufacture of New Amido-Bases and Azo-Colouring Matters therefrom (P)	652	Langhans, R. Manufacture of Insect-scented Bodies (P)	141
Kosmann, B. The Composition of Portland Cement	386	Langley, J. W. Use of Electrolysis in Technical Chemical Processes	615
Kostanecki, St. v. Chrysin	509	Langeville, L. S. Treating Black Ash Residue of Wood-Pulp Making (P)	179
And Tambor, J. Synthesis of Gentisin	795	Laszarynski, S. von. Solubility of Inorganic Salts in Organic Liquids	1195
Kothe, R. See Dressel	875	Latache, C. Actinometric Pyrometer (P) (illus.)	663
Kowalski, H. Apparatus for Filtering and Sterilising Water (P) (illus.)	1084	Latham, E., and Guyot, E. G. Apparatus for Evaporating Liquids (P)	713
Kozal. See Kellner	969	Lauder, A. See Dobbie	1217, 1217
Kraft, F., and Roos, A. Manufacture of Esters, Phenol, and other Ethers (P)	759	Laurie, A. P. Electromotive Force of Alloys in a Voltaic Cell. Preparation of Oxide of Zinc Pigments (P)	1201, 262
And Stern, A. Behaviour of Alkaline Salts of Fatty Acids and of Soaps in Presence of Water	1207	Laub, C. Derivatives of the Oxazine and Eupholine Series. The Carboxyl Derivatives of Dimethylaniline	628, 244, 629
Krause, L., and Mayer, J. Separation of Cholesterol and Fatty Acids from Wool Fats (P)	162	Lavecat, L. E. A., and Candiot, E. L. Cement Kilns (P)	1062
Kreihagauer, A. Quantitative Electrolytic Estimation of Lead	547	Lavy, A. Distilling Apparatus (P)	751
Kriens, C. W. Manufacture of Animal Charcoal (P)	1075	Lawrence, W. Discussion on Sterilisation of Liquids	1191
And Harper, J. R. Recovering Tin from Plates or Waste (P)	641	Lazarus, J., and Jefferson, M. Machines for Washing Fibres (P)	245
Krocker, Estimation of Sugar in Beetroots	823	Lee, W. E. Protecting Gas Cylinders during Transit (P)	1189
Kruger, M. Determination of Nitrogen in Benzene, &c., by Kjeldahl's Method	425	Leaker, R. H. Method and Apparatus for Making Vinegar and Maturing Spirits (P)	635
Determination of Nitrogen in Nitrates, &c.	1226	Lebl, H., and Atlass, E. Treatment of Leather (P)	51
Kruger, P. See Tiemann	272	Le Blanc, M. On the Minimum E. M. F. required for the Decomposition of Electrolytes	742
Krüss, H. A Colorimeter with Lummer-Brodhun Prism System (illus.)	418	Le Brocqny, L. Substitute for India-Rubber and Gutta-Percha (P)	964
Kubin, E. Improvements in Explosives (P)	660	Lecco, M. T. The Use of Lead Pipes for Conveying Water	547
Kühler and Buff. Manufacture of Amido-alkylsalicylic Acid and Acetamidalkylsalicylic Acid (P)	60	Le Chatelier, H. Fusibility of Mixtures of Isomorphous Salts. Manufacture of Oxygen	634, 27
Kuhn, E. W. The Rational Sterilisation of Alimentary Liquids	1133	Le Chatelier, L. Manganese Steel	1198
Kukla. Employment of Sulphurous Acid in Malting	968	Ledebur, A. Examination of Ordinary Methods for Estimation of Carbon in Iron	281
Influence of Ferruginous Water in Malting	1080	Lederer, L. Manufacture of Yeast and Distillation of Spirits (P)	827
Kuliseh, P. Detection of Boric Acid in Ash of Wine, &c.	294	Leechman, W. C. Plates for Oil- and other Presses (P)	377
Kumagaya, N. A Material for Substituting Leather and Ordinary Paper (P)	754	Leeds, F. H. Notes on Lithographie Varnish	203
Kunkler, A. A Viscosimeter for Lubricating Oils (illus.)	543	Leemann, J. Apparatus for Illuminating and Heating by Petroleum (P)	141
Kunwald, A. de. See Cordewener	279	Leent, F. H. van. See Lobry de Bruyn	963
Kunze, W. E. Estimation and Separation of the Cacao-Alkaloids	378	Leffler, C. J. L. Manufacture of Ferro-Bronze	1204
Kurbatow, A. Extraction Apparatus for Liquids (illus.)	978	Léger, E. See Jungfleisch	58, 637
Küster, F. W. Acidimetric Estimation of Naphthalene Acenaphthene and $\alpha$ - and $\beta$ -Naphthol	514	Lehmann, A. Electrode Plates for Secondary Batteries (P)	1206
Sulphuretted Hydrogen Apparatus (illus.)	278	Lehmann, N. Extraction Mill (illus.)	959
Kyle. Vanadiferous Lignite found in Argentina, and Analysis of the Ash	738	Lehming, R. Burners for Hydrocarbon Oils (P)	793
		Lelme, A. Tabellarische Uebersicht über die Künstlichen Organischen Farbstoffe, &c.	73, 429, 768, 1231
L		Lehner, F. Method and Apparatus for Producing Artificial Silk (P)	1192
Labhardt, E. Apparatus for Dyeing and Washing Fabrics (P)	391	Lencauchez, J. A. Manufacture of Steel in the Open Hearth	933
Laborde. See Grimaux	1083	Lenoble, E. Corrections to be Applied in Titrating Liquids containing Suspended Matter	762
Laeb, B., and Benies, H. Purifying and Decolorisation of Saccharine Juices and Solutions (P)	824	Lensi, M. Influence of Alcohol, Glycerol, and Fatty Acids on Bactericidal Action of Phenol and Corrosive Sublimates	963
Laeroix-Hunkibéyendian. Cresol as an Antiseptic	273	Leht, K. A New Accumulator	158
Lacy, B. T., and Storer, J. Process and Apparatus for Dissolving, Leaching, and Filtering Ores (P)	741	Leht, W. The Employment of "Micro-Chemical" Reagents in Analytical Chemistry	766
Ladd, J. H. Treating Malleable or Wrought Iron (P)	527	Leonard, M. E. An Improved Gunpowder (P)	61
Ladenburg, A. Pure <i>d</i> -Conine	756	Leonard, N. Detection of Nitronaphthalene in Mineral Oils	69
Laer, H. van. The Action of Oxygen on Yeast	1078	Leonhardt, A., and Co. Manufacture of Colouring Matters (P)	722, 724
Lafont, J. See Bouchardat	658	Lepetit, R. Hard Glass Beakers for Dyeing Tests	978
Lagarde. Note on Gutta-Percha	746	Le Roy, G. A. Estimation of Boric Acid in Boronatrocalcites	1094
Lagneau, E. Manufacture of Matches (P)	61	Lesinsky, J. See Jannasch	67, 548
Lagnesse, J. V. Apparatus for Galvanising Sheet Metal (P)	158	Lettenmayer, L. The Oxygen Absorption, Acid and Iodine Numbers of Various Drying Oils	258
Lagodzinski, K. $\beta$ -Anthraquinone	876	The Properties and Employment of Essential Oils	273
Laird, R. H. and W. H. Apparatus for Vaporising Oil (P)	873	Leuchs, G., and C. Manufacture of Glazes, Enamels, &c. (P)	519
Laire, G. de, and Tiemann, F. Iridin, the Glucoside of the Iris Root	272	Levat, L. A. The Improvement of Oils by Electrical Treatment	258
Lajoux, H., and Grandval, A. Mercurial Salicylates	58	Levinstein, I. Apparatus for Concentration of Sulphuric Acid (P)	152
Lake, H. H. Burning Sulphur for Disinfecting Purposes (P). Manufacture of Food for Horses (P)	657, 752	Discussion on Modern Systems of Sewage Treatment	608
Lambert, A. Lead Pipes in Vitriol Manufacture	733	Discussion on Oxidation and Corrosion of Iron and Steel	129
Lambert, H. See Greenwood and Batley	496	Iron Compounds for Treating Sewage, &c. (P)	1216
Lambilly, P. R. de. Production of Ammonium Formate (P)	35	Method and Apparatus for Recovering and Concentrating Waste and Dilute Acids (P)	152
Land, C. H., and Caulkins, H. J. Focussing or Controlling Flame or Heat (P)	937	On Behring's Antitoxin Experiments	1142
Landshoff and Meyer. Recovery of Iron from the Ferruginous Residue of Amine Bases (P)	42	Presentation to Mr. J. Carter Bell	607
Lane, H., and Pullman, J. Method and Apparatus for Producing Carbonic Acid Gas (P)	510	Remarks on Mr. Carter Bell's Paper on Flash Point of Mineral Oils	116
Lang, L. Improvements in Calico Printing (P)	150		

	PAGE
Levy, L., and S. Preparation for Cleansing Vulcanite (P).....	1072
Levy, M., and others. Manufacture of Artificial Whalebone and Apparatus therefor (P).....	1073
Lewes, V. B. Action of Heat upon Ethylene.....	565
Lewis, I. H. Process and Compound for Waterproofing Shoe- soles (P).....	552
Waterproofing Leather (P).....	552
Water-proofing Leather, and Compounds therefor (P).....	552
Lewkowitsch, J. Discussion on Action of Antiseptics on Yeast.....	1161
Discussion on Spontaneous Combustion of Oils.....	1166
Discussion on Substantive Dyes and Dyeing.....	125
Soaps most Useful for Scouring Wooden Goods.....	258
The Analysis of Fats. Part IV. Colour Reactions.....	617
Lévy, J. D. Composition of Sugar during Evaporation.....	116
Evaporation and Boiling of Sugar Liquor.....	822
Leybold, W. Difficulties in Working Parallel Series of Gas Apparatus.....	1049
Leitz, R., and Hilsenrath, E. Testing Milk with Rennet.....	1699
Licht, E. F. Machine for Dyeing Hats, &c. (P).....	391
Lieber, A. Determination of Formic Acid.....	288
Liebermann, C. On Eulhorn and Willstätter's Paper on Cocaine.....	1218
The Theory of Mordant Dyes.....	28
Liechti, L., and Hummel, J. J. On the Mordanting of Wool with Chromium.....	222, 356
Liedtke, C. Purification of Diffusion Juice.....	749
Lienard, L. A. P., and H. E. A. The Electrolysis of Saline Solutions (P).....	818
Linde, C. Refrigerating Apparatus (illus.).....	502
Linder, H. von der, and Hess, C. Purifying Water by Oxide of Tin (P).....	56
Linder, O. Examination of Liquid Extracts.....	969
Linden, O. Centrifugal Separators (P).....	871
Lindet, L. Development and Ripening of the Cider Apple.... Influence of Acidity in Worts on Composition of "Low- Wines".....	54
Formation of Cane-Sugar during Germination of Barley, and during Ripening of the Apple.....	750
Production of Saccharose during Germination of Barley....	265
Lang, A. R. Manufacture of Anhydro-ortho-Sulphamine Ben- zoic Acid or its Salts (P).....	836
Linnemann, F. An Improved Fabric for Garments (P).....	802
Lintner, C. J., and Benzenr, A. The Chemistry of Hops.... And Dull, G. Degradation of Starch by the Action of Diastase.....	52
53	
Lishman, W. W. L. See Clapham.....	390, 728
Lisley, P. See Vignon.....	32
Lissenko, K., and Stepanow, A. The Alkali Test for Kerosine, Naphtha Products, &c.....	177
Lithaude and Gen. Electric Co., and Niblett, J. T. Elements for Secondary Batteries (P).....	256, 744
Secondary Electric Batteries (P).....	818
Liveng, E. H. Preparation of Carbon Electrodes (P).....	161
Liversidge, A. The Condition of Gold in Quartz and Calcite Veins.....	398
The Origin of Gold Nuggets.....	398
Lobry de Bruyn, C. A. Analysis of Mixtures of Sperm Oil and Mineral Oil.....	426
Connection between Tenacity of and Moisture present in Hemp and Flax.....	385
Mineral Oils on Ships in the Tropics.....	237
Valuation of India-Rubber Goods.....	986
Lochatin, P. Fire-proofing Compounds.....	512
Loekhart, W. S. Process and Means for Disintegration of Minerals (P).....	889
Lockwood, H. Tanks for Subsidence of Sewage Sludge (P)....	970
Lomes, J. See Walker, jun.....	1206
Long, C., and Burkitt, W. H. Raisers for Acids, Alkalis, and Liquids (P) (illus.).....	930
Loft, E. New Constituents of Wood-Oil.....	1188
Lossen, C. Treating Auriferous Ores with Bromine.....	1198
Lotherington, C. B., and Spencer, I. Furnace Pan and Con- centrator for Baling Fish Livers, &c. (P).....	162
Louis, H. A Handbook of Gold Milling.....	429
An Improved Specific Gravity Bottle (illus.).....	522
Discussion on the Treatment of Gold Ore in the Transvaal.....	339
Love, J., and Weston, D. Apparatus for Carburetted Gas or Air (P).....	141
Loveland, J. W. Colorimetric Analysis (illus.).....	308
Discussion on Colorimetric Analysis.....	318
Low, A. H. The Technical Estimation of Lead.....	250
Indhoff, P. Centrifugal Machines for Liquids (P) (illus.).....	210
Ludy, F. Investigation of the Resins: The Gum Benzoin of Siam.....	821
Lugo, O. See Van der Weyde.....	265
Lührmann, F. W. Manufacture of Artificial Building Materials (P).....	42
Lumière, A. and L. Photographic Properties of Vanadium Salts.....	100

	PAGE
Lundström, C. J., and others. Centrifugal Liquid Separators (P) (illus.).....	24
Lundy, J. J. See Wilson .....	746
Lunge, G. Discussion on Lunge-Rohrmann Plate Towers ....	1037
On the Manufacture of Sulphuric Acid in America (illus.) ..	884
Practical Results of the Lunge-Rohrmann Plate Towers ....	1034
The Manufacture of Water-Gas in New York .....	872
Titration of Aniline and other Organic Bases. ....	66
And Biehofen, F. The Specific Gravities of Solutions of Bleaching Powder .....	367
And Maxwell-Lyte, F. Producing Basic Lead Salts and By-Products (P) .....	734
And Maxwell-Lyte. Production of Nitric Acid, Caustic Alkali, and Ferric Oxide (P) .....	734
See Maxwell-Lyte .....	734
Lunewitz, E. See Schweitzer .....	614, 709, 878, 888, 1178
Lusby, E. J. Manufacture or Treatment of Paper (P).....	902
Lynchenheim, J. Determination of Phosphorus in Coal and Coke .....	422
Lynen, L. V. T. Process and Apparatus for Extraction of Zinc (P) .....	402
Lyon, D. H. Separating Impurities from Feed-Water (P) ....	377
Lyte, C. H. M. Production of Iron Oxide for Use as Pigment (P) .....	962
Lyte, F. Maxwell. Electrolytical Decomposition of Chlorides and Apparatus therefor (P) .....	958
Electrolytical Decomposition of Chlorides and Mixtures thereof (P) .....	404
Production of Chlorine and Refined Lead, and Recovery of Silver (P) (illus.) .....	644
And Lunge, G. Production of Caustic Alkali and Chloride of Lead (P) .....	734
See Lunge .....	734

## M

Maag, J. Apparatus for Sizing Yarn (P) .....	880
Malbery, C. F. Examination of Canadian Sulphur-Petroleum .	507
And Smith, A. W. The Sulphur Compounds in Ohio Petroleum .....	507
Macadam, I. Speech at Annual Dinner .....	704
Macadam, S. Proceedings at Annual General Meeting ...	698, 694
Macadam, W. I. Processes for Manufacturing Gas from Oil ...	378
MacArthur, J. S. Discussion on Treatment of Gold Ore in the Transvaal .....	338
And others. Extracting Gold and Silver from Ores (P) ..	526
Macdonald, A. Manufacture of Sulphate of Lead Pigment (P)	803
Treatment of Galena for Production of Lead, &c. (P) ....	401
Macdonnell, A. Alloys for Use in Manufacture of Wire (P)...	526
Macer, M. Apparatus for Raising Liquids (P) (illus.) .....	23
Macfarlane, W. See Addie .....	635
See Mills .....	237
Macfalski, F. J. Apparatus for Extracting Fat and Glue (P).	648
MacIlwaine, A. W., and Hardingham, G. G. M. Extraction of Oil by Volatile Solvents, and Apparatus therefor (P) ...	745
Macindoe, G. W. Discussion on Sterilisation of Liquids .....	1136
Mackenzie, W. C. Nitrate of Soda in Egypt .....	611
Mackey, W. McD. Discussion on Spontaneous Combustion of Oils .....	1166
Spontaneous Combustion of Oils Spread on Cotton .....	1164
Macnab, J., and Dickson, J. Charging Vessels with Volatile Liquids and Sealing Same (P) .....	920
Improvements in Blasting and Blasting Cartridges (P) ...	769
Macnab, W. Discussion on Smokeless Powder .....	583
And Ristori, E. Researches on Modern Explosives .....	978
MacPhail, M. Calico-printing Machinery (P) .....	1194
Madsen, H. P. Food Preparations (P) .....	1215
Magerstedt, O. Manufacture of Photographic Plates (P) ....	276
Mahlke, A. Mercury Thermometer for High Temperatures...	62
Mahon, R. W. Effect of Platinum in Iron Solutions .....	548
Modification of Stannous Chloride Method for Titration of Iron .....	282
Slag Cement Experiments .....	736
Mai, J. See Janonasz .....	176
Maiche, L. and C. Purification of Water and Apparatus therefor (P) .....	831
Maichen, P. A. Use of Asbestos for Filtration of Sugar Juices	264
Maquenne, M. Composition of Lime-Tree Honey .....	52
Malard, G. Process and Apparatus for Scouring and Degreasing Wool (P) .....	880
Malbot, H. and A. The Formation of Mannite in Wines .....	750
Mallet, P. Commercial Position of Bye-Products of Gas Manu- facture .....	719
Mallman, W. Manufacture of Artificial Musk (P) .....	659
Manchester Oxygen Co. and Jackson, W. M. Gas Cylinder Coupling (P) (illus.) .....	22
Utilising the Pressure in Gas Cylinders in the Employment of the Gases (P) .....	715
Utilising the Pressure of Compressed Gases (P) (illus.) ...	1180

	PAGE		PAGE
Manger, A. W. See Boulton	822	McTear, G. Manufacture of Ethyl or other Vegetable Alcohols	100
Mangold, C. Analysis of Sealing Wax	1100	Roading (P)	747
Mangoldt, G. A. Method of Setting up Vessels for Boiling	239	Meacham, C. S. See Brant	31
Mann, F. W. Improvements in the Paraffin and Mineral Oil Industries	875	Medina Santurio, R. See Prad	841
Mann, H. H. Action of Antiseptic Substances on Yeast	1460	Meyer, E. See Sachs	602
Discussion on Action of Antiseptics on Yeast	1064	Meikle, J. Apparatus for Extracting Solubilities by Volatile Solvents (P)	632
Mansfield, E. Manufacture of Gas (P)	874	Meillère, Estimation of the Insoluble N. A. in the Fatty Acids	584
Mar, P. W. Estimation of Barium in Presence of Calcium and Magnesium	282	Meinke, C. Sodium Thiosulphate for Bleaching	420
Mareel, A. Utilisation of Waste Carbonic Acid Gas in Breweries (illus.)	825	Meissels, K. See Kuhn	812, 905
Mareh, O. Apparatus for Artificial Production of Chalybeate Water (P)	529	Meissner, J. M. An Heating Composition for Matches (P)	437
See Hough	161	Meldola, R. See Hummel	864
Marchlewski, L. Critical Studies of Methods for Estimation of Sulphur	284	Meldrum, J. J. and T. E. Gas Producers (P) (illus.)	21
See Schunck	509, 633	Purification of Sewage, &c. (P)	103
Murie, T. The Extraction of the Free Acids of Beeswax	1207	Melchior, M. Advantages of Heating Diffusion Jugs with Steam from another Diffusion	673
Marnio, P. Plating Surfaces by Electro-Chemical Deposition (P)	958	Mellinger, J. Soap for Removing Hair from the Skin (P)	406
Maris, E. Apparatus for Treatment of Alimentary Substances (P)	808	Melmon, Separation and Estimation of Tin and Antimony in Alloys	1224
Markownikoff, W., and Reformatsky, A. Bulgarian Oil of Rosin	272, 758	Menyaza, G. P. Behaviour of Strychnine towards Vital's Atropine Reaction	181
Marr, J. See Wagner	751	Menzies, W. J. Treatment of Manganates for Production of Permanganates (P)	38
Marshall, Discussion on Electric Furnace for Laboratory Work	375	Meran, J. E. G. Manufacture of Pottery (P)	1197
Discussion on Preparation of Carbon Blacks	141	Meerk, E. Champaol	834
Marshall, P. D. Apparatus for Washing and Purifying Gas (P) (illus.)	716	Mering, J. F. Freiherr von. Production of Acetyl and Propionyl Compounds of Parahydroxyphenylamines or their Ethers (P)	60
Marshall, T. R. Discussion on Production of Oil-Gas	501	Meslans, M. Apparatus and Method for Determining Density of Gases	64
Martin, F. See Storer	846	See Freres	378
Martin, Freund, and Frankforter. Investigations on Narcotine	58	Metzner, R. See Dittie	43
Martin, J. W. Making Glazed Bricks and Tiles, and Glazing Walls, &c. (P)	40	Meunier, S. Constitution of the Rock Matrix of Platinum	639
Martindale, W. Analyses of Twelve Thousand Prescriptions	768	Meyer, See Landshoff	42
Martyn, W. Discussion on Lunge-Rohrman Plate Towers	1037	Meyer, E. Estimation of Sulphur in Recovered Gas-Purifying Material (illus.)	283
Mason, A. H. Address to New York Section	1167	Method and Apparatus for Analysis of Zinc Dust (illus.)	1225
Mason, Consul-General. Electrical Smelting	741	Testing Zinc Ores and Products	1225
Masson. See Balland	169	Meyer, L. Aluminium Drying Oven (illus.)	1220
Matheson, W. J. Making Solid and Friable Colouring Matters (P)	1190	The Explosive Nature of Acetylene	1482
Mattei, D. Process and Apparatus for Dyeing Textiles (P)	728	Meyer, L. L. B. Manufacture of Varnish, and a Substance therefor (P)	1200
Matthews. Discussion on Testing Hydraulic Cements	462	Meyer, R. Jahrbuch der Chemie	846
Matthey, E. The Liquefaction of Silver-Copper Alloys	889	Meyer, T. Potassium Phosphate as Manure	469
Mayall, C. Apparatus for Shaking Bottles (illus.)	1220	Meyer, V. See Altschul	415
Maury, L. Detection of Castor Oil in Copaiba Balsam	981	Michael, K., and Frank, M. Method and Apparatus for Collecting Carbonic Acid during Fermentation (P)	655
Maurer, H. Manufacture of Porous Bricks (P)	812	Michaelis and Co. Dyeing Textiles (P)	631
Maurette, A. An Explosive Composition (P)	690	Michel, O., and Grandmaugin, E. The Action of Ammonia Gas on Peroxides	57
Maxim, H. Explosives (P)	1219	Mieck, C. Determination of Malic Acid in Wines	286
May, P. W., and Newlands, B. E. R. Apparatus for Sterilising or Preventing Fermentation or Acetification of Beers, &c. (P)	751	Mischel, J. B. Extraction of Gold and Silver from Ores and Matte (P)	1067
Mayer, J. See Krause	162	Miller, T. P. Apparatus for Treating Fibrous Materials (P)	884
Mayer, J. A. Process and Apparatus for Separating Metals, &c. (P)	740	Miller, W. von, and Rohde, G. The Colouring Matter of Cochineal, and others. Compounds of Hydrogen Cyanide with the Amides of the Sugars	142
Separating and Purifying Metals (P)	815	Milliau, E. The Methods of Testing Fats and Oils	181
McCombie, C. Phosphor Copper and Bronze	1139	Mills, E. J. Discussion on the Oxidation of Aniline	238
McConnell, W. Gases Enclosed in Coal and Coal-Dust	25	And Macfarlane, W. On the Oxidation of Aniline (illus.)	257
McDougall, A. Covering Lead Surfaces to Protect Water from Contamination (P)	893	And Watson, G., jun. On the Oxidation of Aniline	258
McDougall, I. S., and J. T. Disinfectants (P)	657	Mills, W. Metallurgy of Metallic Sulphates (P)	74
Fumigants (P)	833	Milnes, J. M., and A. Apparatus for Manufacture of Bleaching Powder (P)	1050
Production of Insecticides (P)	833	Minor, W. Preparing Lead Peroxide for the Anilin Industry	910
McDougall, J. S. Oil Separator for Use with Feed-Water Apparatus (P) (illus.)	22	Miolati, A. Paracrosaniline and its Salts	143
McDougall, W. S., and J. T. Fumigants (P)	657	Mitchell, C. R. Note on Estimation of Iron	104
Insecticides and Fungoid Exterminators (P)	657	Mitscherlich, A. Utilising Lyes of Sulphite Cellulose (P)	824
McElroy, K. P. Detection of Salicylic Acid in Food	670	Mittelmann. Defecation and Filtration of Diffusion Liquor	965
And Bigelow, W. D. Detection of Eosin in Coloured Tomatoes	284	Defecation of Diffusion Juice with Pulverised Slaked Lime	10
McIlhenny, P. C. Analysing Fats and Resins	608	Purification of Juice in Triple-Effect Apparatus	166
The Iodine Number of Rosin	608	Use of Sulphurous Acid in Diffusion	145
McLean, A., and Wilson, C. W. Manufacture of Artificial Stone (P)	888	Mixer and Dubets. Determination of Iron in Iron Ore	507
McLeod, H. Note on Liberation of Chlorine	272	Moeller, J. Incandescent Gas Burners (P)	247
McMeekin, A. Method and Means for Bleaching, Dyeing, &c. Fibres (P)	249	Manufacture of Oil Gas (P)	379
McMillan, W. G. Colour Gauzes for Carbon Testing	978	Note on Ipecauanha	900
Discussion on Strength of Leather	592	Moeller, K. Filter for Air, Gas, Steam, &c. (P)	714
Discussion on the Colour of Brass	595	Mohrdieck, P. See Dubers	148
Experiments on the Strength of Leather (illus.)	585	Mohsan, H. Impurities of Commercial Aluminium	1199
Note on the Colour of Brass	596	Preparation of Crystalline Carbide of Aluminium	1199
McMurtrie, J. McL. Apparatus for Distribution and Burning of Fluid Hydrocarbons (P) (illus.)	937	Moisset, J. C. See de Fursac	400
McNaught, J. and W. Machinery for Scouring and Washing Wool and other Fibres (P)	803	Moldenhauer, C. Precipitating Precious Metals in Solution (P)	825
McRoberts, J. G. Steel Founding (P)	645	Molyneux, P., and Potts, G. R. Disinfectants (P)	413

	PAGE		PAGE
Wheeler, J. A. and Beard, M. B. A New Source of Rhodine.....	678	Nagel, J. Apparatus for Distilling and Sterilising Water (P) (illus.).....	623
Wheeler, J. C. Extraction of Gold and Silver from Ores by Acids (P).....	656	Apparatus for Obtaining Distilled and Sterilised Water (P).....	169
Wheeler, J. S. T. ....	100	Nangle, W. C. Paint or Paint-like Composition (P).....	648
Wheeler, J. S. T. ....	284	Naschold, W. See Hausserrmann.....	885
Wheeler, J. S. W. ....	147	Naylor, W. Comparison of Modern Systems of Sewage Treatment.....	340
Wheeler, J. S. W. Treating Fabrics to Obtain a Substitute for Patent Leather, Tapestry, Brocade, &c. (P).....	31	Discussion on some Modern Systems of Sewage Treatment.....	609
Wheeler, J. S. W. Crushing Apparatus (P).....	623	Neill, F. A. R. See Rigby.....	638, 948
Wheeler, J. S. W. Improved Food (P).....	898	Neitzel, C. Camphor as a Reagent for Sugar.....	843
Wheeler, J. S. W. Tubular Scheme for Iron Ore Analysis.....	1623	Neitzel, E. Quantitative Colorimetric Estimation of Carbohydrates.....	285
Wheeler, J. S. W. Apparatus for Heating or Evaporating Liquids (P).....	622	Netter, See Wolf.....	42
Wheeler, J. S. W. Portable Primary Batteries (P).....	1067	Nettelton, J. A. The Flavour of Whisky as Influenced by the Materials of its Manufacture.....	651
Wheeler, H. F. R. See Muir.....	989	Neubauer, H. Reliability of Magnesium Pyrophosphate for Estimating Phosphoric Acid.....	1092
Wheeler, J. A. and Frommery, J. Apparatus for Evaporating Liquids or Saccharine Solutions (P).....	377	Neumann, G. Qualitative Detection of Aluminium.....	840
Wheeler, J. A. and Emy, W. Apparatus for Distilling Spirits and Liquids (P) (illus.).....	504	Neumann, M. See Salberg.....	832
Wheeler, J. A. and Harris, Discussion on Sterilisation of Liquids.....	1147	Newbury, S. B. Report on Chemical and Pharmaceutical Products at the Paris Universal Exposition.....	280
Wheeler, J. H. See Hanson.....	119	Newlands, B. E. R. Discussion on Colorimetric Analysis.....	317
Wheeler, F. W. Oven for Drying in Hydrogen (illus.).....	174	Discussion on Composition of Alloys.....	1019, 1020
Wheeler, H. The Pneumatic Pyrometer (illus.).....	869	Discussion on Oxidised Linseed Oil.....	1023
Wheeler, A. Precipitating Solid Animal, Mineral, and Vegetable Matter contained in Sewage Water (P).....	900	Discussion on Sterilisation of Liquids.....	1136, 1137
Wheeler, S. L. and others. Cleansing Composition (P).....	153	See May.....	751
Wheeler, C. A. and Prior, J. Preparation and Employment of Cerium (P).....	412	Newton, O. T. Hot Air Evaporating and Drying Machine (P).....	751
Wheeler, C. A. and others. Containing Vanadium.....	280	Niblett, J. T. See Lithanode Co.....	256, 744, 813
Wheeler, C. A. and others. Zinc Smelting at the Bertha Works, Virginia.....	41	Nicholson. Discussion on Rectification of Oil of Vitriol.....	211
Wheeler, C. A. and others. Diffusion in Vacuo by Heckmann's Process.....	1210	Nichols, W. W. R., W., and F. H. System and Apparatus for Refining China Clay (P).....	251
Wheeler, C. A. and others. Manufacture of Yellow and Orange Colouring Matters (P).....	632	Nickel, E. Detection of Mineral Acids in Presence of Organic Acids.....	423
Wheeler, C. A. and others. Silicon Carbide ("Carborundum").....	462	New Colour Reaction of Iridol.....	926
Wheeler, C. A. and others. The Crude Petroleum in Trenton Limestone.....	1073	The Colour Reaction of Hydrazine Sulphate with Lignin, &c.....	423
Wheeler, C. A. and others. The Production of Various Carbides.....	31	Nicolai, O. Flux for Soldering Aluminium and other Metals (P).....	896
Wheeler, J. Stanley. Speech at Annual Dinner.....	765	Solder for Aluminium and other Metals (P).....	643
Wheeler, M. M., Pattison, and Morley, H. Forster, Watts' Dictionary of Chemistry, Revised and Re-written Edition.....	989	Niederlander, T. Cadmium Yellow: Its Preparation and Properties.....	263
Wheeler, M. M., Pattison, and Morley, H. Forster, Watts' Apparatus for Obtaining Carbonic Acid (P) (illus.).....	636	Notes on Asphaltum.....	263
Wheeler, M. M., Pattison, and Morley, H. Forster, Watts' Method and Apparatus for Absorbing Carbonic Acid (P).....	152	Strontium Uranate.....	263
Wheeler, M. M., Pattison, and Morley, H. Forster, Watts' and Dubois, H. Production of Carbon Tetrachloride (P).....	972	Niemann, J. H. A New Meat Extract (P).....	537
Wheeler, M. M., Pattison, and E. O. Apparatus for Producing Pure Colours (P).....	800	Nienstaedt, E., and Goldmark, L. Improvements in Inks (P) and others. Preparation of Colouring Matters (P).....	648, 649
Wheeler, J. A. Optical Titration of Maminol.....	767	Niethammer, F. See Hansermann.....	38
Wheeler, K. Clarification of Beer (P).....	269	Niewerth, R. Electric Smelting and Furnaces therefor (P).....	160
Wheeler, P. T. The Multitration of Sugars.....	718	Manufacture of Carbon for Electrical Purposes (P).....	1200
Wheeler, J. H. Cleaning Preparation for Clothing (P).....	941	Nightingale, D. W., and Wicking, S. G. An Improved Fuel (P).....	141
Wheeler, J. H. Cotton-dyeing with Benzidine Colours.....	883	Nihoul, E. Gravimetric Methods for Estimating Sugars.....	843
Wheeler, J. H. Cotton-dyeing in Turkey-Red Dyeing.....	150	Nilson. See Wahl.....	806
Wheeler, J. H. The Electrolytic Reduction of Indigo.....	587	Nitzberg, J. See Polonovsky.....	143
Wheeler, A. Estimation of Starch by Alcoholic Fermentation.....	985	Noad, J. H. See Ferranti.....	533
Wheeler, A. Utilisation of Vantage Beers.....	1081	And Ferranti, S. Z. de. Manufacture of White Lead (P).....	107
Wheeler, J. J. Manufacture of Spirits and Apparatus therefor (P).....	751	Nobel, A. Artificial India-Rubber (P).....	107
Wheeler, Dr. J. Speech at Perth Banquet.....	716	Explosive Compounds (P).....	769
Wheeler, J. B. and Beard, M. B. Recovering Alkali from Waste (P).....	1074	Nobel, R. Method of Burning and Controlling Combustion of Solid Fuel, and Hearths therefor (P).....	87
Wheeler, K. S. See Brin's Oxygen.....	866	Noelting, E. Note on Abrastol.....	53
Wheeler, T. R. Pumps for Compressing Gases or Vapours (P) (illus.).....	377	See Kolp.....	72
Wheeler, J. and Dixon, G. Means for Superheating Steam (P).....	1048	Norden, E. Photographic Printing in Bromide of Silver (P).....	27
Wheeler, L. K. Discussion on Inventions.....	1026	Noriega, Eloy. Preparation of Reagents for Working Gold and Silver Ores (P).....	4
Wheeler, L. K. Discussion on Rectification of Oil of Vitriol.....	211	Normandy, A. and F. Apparatus for Heating, Evaporating, &c. (P).....	2
Wheeler, L. K. Apparatus for Manufacture of Bleaching Powder (P).....	38	North, E. G. N. Elimination and Recovery of Arsenic from Copper Precipitates (P).....	120
Wheeler, L. K. Estimation of Cadmium.....	211	Nourrisson, C. On the Minimum E.M.F. required for the Electrolysis of Dissolved Salts of Alkali.....	74
Wheeler, F. Detection of Sesame Oil in Olive Oil.....	423		
Wheeler, L. Causes of Difficulty of Sizing Paper in Hot Seasons.....	67		
Wheeler, L. Discoloration of Papers made from Sulphite Cellulose.....	75		
Wheeler, E. G. Determination of Phosphorus.....	67		
Wheeler, L. and Frommery, J. Separation of Metals from their Solutions.....	107		

N

Nagel, M. L. and Kinross, B. and Baschmakoff, S. d. Apparatus for Treatment of Sewage Matter (P).....

Naschold, W. See Kellner.....



	PAGE		PAGE
Oettel, F. Electrolysis of Alkaline Chlorides .....	403	Pearce, J., and O. G. W. B. Macdonald, C. S. (P) .....	21
The Ann of Electrical Chemical Investigations .....	417	Pearce, J. C. See Tremble .....	21
Oliphant, W. Filtering Apparatus (P) .....	1049	Peake, S. Apparatus for Dyeing Soap and Dyeing .....	1049
Oliveri, V. Adulteration of Olive Oil .....	47	Liquids .....	1049
Olivers, The Volatile Hydrocarbons of Essence of Valerian .....	758	Pears, jun., A. English .....	1049
Omliehl, J. See Hurter .....	393, 409	Pearson, J., and Taunt, F. H. Treatment of and Prevention .....	1049
Onimus, E. Phenomena of the Dialysis of Cells of Beer Yeast .....	1214	from the Seed of the Locust Bean (P) .....	1049
Oppenheimer, Sir C. Report on German Mining Industry .....	495	Pearson, W. E. S. Tattersall .....	1049
Oppermann, C. T. J. Preparation of Iron or Steel Plates for .....	641	Pense, E. N. See Dudley .....	1049
Plating (P) .....	641	Peco, S. Gilbert .....	1049
Ortmann, J. H. W., and Herbst, C. W. C. Apparatus for .....	137	Pedersen, M. Milk Testers (P) .....	1049
Sterilising or Disinfecting Liquids (P) .....	137	Pellegrini, J. E. Manufacture of Sugar and Vinegar .....	1049
Osborne, T. B. The Proteids of the Kidney Bean .....	828	(P) (illus.) .....	1049
And Voorhees, C. G. The Proteids of Cotton-Seed .....	828	Pellet, H. Determination of Crystallisable Sugar .....	1049
Osmond, F. Alloys of Iron and Nickel .....	641	Pemberton, Discussion on Spontaneous Combustion of Oil .....	116
The Structure of Steel .....	1065	Pemberton, jun., H. Determination of Phosphate Acid .....	176, 667
Osterseker, O. Printing and Dyeing on Fabrics (P) .....	390	Peniakoff, D. A. Manufacture of Alumina, Aluminium, and .....	1049
Ostrogovich, A. See Schiff .....	797	Aluminous Compounds and Obtaining By-Products (P) .....	1049
Ostwald, W. The Scientific and Technical Electrochemistry .....	1203	Pennington, J. C. Manufacture of Beer (P) .....	1049
of the Present and Future .....	1203	And Allison, W. O. The "Retting" of Flax .....	1049
Zinc Ferrocyanide as a Fixing Agent .....	1193	Peptide Maltine, Linn, and Bartlett, G. Peptoms and Extracts .....	1049
Orto, C. A. Roasting Coffee, &c. (P) .....	1083	(P) .....	1049
Orto, M. Apparatus for Fractional Distillation (illus.) .....	857	Perkin, A. G. Discussion on Determining Shade of Alizarine .....	1049
And Verley, A. Manufacture of Ethers and Alcohols in .....	1219	Pastes .....	1049
Natural Essences (P) .....	1219	Discussion on Indian Dyestuffs .....	1049
Orto, R. Spontaneous Formation of Pyruvic Acid from .....	733, 733	Discussion on Mordanting Wool with Chromium .....	1049
Aqueous Solution of Tartaric Acid .....	733, 733	Rapid Method of Determining the Shade of Alizarine .....	1049
Overton, J. W. An Improved Paint (P) .....	163	Pastes .....	1049
Composition of Materials for Blocks of Paving Cement, &c. .....	797	See Hummel .....	1049
(P) .....	797	And Hummel, J. J. Colouring and other Principles in .....	1049
Owens, C., and Whiteley, J. Machines for Washing and Treating .....	390	Mang-Kondu .....	1049
Fabrics (P) .....	390	And Hummel, J. J. The Colouring and other Principles .....	1049
		in Clay Root .....	1049
		And Hummel, J. J. The Colouring Principles of Rubia .....	1049
		Sikkimensis .....	1049
		Perkin, W. H. See Hummel .....	1049
		Perkins, A. B. See Rhodes .....	1049
		Perl, J. Determination of Oil of Turpentine in Alcohol .....	127
		Varnishing in Colours (P) .....	164, 164
		And Herrmann, P. Varnishing Metal in Light-Resisting .....	1049
		Colours (P) .....	1049
		Perry, A. See Hantefenille .....	1049
		Peter, A. H. See Waldstein .....	1049
		Peters, C. A. Is dating or Non-Conducting Material (P) .....	1049
		Petersen-Müller, F. Manufacture of Colouring Matters, and .....	1049
		their Salts and Sulpho-Derivatives (P) .....	1049
		Petit, A., and Fèvre, A. On 8-Rosalin .....	1049
		And Polonovsky, M. A Study of Lserine .....	1049
		Petit, P. Influence of Iron on the Vegetation of Barley .....	1049
		The Oxidation of Beer Wort .....	1049
		Petrie, F. W. Apparatus for Manufacture of Caustic Soda or .....	1049
		Potash (P) .....	1049
		Petrie, J. B. See Sharp .....	1049
		Petruschevsky, T. Importance of Study of Chemistry of Oil .....	1049
		Painting .....	1049
		Petschel, W. Secondary Electric Batteries (P) .....	1049
		Pettigrew, G. Still and Apparatus for Distillation of Ammo .....	1049
		niacal Liquor, &c. (P) .....	1049
		Pettitt, C. E. Manufacture of Photographic Plates and Films .....	1049
		(P) .....	1049
		Penchen, S. C., and Clarke, P. Concentration of Sulphuric .....	1049
		and Acetic Acids (P) .....	1049
		Vaporisation of Petroleum and other Liquids (P) .....	1049
		Peyrussan, A. E. Secondary Batteries (P) .....	1049
		Pfeifer, The Internal Rusting of Boiler Shells .....	1049
		Pfeiffer, G. Apparatus for Analysis of Gases (illus.) .....	1049
		Pharmakoffsky, N. The Tar of the Bark of Populus Tremula .....	1049
		Philips, A. See Graebe .....	1049
		Phillips, F. C. Natural Gas - its Composition and Origin .....	1049
		Researches on the Oxidation and Chemical Properties of .....	1049
		Gases .....	1049
		Phillips, H. J. Elimination of Sulphur from Molten Iron or .....	1049
		Steel (P) .....	1049
		Engineering Chemistry. Second Edition .....	1049
		Picard, H. F. K., and Thame, H. Manufacture of Plates for .....	1049
		Secondary Batteries (P) .....	1049
		Picard, J. See Clapham .....	1049
		Pickering, S. C. Specific Gravity of Calcium Chloride Solution .....	1049
		The Densities of Solutions of Soda and Potash .....	1049
		Pickles, J. W. Compositions for Preserving Wine, &c. (P) .....	1049
		Pieter, Soc. Anon. del'Institut Raoul. Disinfecting and Appa .....	1049
		ratus therefor (P) .....	1049
		Pie, P. Apparatus for Creating a Vacuum in Vacuum Pans .....	1049
		(P) .....	1049
		Pieck, A. See Baumert .....	1049
		Pieper, C. Manufacture of Explosives (P) .....	1049
		Production of Dextrins and Leicomes (P) .....	1049
		Pikhart, J. Manufacture of Beer (P) .....	1049
		Pilory, O. Quantitative Analysis by Electrolysis .....	1049
		Pitt, S. A New Antiseptic Material (P) .....	1049
		Preparation of Antiseptics (P) .....	1049

PAGE

Phalet, E. and Boute J. Extraction of Chromium by Electrolysis (P).....	646
Preparation of Chromic Acid, and Extraction of Pure Acids from Alkaline Salts (P).....	37
Platten, F. Discussion on Estimation of Arsenic in Copper.....	326
Moderation of Ferric Chloride Distillation Process for Estimation of Arsenic in Copper.....	324
Platz, R. T. See Travers.....	277
Pöschel, J. See W. von Miller.....	767
Polewsky, A. von. Rotary Drying, Pulverising, and Exporting Machinery (P).....	871
Pokorny, J. The Synthesis of Azo Colours on Animal Fibres.....	388
Pokorny, M. The Formation of Resonance-green on Cotton, Wool, and Silk.....	757
Polakowsky, H. The Nitro Question.....	559
Polek, T. Use of Sodium Peroxide for Analytical Purposes.....	960
Polak, C. Electrodes for Accumulators (P).....	41
Polonovsky, M. See Petit.....	758
And Nitzberg, J. Dyeing Indigo Blue combined with other Colouring Matters.....	149
Polsky, F. M. Manufacture of Yeast (P).....	55, 268
Poussin, J. See Dunn.....	1083
Pommeranz, G. Producing Consistent Fats and Oils without Heat (P).....	531
Pond, G. G. Apparatus for Rapid Determination of Combustible Gases (illus.).....	544
Ponzo, G. Composition of Rape-seed Oil.....	257
Potts, G. R. See Molyneux.....	413
Powell, A. E. Proceedings at Annual General Meeting.....	623
Powell, J. R. Miners' Signs (P).....	760
Payer, P. B. Catalogue of Essential Oils and Organic Chemical Preparations.....	980
Prado, A. da S., and Medina-Santurio, B. Manufacture of Earthenware or Porcelain Filters (P).....	811
Prager, A. Gravimetric Estimation of Cupric-Reducing Sugars.....	1227
Pringley, L. E. A. Continuous Refining or Bleaching of Sugar (P).....	265
Prst, J. P. Method and Apparatus for Filtering (P).....	137
Prentice, M. Apparatus for Continuous Production of Nitric Acid (illus.).....	523
Discussion on Improved Specific Gravity Bottle.....	324
Manufacture of Acids and Superphosphate of Lime (P).....	250
Manufacture of Nitric Acid (P).....	250
Frestwich, W. H. Silver Haloid Preparations for Photographic Purposes (P).....	417
Price, J. Apparatus for Cooling Liquids or Gases (P).....	1048
Priest, W. B. See Wilson.....	1071
Prigster, H. Compound for Manufacture of Matches (P).....	661
Prinsen-Greelies. The Decomposition of Invert Sugar by Alkalis, and Practical Application thereof.....	410
Prior, E. Estimation of the Acidity of Malt.....	986
Influence of Cane Sugar on Valuation of Malts and Worts.....	1080
Prior, J. See Mountfort.....	412
Procter, J. Discussion on Substantive Dyes and Dyeing.....	127
Procter, H. R. Discussion on Action of Antiseptics on Yeast.....	1164
Discussion on Bacteriological Analysis of Water.....	1166
Discussion on Estimation of Tanning Matter.....	495
Discussion on Mordanting Wool with Chromium.....	367
Discussion on Spontaneous Combustion of Oils.....	1166
Do's Tannin Tan Hides.....	747
On the Estimation of Tanning Matter (illus.).....	494
On the Quantitative Determination of Tanning Materials.....	187
See Hummel.....	696
Prud'homme, M. Teinture et Impression.....	429
The Quinonoximes in Steam Colours.....	118
And Rabaut, C. The Constitution of Colouring Matters of the Magenta Group.....	382
And Rabaut, C. The Formamides of Alizarin.....	113
Prud'homme, L. See Ehrenbach.....	726
Prussing, C. Testing Hydraulic Cement.....	949
Prussing, W. J. Purifying Waste Waters of Tin-Plate Works (P).....	957
Pullar, R. Proceedings at Annual General Meeting.....	692
Speech at Annual Dinner.....	704
Speech at Perth Banquet.....	706, 707, 708
Pullman, J. See Lane.....	810

Q

Quarez-Figues, Limulus (P).....	1062
Quilliam, S. Manufacture of Sanitary Paper, Felt, &c. (P).....	834
Quine, R. H. Bottles for Holding Poisons and Mixtures (P).....	713

R

Rabaut, C. See Prud'homme.....	113, 382
Radtner, S. A New Anti-pyretic and Anti-neuralgic Preparation (P).....	542
Raleigh, C. Extracting Gold and Silver from Solutions (P).....	492
Treatment of Slimes or Ores containing Precious Metals (P).....	401
Raschen, J. See Brock.....	1135
Raspe, C. Manufacture of Carbonate of Ammonia (P).....	334
Rathenau, W. See Kiliani.....	1068
Raumer, E. von. Composition of Honey-Dew, and its Influence on Honey.....	1075
Ravizza, F. Action of Calcium Sulphite and Potassium Bisulphite on Alcoholic Fermentation.....	266
Rawson, C. Discussion on Indian Dyestuffs.....	353
Discussion on Mordanting Wool with Chromium.....	365
Discussion on Substantive Dyes and Dyeing.....	127
Raze, A. Avoiding Choking of Mouth of Retort in Manufacture of "Thomas" Steel (P).....	158
Reid, A. A. See Arnold.....	740
Reid, Holliday and Sons, and Bindsehader, H. Production of an Indigo Vat or Bath (P).....	399
And Holliday, R. Production of New Colouring Matters (P).....	511
Readman, J. B. Discussion on Electric Furnace for Laboratory Work.....	576
Discussion on Preparation of Carbon Blacks.....	131
Effect of Saline Constituents of Water on Character of Beer.....	367
Electric Furnace for Laboratory Work.....	375
Proceedings of Annual General Meeting.....	700
Rehony, Continuous Saturation.....	719
Recklinghausen, M. V. Mercury Thermometer for High Temperatures.....	67
Redwood, Boverton. Discussion on Colorimetric Analysis.....	317
Discussion on Distillation of Peat.....	606, 607
Discussion on Treatment of Gold Ore in the Transvaal.....	339
See Deering.....	569
The Transport of Petroleum in Bulk (illus.).....	1182
Rée, A. See Chaus.....	384
Reed, A. Bottles for Containing Poisonous Substances (P).....	788
Reid, J. Method and Apparatus for Dyeing, &c., Fabrics and Yarns (P).....	513
Reformasky, A. See Markownikoff.....	272, 758
Rehnstrom, A. W. Manufacture of Feeding Cakes (P).....	1215
Reich, J. Manufacture of Alkaline Carbonates and Silicates (P).....	1059
Reichard, C. Reactions of Acid Potassium Arsenite with Metallic Salts.....	732
Reid, W. F. Discussion on Electrolysis of Fused Salts.....	454
Discussion on Oxidised Linseed Oil.....	1024
Discussion on Smokeless Powder.....	582
Discussion on Testing Hydraulic Cements.....	461
Discussion on the Colour of Brass.....	595
Note on Oxidised Linseed Oil.....	1020
Reifsnnyder, S. K. See Shimer.....	277
Reinicke, P. Machine for Straining Pulp, &c. (P).....	713
Reinsch, P. F. Spray-tanning.....	651
Reisert, H. Process and Apparatus for Cleansing Filtering Material (P).....	378
Reissner, R., and Hauser, G. C. F. Making Photographic Films (P).....	973
Reitzel, E. Colorimetric Methods of Estimating Carbohydrates.....	985
Remmler, W. See Jannasch.....	66
Renard, A. Pine Tar.....	1188
Tar of Pinus Maritima.....	1188
Renard, P. V. Clouding Textile Materials in Dyeing (P).....	1194
Renault, B. See Bertrand.....	231
Remison, J. Material for Varnish, Cement, &c. (P).....	648
Retzlaff, J. Etching on Glass, Porcelain, &c. (P).....	154
Reychler, A. Diffusibility of Gases through Caoutchouc Membranes.....	181
Essence of Ylang-Ylang.....	758
Reychler, M. A. The Essence of Ylang-Ylang.....	1085
Rey-Pailhade, J. de. Chemical Properties of Alcoholic Extract of Beer-Yeast.....	751
Rhodes, J., and others. Apparatus for Dyeing and Washing Yarn (P).....	399
And Perkins, A. B. Apparatus for Dyeing Slabbing or Slivers (P).....	883
Richard-Lacaze, E. Apparatus for Extracting Suint from Wool (P).....	962
Richardson, A. Action of Light on Oxalic Acid.....	516
Richardson, C. G., and English, A. B. Treatment of Metallic Ores (P).....	401
Richardson, F. W. Bacteriological Analysis of Water.....	1157
Discussion on Action of Antiseptics on Yeast.....	1153



	PAGE
Richardson, E. W. Discussion on Bacteriological Analysis of Water.....	1160
Discussion on Determining Shade of Alizarine Pastes.....	486
Discussion on Estimation of Tanning Matter.....	485
Discussion on Merdanting Wool with Chromium.....	393
Discussion on Substantive Dyes and Dyeing.....	127
Richardson, J. C. Safety Appliances for Electrolytic Apparatus (P).....	358
Tanks for Electrolysis of Solutions of Sodium or Potassium Chloride (P).....	646
Riche and Roume. Removal of Sulphur from Mineral Oils at Chicago.....	719
Riehet, C. See Hanriot.....	755, 755
See Chassevant.....	293
Riehter, M. F. H. Preventing Spontaneous Ignition of Liquid Hydrocarbons (P).....	1192
Rideal, S. Discussion on Colorimetric Analysis.....	317
Discussion on Sterilisation of Liquids.....	1136
Discussion on Strength of Leather.....	532
Riedel, J. D. Production of Para-Phenetolcarbamide ("Dulcin") (P).....	542
Ries, R. Carburetting Coal-gas with Benzene.....	873
Rigby, R. K., and others. Manufacture of Cement from Alkali-Waste (P).....	688, 948
Riley, J., and The Thornhillbank Co. Bleaching and Cleansing Yarns and Fabrics (P).....	728
Rimlach, E. Ratio of Saccharimeter Degrees on Ventzke Scale to Angular Degrees for Sodium Light.....	1222
Rimmer, H. Soap Cakes and Bars (P).....	618
Rimington. Discussion on Commercial Analysis of Lard.....	708
Riordon, C. Storage Batteries (P).....	1206
Ripley, R. Manufacture of Black Lead (P).....	162
Ristori, E. See Macnab.....	973
Rix, W. P. See Doulton.....	395
Robbins, W. O. Apparatus for Treating Substances containing Fats and Oils (P).....	1070
Roberts-Austen, W. C. Discussion on Japanese Shirogane and Copper.....	470
Researches on the Properties of Alloys.....	134
Robertson, A. Scab Wash or Lotion for Sheep (P).....	833
Robinson, A. E. See Parker.....	250
Robinson, W. See Gaskell.....	154
Rockwood, G. G. See Forster.....	417
Rohde, G. See Miller.....	142
Rolfe, J. R. See Chapman.....	843
Roman, R. I. An Improved Aluminium Alloy (P).....	956
Solder for Aluminium or Aluminium Alloys (P).....	956
Roos, A. See Kraft.....	756
Ropes, C. A., and Sellars, J. C. Improvements in Cements (P).....	639
See Sellars.....	888
Roscoe, Sir H. E., and Schorlemmer, C. A Treatise on Chemistry. Revised by Sir H. E. Roscoe, H. G. Colman, and A. Harden. Vol. 1. The Non-Metallic Elements.....	1107
Rose, E. See Jannasch.....	1224
Rosenfeld, M. Production of Chemically Pure Caustic Soda and Hydrogen from Sodium.....	240
Rosenstiel, A. Blue Coloration produced by Leuco-aureamine in contact with Acids.....	629
Comparison of Coloured and Colourless Derivatives of Di- and Triphenylmethane.....	798
Rosenstiel, M. A. On the Nature of the Forces concerned in the process of Dyeing.....	726
Report on Vignon's Note on Theories on Dyeing.....	249
Rosset, A. Preparation of Phosphorus by means of the Reducing Action of Aluminium.....	845
And Frank, L. Decomposition of Sodium Peroxide by Aluminium.....	516
And Frank, L. Formation of Phosphorus from Phosphates.....	515
Rositter, E. C. See Crowther.....	857
Roth, C. Apparatus and Method for Igniting Blasting Charges (P).....	650
Rothenburg, R. von. Pyrazolone Reactions.....	765
Roume. See Riche.....	719
Rous, H., and West, G. N. Rendering Textiles Waterproof and Flame-proof (P).....	725
Rousseau, C. Plates for Electric Batteries (P).....	744
Rowlands, W. E. See Currie.....	711
Royle, J. J. Apparatus for Regulating Temperature of Fluids (P).....	1179
Rubricius, H. Variations in the Silicon Percentage of Samples of Pig Iron from same Tap.....	889
Rudeloff, M. Influence of Temperature on Tensile Strength of Metals.....	520
Rudolmzer, J. Electro-deposition of Metals (P).....	329
Rudorff, F. Quantitative Electrolytic Analysis.....	1223
Rürup, L. Volumetric Estimation of Sulphuric Acid in Crude Hydrochloric Acid (illus.).....	904
Rushford, B., and Edwards, J. Filtering Sewage (P).....	271
Rushton, A. See Worcester Porcelain Co.....	1197
Russell, Sir J. A. Speech at Annual Dinner.....	701

Russell, W. J. See Hummel.....	146 P
Rustin, A. See Levy.....	803
Ruybeke, J. V., and Johnson, W. F. Process and Apparatus for obtaining Glycerin, &c., from Spent Lyes (P).....	1070
Ryu, J. J. L. van. Carpin.....	757

## S

Sabancjeff, A., and Antouchevitch, A. The Properties of Caramel.....	1076
Sabatie, P. Delicate Reaction for Copper Compounds.....	1062
Sachs, J. Manufacture of Coated Metallic Powders (P).....	893, 958
And others. Manufacture of Pigments (P).....	532
Sachse, R., and Becker, A. Action of Lime, Salts, and some Acids on Clay.....	409
Sachsenroder, G. Manufacture of a Substitute for Leather (P).....	964
Sadlon, C. Course of Absorption of Tannin in the Layers.....	49
Sahfeld, F. E. See Knebel.....	829
Sahlstrom, C. A. Preservation of Alimentary and other Substances (P).....	56
And Parr, E. Saponifying or Mixing Tars, Oils, &c. (P).....	139
Raw-Grain Conversion in the Brewery.....	416
Discussion on Sterilisation of Liquids.....	1136
See Hood.....	1195
Salberg, F. S., and Neumann, M. Disinfection of Refuse and Apparatus therefor (P).....	832
Salkowski, E. The Carbohydrates of Yeast.....	411
Yeast Gum.....	1243
Salmon, H., and Whiting, J. E. Substitute for Pumice Stone, and Manufacture thereof (P).....	735
Salzer, T. Value of Borax as the Basis of Acidimetry.....	280
Salzmann, T. Drying Dyed Cotton Yarns (P).....	947
Samelson, Lard.....	766
Sandoz and Co. Production of Colouring Matters (P).....	651
Sanford, P. G. The Gelatin Explosives.....	276
Sangle-Ferriere. Detection of "Abrastol" in Wine.....	177
Santer, E. H. Discussion on Methods for Estimation of Manganese.....	115
Purification of Iron (P).....	32
Review of Methods for Estimation of Manganese.....	112
Sarel, E. Capacity of Yeast to Live in Media containing Hydrofluoric Acid.....	655
Sartiz, J. The Carbonisation of Wood.....	380
Sass, C. F. H. See Axorfer.....	1050
Sattmann, A., and Homatsch, A. Manufacture of Iron and Steel (P).....	491
Sauer, E. New Form of Air-Bath (illus.).....	662
Sauerbeck, A. Course of Average Prices of General Commodities in England.....	Facing p. 762
Sawyer, J. C. Odorographia: A Natural History of the Drugs and Raw Materials used in the Perfume Industry.....	429
Sayer, R. C. Means for Treating Refuse (P).....	820
Saytzeff, M. C., and A. Action of Hydrogen, Sodium Sulphite, and Sulphurous Acid on Oleic and Erucic Acids.....	1208
Seammel, L. R. Extraction and Estimation of Eucalyptol (P).....	1106
Schachtrupp. See Spunt.....	263
Schaeffer, M. Reports on Modified Starches made by Siemens and Halske.....	264
Schall, C. Determination of Vapour Density, and Exhausting without Air or Water-Air Pump (illus.).....	1221
Schauffelen, C. A. Finely Granulated Paper, and Production of Same (P).....	543
Scheurer, A. Japanese Ponceau-Red Dye.....	508
Scheurer, A. Report on Aluminium Bronze "Doctor".....	388
Tendering of Cotton Goods by Iron Merdants.....	118
The Tendering of Cotton Fibre by Tartaric Acid.....	118
Scheurer-Kestner, M. The Chemical Action of Abrastol on Wine.....	654
Cause of Loss of Heat in Boilers.....	1014
Schicht, R. Method and Apparatus for Drying Viscous Substances (P).....	1182
Schiff, H., and Ostrogovich, A. Some Reactions of Orthotolidine.....	797
Schimitzek, K. See Thiel.....	497
Schindler, A. Manufacture of Gunpowder (P).....	977
Schinz, C. Incandescent Vapour Lamp and Generator (P).....	1052
Schjerning, H. Quantitative Separation of Nitrogenous Organic Compounds in Beer Wort.....	110
Schlagdenhauffen. See Heckel.....	47
Schlickeyen, C. Manufacture of Bricks, Terra-cotta, &c. (P).....	154
Schloesing, jun., M. T. The Hygroscopic Properties of Various Textiles.....	146
Schloesing, T. Separating Salts of Soda and Potash from Solutions of Magnesium Chloride (P).....	517

	PAGE		PAGE
Blasen, T. van. Technical Production of Bodies rich in Nicotine.....	1087	Scotch and Irish Oxygen Co. and Brier, H. Apparatus for Testing Gas Cylinders (P) (illus.).....	789
Bismann, T. Examination of Glue for Manufacture of Coloured Papers.....	424	Scott, A. Centrifugal Methods of Milk Analysis.....	716, 1101
Bland, C. S. Schraube.....	508	Scott, A. Ross. Manufacture of Cleansing Powder (P).....	1070
Bond, E. Alkaloids of Papaveracea.....	58	Scott, E. New Combustible and Production of Same (P).....	871
Bond, F. W. A White Form of Tin Sulphide.....	1231	Scott, E. G. and P. W. Apparatus for Evaporating and Cooling Liquids (P) (illus.).....	23
Bond, F. W. Estimation of Nickel.....	1225	Scott, F. W., F. W. jun., and E. G. Evaporating Brine and other Liquors, and Apparatus therefor (P) (illus.).....	622
Bond, F. W. Ammoniacal Mercuric Cyanide. Quantitative Analysis.....	518	Scudler, F. Discussion on Modern Systems of Sewage Treatment.....	608
Bond, O. Recovery of Sediment from Clarifiers (P).....	1216	Söber, G. Antimony Blue.....	46
Bonshoke, R. R. P., and Gude, L. O. Apparatus for Preparation of Malt (P).....	827	Sedgwick, W. T. Tests of a Disinfectant for Railway Sanitation.....	57
Bonitz-Bumont, W. S. Schraube.....	1101, 1227	Seekamp, W. Decomposition of Tartaric and Citric Acids by Sunlight.....	810
Brauer, S. C. The Electrolytic Separation of the Metals of the Second Group.....	743	Seitz, T. F. Filter-Presses (P) (illus.).....	714
Brauer, S. C. The Electrolytic Separation of Gold from Silver.....	250	Seldner, J. J., and Watson, J. jun. Manufacture of Fertilisers (P).....	533
Brauer, E. Tinting Light Shades on Wool by means of Alkaline Solutions of Alizarine Cyanine.....	388	Sellers, J. C. See Ropes.....	639
Brauer, C. The Determination of High Temperature by Fusion Tests.....	702	And Ropes, C. A. Stoneware for Building Blocks, &c. (P).....	888
Breider, G. A. E. Incandescence Bodies for Gas Lamps (P).....	715	Semmler, F. W. See Tiemann.....	417
Breider, L. Estimation of Chromium and Manganese in Steel.....	546	Sepulchre, L. Round Burners for Mineral Oil (P).....	793
Breider, A. See Wissen.....	160	Seybold, C., and Heeder, F. Manufacture of Manures (P).....	748
Brown, C. Tannin Orange.....	1189	Seyda and Woy. Comparison of Analytical Methods for Detection of Margarine in Butter.....	981
The Action of Light on Metastable State of Soda and its Applications.....	588	Seyffert. Calculation of the Rendement of Raw Sugars.....	965
Bußer, F. W., and G. F. W. Manufacture of Electric Accumulators (P).....	830	Sharp, H. B., and Petrie, J. B. Precipitation of Compositions for Treatment of Sewage (P).....	1216
Burke, F. On the Question of Atmospheric Hydrogen Peroxide.....	753	Sharples, G. B. Purification and Filtration of Sewage, &c. (P).....	656
Burke, P. Barren's Theory of the Lead Secondary Battery, and Life of Lead Accumulator Plates.....	1203	Shedlock, W. Apparatus for Removing Impurities from Water (P).....	23
Secondary Batteries (P).....	636	Shelley, P. Decorating Pottery, Glass, &c. (P).....	949
Burkholder, C. S. See Roscoe.....	1107	Shiels, A. Improvements in Thermostats (P).....	137
The Rise and Development of Organic Chemistry.....	1106	Slimer, W. P., and Reifnyder, S. K. Sampling by Quotation.....	277
Burns, J. Quinine and its Compounds.....	835	Shorey. Precipitates Obtained in Apparatus for Evaporating Cane-Sugar Juice.....	750
Burt, E. Effect of Addition of Gypsum to Portland Cement.....	251	Shotter, A. H. See Anderson, G. R.....	729
Burt, A. J. Bleaching Cotton Cloths, &c. and Apparatus therefor (P).....	634	Siemens, P. Converting Non-Gaseous Hydrocarbons into Combustible Gas (P) (illus.).....	139
Burder, J. Purifying Petroleum (P).....	1054	Siemens and Halske. Bleaching Raw Starch, Starch Gum, and Solutions thereof (P).....	34
Burder, C., and Schmidt, C. Diaz Compounds and Nitrosamines.....	505	Siemens Bros. and Co., and Olach, F. A. Apparatus for Electrolysis of Water (P).....	529
Burder, H. The Purification of Effluents.....	829	Siepmann, W. Production of Alkali Cyanides (P).....	250
Schrijnen, D. Electrical Phenomenon in Preparation of Salicylic Acid.....	628	Silber, P. See Clamann.....	416, 756, 1217
Burder and Bartel, A. Extraction of Tanning Materials.....	649	Silbermann, A. See Bernstein.....	793
Burder, C. A. J. H., and H. E. R. Electro-positive Electrodes (P).....	257	Silbermann, H. Influence of "Weighting" on Specific Gravity of Silk.....	907
Galvanic Batteries (P).....	528, 160, 160	Treatment of Tussah Silk (illus.).....	880
Burder, M. See Grillo.....	408	Sample Silk.....	1056
Burder, V., and others. Estimation of the Sugar-Content of Tanning Materials, &c.....	1227	Wild Silk.....	884
Schroter, H. Contributions to the Knowledge of Albumenosis.....	273	Silcock, R. Method and Apparatus for Removing Fibre from Cotton seed (P).....	945
Hubert, B. Basic Salicylate of Bismuth.....	57	Simon Thomas, J. C. A. The Dervaux Feed-Water Purifier.....	376
Hull, G. Carbonisation of Rags, Wool, &c. (P).....	945	Simonet, V. Non-poisonous Matches (P).....	173
Hultz, G., and Julius, F. Systematic Survey of the Organic Colouring Matters. Translated and Edited by A. G. Green.....	671	Simpkin, S. J. Manufacture of Refined Indigo (P).....	382
Hultz, E. Quantitative Estimation of Carbohydrates.....	985	Sims, W. E. See Holt.....	526
And Frankfurt, S. Crystalline Levulose.....	553	Siman, A., and H. Clarifying or Decolourising Tannic Extracts (P).....	164
And Frankfurt, S. Dissemination of Saccharose in Seeds. And Frankfurt, S. Occurrence of Raffinose in Wheat Germs.....	553	Sinclair, G. Boilers for Treating Exports and other Fibrous Material (P).....	754
And Frankfurt, S. Occurrence of Trigoneillin in the Seeds of Pisum Sativum and Cannabis Sativa.....	653	Sinding-Larsen, A. Process and Apparatus for Electrolytical Decomposition of Alkali Salts (P).....	1206
And Frankfurt, S. Preparation of Betaine and Choline.....	757	Singer, C. F. See Koneman.....	42
Sack, I., and Marchlewski, L. Dyeing with Glucosides and Rhamnosides.....	683	Singer, E., and others. Process for Soldering Aluminium (P).....	645
And others. A Colouring Matter occurring in the Leaves of the Grape Vine.....	509	Singer, L. See Engler.....	45
Schuppahaus, R. C. Natural Colours (P).....	1220	Simbaldi. Detection of "Abrastol" (Calcium- $\beta$ -naphthol-sulphonate) in Wines.....	548
Schuppahaus, R. S. Securing the Chemical Stability of Nitro-Compounds (P).....	174	Preservation of Wines by $\beta$ -Naphthol Sulphonic Acid.....	534
Schuster, A. Action of Copper Sulphate and Sulphuric Acid on Copper.....	957	Sjostrom, D. See Anderson.....	800
Schuster, C. A. Relations between the Optical Rotatory Powers of Cinchonidine and its Salts.....	72	Skinner, W. See Dixon.....	957
Schuster, H. Removal of Iron from Drinking Water.....	1083	Skrup, Z. H. Some Decompositions of Quinine.....	171
Schwartz, C. See Bloch.....	1183	Slack, J. Apparatus for Manufacturing Pottery (P).....	1032
Schwartz, A. W. Method and Apparatus for Blasting (P).....	975	Slater, J. See Boulton.....	637
Schweitzer, H. Discussion on Detection of Soap in Lubricants.....	1179	Slatter, G. W. Discussion on Determining Shade of Alizarine Pastes.....	496
And Lungwitz, E. Apparatus for Weighing Out Liquids.....	838	Discussion on Indian Dye-stuffs.....	353
And Lungwitz, E. Commercial Analysis of Lard.....	614	Discussion on Spontaneous Combustion of Oils.....	1166
And Lungwitz, E. Discussion on Commercial Analysis of Lard.....	709	Slier, C. H. Production of Asphaltic Composition as Powder or Mastic (P).....	638
And Lungwitz, E. Mill for Sugar Assay (illus.).....	838	Slocum, F. L., and Wheeler, C. Y. Making Green Oxide of Chromium (P).....	1071
And Lungwitz, E. Reaction for Detection of Soap in Lard.....	1178	Slyke, L. L. Determination of Casein in Cows' Milk.....	181
See also H. H. S. for Bricks.....	1299	Smith, A. Discussion on Manufacture of Straw Cellulose.....	107
		Smith, A. Apparatus for Softening and Purifying Water (P).....	338
		Smith, A. H. A Porous Material for Tobacco Pipes (P).....	395

	PAGE		PAGE
Smith, A. J. Pickling Metals and Alloys to remove Impurities (P).....	526	Stanger, W. S. Diss. Assoc. on Sodium Peroxide.....	109
Smith, A. W. See Mabery.....	597	Manufacture of Yeast (P).....	109
Smith, C. Natural Oxycelluloses. I. Celluloses of the Gramineae.....	597	Staudt, G. Lithium. (P).....	109
Smith, C. A. Artificial Lumber or Composition (P).....	811	and Anhydrous.....	109
Smith, E. A. On the Cupellation of Bismuth-Silver Alloys.....	767	Staudt, Dr. Discussion on Extraction of Lithium.....	109
Smith, J. Apparatus for Producing Gas from Coal-Tar (P).....	625	Matters.....	109
Dyeing, Ac., Fabrics and Fibres (P).....	513	St. A. P. P. Personal Communication.....	109
Production of Illuminating Gas (P).....	873	Particulars in First Communication.....	109
Smith, R. Greig. An Abnormal Milk.....	613	The Copper Ores of Persia.....	109
Smith, Watson. Discussion on Distillation of Peat.....	636	Staudt, E. C. C. Discussion on Lithium.....	109
Discussion on Treatment of Gold Ore in the Transvaal.....	393	Laboratory Work.....	109
Eulogium on Dr. Alder Wright.....	1020	Discussion on Waters for Water-Purification.....	109
Obituary Notice of C. R. Alder Wright.....	785	Presidential Address.....	109
Obituary Notice of Dr. H. J. Grunberg.....	783	See "at Annual Dinner.....	109
Remarks concerning Dr. Alder Wright's last Work.....	1013	Speech at Annual General Meeting.....	109
Smithells, A. Address to the Yorkshire Section.....	18	Speech at Fifth Banquet.....	109
Discussion on Estimation of Tanning Matter.....	495	St. George, W. H. Discussion on Testing Hydraulic Cements.....	40
Discussion on Indian Dyestuffs.....	353	And Blount, B. The Testing of Hydraulic Cements (illus.).....	109
Proceedings of Annual General Meeting.....	760	Stanley, J. A. W. Manufacture of Black Portland Cement.....	109
Schorlemmer's Rise and Development of Organic Chemistry.....	1163	Refuse (P).....	109
Discussion on Action of Antiseptics on Yeast.....	1164	Treatment of Fish and Fish-Oil (P).....	109
Discussion on Spontaneous Combustion of Oils.....	1165	Stanley, E. Ovens for Bent Briquettes, &c. (P).....	71
Smithson, S. Apparatus for Treating Excreta, Refuse, &c. (P).....	271	Stanley, E. See Spindler.....	896
Smithson, T. Fumigant for Use in Horticultural Buildings (P).....	900	Stear, W. H. Treatment of Cotton Seed for Removal of Fibrous Matters (P).....	820
Smolnichowski, Th. v. See Herzog.....	798	Steffen, C. Purifying and Decolorising Saccharine Jellies and Solutions (P).....	111
Snelus, G. J. The Walrand-Lezenisiel Process as Applied to Steel Castings.....	642	Purifying and Decolorising Sugar Solutions (P).....	106
Société Anonyme de l'Institut Raoul Pictet. Disinfecting, and Apparatus therefor (P).....	900	Steffen and Bruckner. Purifying and Decolorising Beetroot Juice and Sugar Solutions.....	823
Société Anonyme des Hauts Fourneaux de Duclouange. Manufacture of Steel or Wrought Iron (P).....	42	Stein, G. Use of Benzidine Colours in Dyeing.....	822
Société Anonyme des Matières Colorantes, &c., St. Denis. Manufacture of Colouring Matters (P).....	725	Stein, G. W. A. Manufacture of Hydraulic Cement (P).....	109
And Chapuis. Manufacture of Monosulphonated Anthracene Acid (P).....	32	Manufacture of Hydraulic Cement (P).....	109
Société Electrique Phœbus, La. Plants for Secondary Batteries (P).....	161	Stendhart, A. See China.....	177
Société "L'Accumulateur Fulmen." Storage Batteries (P).....	819	Stepanow, A. See Lissenko.....	177
Société Marchville Daguin et Cie. Calcining Apparatus for Bicarbonate of Soda (P).....	518	Stern, A. See Kraft.....	1197
Society of Chemical Industry. Basic. Manufacture of Blue Acid Colouring Matters of the Triphenylmethane Series (P).....	1055	Stern, A. L. Cellulose Sulphuric Acid and the Products of it.....	1200
Manufacture of Blue Colouring Matters (P).....	31	Hydrolysis.....	1200
Manufacture of Compounds of <i>p</i> -Aminodiphenyl Ethers and Aromatic Oxaldehydes (P).....	274	Sternberg, L. Apparatus for Obtaining Ammonia and Ammonia Salts (P).....	887
Manufacture of Polyoxythionine Colouring Matters (P).....	879	St. George, A. F. Manufacture of Artificial Caoutchouc (P).....	49
Sohège, P. Manufacture of Cellulose Pulp (P).....	1216	Stillman, T. B. Analysis of Lubricating Oils containing Blow.....	286
Sohn, C. E. Dictionary of the Active Principles of Plants, Alkaloids, &c. (P).....	72	Rape and Cotton-Seed Oils.....	286
Soldani, A. Alkaloids from the Seeds of <i>Lupinus Albus</i> .....	757	Chemical and Physical Examination of Portland Cement.....	637
Soldnerhoff, R. de. The Manufacture of Coke.....	933	The Chemical and Physical Examination of Portland Cement.....	278
Solms-Baruth, Count. Manufacture of Glass Articles in Imitation of Agate (P).....	39	Stock, A. The Aurumines.....	449
Sommer, A. Solutions of Sweet Carbamides in Oils, &c., and Making same (P).....	1071	Stocketh, G. Liquid Fuel.....	625
Sommer, G. Schack. Sugar-Beet Growing Experiments in 1893.....	213	Stollert, H. Sewage Receptacles and Desodorising same (P).....	170
Sousladt, E. Preparation of Extracts of Tea and Coffee (P).....	270	Stollia, F. The Volumetric Determination of Potassium Borofluoride.....	250
Sorel, E. Rectification of Alcohol.....	1081	Stone, F. R. Apparatus for Aerating Wort and other Liquids (P).....	157
See Joly.....	634	See Walters.....	263
Sorley, D. Notes on Sampling.....	975	Stone, R. Manufacture and Treatment of Fireproof Plastic Material for Cement (P).....	811
Sostegni, L., and Carpentieri, F. Detection of Coal-Tar Dyes in Wines.....	906	Stone, W. E., and Test, W. H. The Carbohydrates of the Fruit of the Kentucky Coffee-Nut Tree.....	976
Southey, A. W. See Lubbe.....	45	And Dickson, C. Characteristics of Glucose Syrups.....	178
Soxhlet. Vacuum Evaporating Apparatus for Laboratories (illus.).....	977	Storer, J. See Lacy.....	711
Soy, E. Vessels for Containing Liquid Sulphur Dioxide (P).....	271	Storer, J. J., and others. Furnace and Apparatus for Working Ores (P).....	316
Spalding, L. Improvements in Filters (P).....	137	Storoy, H. L., and L. H. See Farmer.....	30
Specht, L. See Erban.....	150, 1194	Storner, H. C. F. Process and Apparatus for Electrolytically Treating Salts (P).....	1067
Spence, P. Hydrostatic Apparatus for Automatically Mixing Substances with Water (P).....	713	Stouls, E. Process and Apparatus for Obtaining and Treating Metals Electrolytically (P).....	958
Spence, F. M. and D. D., and Gilchrist, J. Apparatus for Distillation of Ammoniacal Liquor (P).....	394	Strasser, R. See Douth.....	780, 426
Spencer, E. S. Manufacture of Gelatin (P).....	897	Strauss, A. J. Treatment of Hides and Skins (P).....	51
Spencer, I. See Lotherington.....	162	Strauss, R. See W. von Miller.....	767
Spiethoff, F. See Hensel.....	1075	Strawson, G. F., and Freeman, W. Manufacture of Copper Salphate (P).....	57
Spindler, W., and Stantz, C. Extracting Oil from Plants and Fruits, and Production of Fodder from the Residue (P).....	893	Street, E. A. G. See Girard.....	620, 617
Spott, E. See Waldstein.....	947	Strohmmer. Decomposition of Sugar during Evaporation.....	10
Spuller, J., and Kalman, S. Estimation of Chromium in Chromium Steel and Chrome Iron Ore.....	281	Strom, O. J. R. Centrifugal Separators (P) (illus.).....	191
Estimation of Phosphorus in Steel containing Silicon, and in Pig-Iron.....	175	Stroud, W. See Hummel.....	893
Spunt and Schachtrupp. Purification of Beetroot Juice.....	263	Strype, W. G. Apparatus for Controlling Discharge of Liquid from Tanks (P).....	1150
Stanger, W. S. Diss. Assoc. on Sodium Peroxide.....	109	Stubley, G. and J. "Hawking" Machines for Dyeing.....	120
Manufacture of Yeast (P).....	109	(P).....	120
Staudt, G. Lithium. (P).....	109	Sugg, D. W. An Improved Metallic Alloy (P).....	645
and Anhydrous.....	109	Suhr, E. Study of the Methods of Estimating Glycerol.....	27
Staudt, Dr. Discussion on Extraction of Lithium.....	109	Suiter, A. Saturators and Jets for Producing Oxylhydrogen Light (P).....	574
Matters.....	109	Surre, L. Preservation of Wines by Sodium Silico Fluoride.....	524
St. A. P. P. Personal Communication.....	109	Sussmann, M. Secondary Batteries (P).....	528
Particulars in First Communication.....	109	Suter, C. See Kiani.....	1028
The Copper Ores of Persia.....	109	Sutherland, D. A. Discussion on Estimation of Arsenic in Copper.....	320
Staudt, E. C. C. Discussion on Lithium.....	109	Discussion on Treatment of Gold Ore in the Transvaal.....	330
Laboratory Work.....	109		

	PAGE
Swanburne, J. Discussion on Electrolysis of Fused Salts .....	154
Sykes, W. Manufacture of Cements for Building, Paving, &c. (P) .....	396
Syngun, R. R. See House .....	1068
Sahay, S. Solids of Nitrogen .....	61
Sater, H. Influence of Calcium Salts on Yield of Sugar from Masses of Sugar .....	465

# T

Tafel, J. A Colour Reaction of Acid Amides .....	177
Action of Sodium Peroxide of Acids in Alcoholic Solution .....	885
Behaviour of Sodium Peroxide towards Alcohol .....	1495
Taffin, E. O. Composition for Use as Food, and Methods of Preparation Same (P) .....	752
Tambor, J. See Kostanecki .....	797
Tanner, R. C. Treating Sewage by Ferric Compounds (P) .....	538
Treatment of Sewage and other Foul Waters (P) .....	832
Treatment of Sewage Sludge (P) .....	832
Tarret, C. Carbohydrates of the Jerusalem Artichoke .....	52
Pinein: a Glucoside from the Foliage of the Pine .....	1217
Stability of Solution of Corrosive Sublimates exposed to the Atmosphere .....	657
Tat, P. Discussion on Rectification of Oil of Vitriol .....	210, 211
Methods for Rectifying Oil of Vitriol .....	296
Note on Rectification of Vitriol .....	473
Tate, G. The Fermentation of Dextrose, Rhamnose, and Mannitol .....	268
Tatham, E. and J. E. See Caldwell .....	536
Tatlock, R. R. Discussion on Waters for Dietetic Purposes .....	501
Heating Power of Smoke .....	351
And Thomson, R. T. On the Suitability of Waters for Dietetic Purposes .....	497
Tattersall, W. A., and Pearson, W. E. Apparatus for Cleaning Cotton Seeds (P) .....	893
Tauber, E. Nitrohydroxy- and Nitro-amido-azo Compounds .....	28
Taurin, F. H. See Pearson .....	1269
Tausig, E. Extraction of Metals by Means of Electricity (P) .....	403
Purification of Metals in their Moulds (P) .....	401
Taylor, H. J. W. An Improved Soap (P) .....	1208
Taylor, J. Apparatus for Raising Liquids and Semi-liquids (P) .....	531
Taylor, S. Apparatus for Obtaining more Perfect Combustion of Fuel (P) .....	133
Taylor, T. H. See Cochrane .....	741
Taylor, W. R. Application of Aluminium and its Alloys (P) .....	42
Teehn, N. Improved Laboratory Burner (illus.) .....	277
Tedesco, A. L. Manufacture of Sugars, Syrups, Alcohol, and Beer (P) .....	896
Tehrune, R. H. Plant for Granulating Slag .....	522
Terne, B. See Williams .....	842
Terry, H. L. Discussion on Analysis of India-Rubber Articles .....	185
Discussion on Vulcanisation of India-Rubber .....	17
Tessen, E. Compounds for Removing Old Paint (P) .....	1269
Test, W. H. See Stone .....	596
Tetaz, M. Printing Processes of the Japanese .....	1194
Tname, H. R. See Picard .....	256
Thiel, P., and Schinutze, K. Improved Polishing Medium (P) .....	407
Theodor, K. Meal of Sunflower Cake .....	960
Thew, W. H. See Ekin .....	828
Thiel, O. See Bertrand .....	816
Teufel, H. See Fischer .....	1231
Thilo, J. Manufacture and Compression of Nitrous Oxide Gas .....	970
Thioferri, H. Electro-Plating (P) .....	45
Manufacture of Metallic Oxides and Compounds (P) .....	158
Thomlinson, W. Treatment of Pulverised Ore (P) .....	889
Thompson, C. J. See Tichborne .....	663
Thompson, W. G., and Moore, J. Manufacture of a Diamide Base and of Tetracyclic Colouring Matters therefrom (P) .....	354
Thompson, W. P. Discussion on Aluminium Alloys .....	40
Discussion on Invention .....	1026
Thomson, R. T. Discussion on Waters for Dietetic Purposes .....	501
See Tatlock .....	497
Vinegar: its Manufacture and Analysis .....	1058
Thomson, W. (Baron Kelvin.) Improvements in Balances .....	66
Thomson, W. Appliance for Washing Photographic Plates and Prints (P) .....	659
Discussion on Oxidation and Corrosion of Iron and Steel .....	120
India-Rubber. (A Critical) .....	473
Notes on Oxidation and Corrosion of Iron and Steel .....	118
Thorne, L. T. Oxy-Oil Gas, and its Use for Purposes of En- richment .....	158
Thornhill, C. L. M. The. See Riley .....	728

	PAGE
Thorpe, T. E. Discussion on Oxidised Linseed Oil .....	1022
Eulogium on Dr. Alder Wright .....	1019
Proceedings of Annual General Meeting .....	699
See Hummel .....	803
Thorpe, W. Discussion on Colorimetric Analysis .....	316
Discussion on Dissolved Organic Matter in River Waters .....	321, 322
Discussion on Electrolysis of Fused Salts .....	455
Discussion on Estimation of Arsenic in Copper .....	326
Discussion on Japanese Shiromé and Copper .....	471
Discussion on Sodium Peroxide Explosion .....	199
Discussion on Sterilisation of Liquids .....	1137
Discussion on Strength of Leather .....	592
Discussion on the Colour of Brass .....	595
Discussion on the Distillation of Peat .....	607
Discussion on the Theory of Dyeing .....	100
Discussion on Treatment of Gold Ore in the Transvaal .....	338, 340
Proceedings at Annual General Meeting .....	698
Threlfall, G. See Thwaite .....	717
Thum, A. Hyponitrous Acid .....	35
Thwaite, B. H. Electro-Fusion of Steel and its Alloys (P) .....	528
Manufacture of Steel, and Refining Fluid Iron (P) .....	526
And Threlfall, G. Methods and Apparatus for Generating Gaseous Fuel (P) (illus.) .....	717
Tibbitts, J. B. The Sealing of Wires in Glass (P) .....	545
Tichborne, C. R. C., and Thompson, C. J. Determination of Butter Fat in Milk .....	663
Tiemann, F. See Laire, G. de .....	272
And Kruger, P. The Odoriferous Principle of the Violet .....	272
And Semmler, F. W. Compounds of the Citral (Geraniol) Group .....	417
Tiemann, J. C. W. F. Converting Compounds of the Citral Series (P) .....	972
New Chemical Substance from Orris-root (P) .....	542
Substances for Manufacture of Perfumery and Aromatics (P) .....	963
Tietz, W. See Kämig .....	836
Tilden, W. A., and Forster, M. O. Combination of Hydro- carbons with Picric Acid and other Nitro-Compounds .....	172
Tilghman, B. C., jun. Method and Apparatus for Detecting and Measuring Inflammable Gases in Air (P) (illus.) .....	1052
Timmis, W. Receptacles for Storage of Compressed Air, Gas, and Volatile Liquids (P) .....	788
Timpe, H. Soxhlet's Aerometric Method of Determining Fat in Milk .....	1104
Tipper, L. C. Preparation and Treatment of Animal and Vege- table Substances for use as Food (P) .....	752
Tistschenko, W. Products of Distillation of Petroleum Tar .....	1053
Titaner, D. The Manufacture of Porous Cells .....	888
Tobias, G. Preparation of a New $\beta$ -naphthylaminesulphonic Acid and of $\beta$ -naphthylamine, and Preparation of Salts of $\beta$ -naphthylsulfanilic Acid from the former (P) .....	800
Tocher, J. F. Discussion on Production of Oil-Gas .....	502
Production of Gas from Paraffin Oils, &c. (illus.) .....	231
Tollens, B. Cause of Birotation of Glucose (Dextrose) .....	165
Tollner, K. F. Drying Apparatus (P) .....	714
Tornoe, H. The Ebullioscope and its Use for Estimating Alcohol in Beers .....	762
Torres, J. B. Metallurgical Furnaces (P) .....	1067
Totr, J. New Method of Estimating Yellow Phosphorus .....	420
Tourrette, E. Artificial Whalebone (P) .....	657
Traube, H. Preparation of Anhydrous Crystalline Silicates .....	518
Travers, M., and Plimpton, R. T. Metallic Derivatives of Acetylene .....	277
Trillat, A. Detection and Estimation of Formaldehyde .....	177
Trillach, H. Preparation of Substitutes for Coffee (P) .....	656
Trimble, H. The Tannins, Vol. II. .....	550
And Peacock, J. C. Preparation of the Oak Tannins .....	59
Truelle, A. Study of a Variety of Apple used for Cider-making .....	266
Trug, A. Method and Apparatus for Extracting Gold and Silver (P) .....	527
Truman. Discussion on Commercial Analysis of Lard .....	709
Tugby, H. Manufacture of Stoneware Sanitary Pipes, &c. (P) .....	735
Turney, F. N. Cleaning Wool, and Apparatus therefor (P) .....	633, 944
Treating and Purifying Sod and other Oils (P) .....	46
Twyford, T. W. Glazing and Enamelling Heavy Earthenware Goods (P) .....	396
Twynn, T. Utilisation of Sewage Sludge (P) .....	1084
Tyrer, T. Discussion on Improved Specific Gravity Bottle .....	324
Discussion on Sodium Peroxide Explosion .....	199
Discussion on the "Fertilisers and Feeding Stuffs Act" .....	117
Eulogium on Dr. Alder Wright .....	1020
Proceedings of Annual General Meeting .....	692, 693, 694
Speech at Annual Dinner .....	705

# U

Ueling, E. A., and Steinbart, A. Pyrometers (P) (illus.) .....	175
Ullmann, C. Apparatus for Valuation of Manganese Dioxide by Bunsen's Method (illus.) .....	979

	PAGE
Flück, G. Compound for Production of Indigo upon Fibre...	807
Elzer, F., and Holler, A. Naphthalene of Substituted Amino-phenols.....	799
Engerer. Modern Perfumery.....	1177
Union Chemical Co. Apparatus for Electrolytic Production of Soda and Chlorine (P).....	257
Erick, W. P. B. Casting Solid Ingots of Steel (P).....	492
Esher, E. P. Storage Batteries (P).....	162, 162

## V

Van Berkel, A. Process and Apparatus for Extraction of Carbonic Acid (P).....	38
Vander Hoop, J. F. H. Decorative Paper for Walls, &c. (P) ..	1071
Vanderpoel, F. Dropping Flask for Standard Solutions (illus.)	662
Van der Weyde, P. H., and Lugo, O. Manufacture of Sugar (P).....	265
Van de Velde, A. Colloidal Lead Chloride.....	428
Van Furman, H. Purchasing Silver, Gold, and Lead Ores....	539
Van Laer, H. Studies on Secondary Fermentation and "Frets".....	535
Van Ruyambeke, J. Distilling Glycerin, and Treating the Residue (P).....	1208
Vareune, E. New Apparatus for Fractional Distillation (illus.)	761
Vaubel, W. Behaviour of Triphenylmethane Colouring Matters to Nascent Bromine.....	876
Estimating Naphthol- and Naphthylamine Sulphonic Acids	548
Vautin, Claude. Discussion on Electrolysis of Fused Salts.....	455
Discussion on Japanese Shirome and Copper.....	471
Electrolytic Production of Alloy of Lead and Sodium or Potassium (P).....	714
Manufacture of Aluminium Sulphide (P).....	148
Method and Apparatus for Electrolysis of Saline Solutions (P).....	256
Process and Means for Production of Caustic Alkali (P)....	358
The Electrolysis of Fused Salts (illus.).....	418
Vedrdi, D. Discrimination of Waters.....	897
Veley, V. H. The Inertness of Quicklime. II. The Interaction of Chlorine and Lime.....	250
Velna, G. de. Manufacture of Gas, Fuel Blocks, and Coke (P) ..	243
Verdun, H. F. Compositions of Camphor with other Substances (P).....	57
Verley, A. See Otto.....	1219
Very, J. B. J. See de Pursae.....	499
Vetere, V. di. Detection of Castor Oil in Olive Oil.....	981
Vicuna, A. O. Process and Apparatus for Copper Smelting and Refining (P).....	644
Vidal, R. Manufacture of Colouring Matters (P).....	941
Vieille, P. The Agglomeration of Explosive Substances.....	1089
Researches on Manner of Combustion of Ballistic Explosives.....	659
Vigier, J. Method and Apparatus for Preserving Meat, Cereals, &c. (P).....	168
Vignon, L. Note on Different Theories on Dyeing.....	248
Stability and Preservation of Dilute Solutions of Corrosive Sublimates.....	171
Stability of Dilute Solutions of Corrosive Sublimates.....	1087
And Lisley, P. Action of Nitric and Nitrous Acids on Silk	32
Villard, P. Physical Properties of Pure Nitrous Oxide.....	1087
Villavecchia, Y., and Fabris, G. Use of Furfural for Detection of Sesame Oil.....	69
Villedieu, C. See Clapham.....	390, 728
Villiers, A., and Borg, F. Action of Zinc and Magnesium upon Metallic Solutions.....	68
And Fayolle, M. A Reaction of the Aldehydes. Differentiation of Aldoses and Cetroses.....	1068
And Fayolle, M. Detection of Hydrochloric Acid....	1091
And Fayolle, M. Detection of Traces of Chlorine.....	1092
And Fayolle, M. Testing for Hydrobromic Acid.....	1092
And Fayolle, M. The Estimation of Iodine (illus.).....	1093
Villon, A. M. Carnauba Wax.....	744
Continuous Purification of Oils.....	745
Manufacture of Copper-plate Ink.....	721
Manufacture of Pressure Lubricants.....	745
Purification of Water.....	271
Tanning with Extracts.....	746
Vinassa, E. Saffron and its Adulterants.....	71
Viollette, C. Analysis of the Butters of Commerce.....	54
Virgo, W. H. The Cyanide Process.....	739, 950
Virolleand, L. Bleaching and Purifying Refuse Greases.....	1068
Vis, G. N. Isomers and Analogues of Analgene.....	273
Reactions and Constitution of Morphine.....	284
Vitali, D. Poisonous Action of Martius Yellow, and its Detection.....	423
Vitoux, G. Electrical Purification of Sewage on the "Hermit" System.....	753

	PAGE
Vivien. Estimation of Crystalline Sugar in Masses cutes.....	1249
The Soda-Baryta Process.....	169
Vivien, A. Estimation of Quantity of Juice in Beetroots.....	264
Vivien and Dupont. Preparation of Alcohol from Apples.....	962
Vogel, C. jun. Producing Light Prints on Dark Grounds (P) ..	390
Vogt, J. H. L. The Nickel Industry.....	940
Volney, C. W. Apparatus for Fractional Distillation (illus.) ..	662
Von Schroeder. Extraction of Tannin from Myrabolans.....	1075
Water contained in Air-Dried Bark-Tanned Leather.....	1075
Water-Content of the Common Tanning Materials.....	1072
And others. Estimation of Sugar, the Sugar-Content of Tanning Materials, Extracts, &c.....	1191
And Passler, J. Decomposition of Hides during Tanning.....	49
Voorhees, C. G. See Osborne.....	828
Vortmann, G. Electrolytic Analytical Estimation and Separation (illus.).....	68
Voswinkel, A. Preparation of Basic Organic Bismuth Salts ..	273
Vulpus, G. Moist Iodoform.....	962

## W

Wachter, H. A Bleaching Preparation (P).....	656
Wagner, L., and Marr, J. Process and Apparatus for Purifying Liquids (P).....	751
Wahl and Hantke. The Albuminoids contained in Worts and Beers.....	895
And Nilson. Effect of Albuminoids in Manufacture of Beer.....	896
Wahlm, A. Centrifugal Separators for Cream and Butter (P) ..	58
Wainwright, J. H. Discussion on Detection of Soap in Lubricants.....	1178
Waite, C. N. Electrolytic Cells and Diaphragms therefor (P) ..	959
Wallbaum, H. See Bertram.....	756, 756, 756
Waldbaur, R. See Berger.....	831
Waldstein, M. E., and others. Dyeing Fibres (P).....	947
Walker, A. T. Method and Apparatus for Purifying Water (P).....	169
Walker, Sir B. W. See Cowper-Coles.....	1067, 1067, 1067
Walker, W. H. See Briant.....	55
Walker, W., jun., and others. Primary Voltaic Batteries (P) ..	1296
Walker, W., jun., and Wilkins, F. R. Primary Voltaic Batteries. (P).....	529
Wallace, J. O. See Ward.....	497
Waller, E. Manufacture of White Lead (P).....	163
Walter, J. Condensers for Laboratories (illus.).....	791
Preparation of Quinoline.....	758
The Prevention of Scale in Boilers.....	622
The Wearing of Metal Plates by Action of Steam Jets.....	134
Walter, O. Stains on Woollen Cloth from Ammonio-Cupric Oxide.....	246
Walters, W. M. Composition and Treatment of Substances for Use as Glue, Putty, and Cement (P).....	497
And Stone, F. K. Coatings for Iron and Steel (P).....	963
Walton, J. V. Apparatus for Manufacture of White Lead (P) ..	162,
163, 163	
Wanklyn, J. A., and Chapman, E. T. Water Analysis: A Practical Treatise.....	768
And Cooper, W. J. Distillation of Petroleum (P) (illus.) ..	720
And Cooper, W. J. The Method of Fractional Distillation.....	627
Wanner, M. Refrigerating Liquid (P).....	1181
Warl, G. J. Discussion on Extinction of Flames by Proportions of Carbon Dioxide, and Nitrogen in Air.....	1155
Ward, W., and Wallace, J. O. New or Improved Paint (P) ..	497
Warden, C. J. H., and Billings, W. A. Treatment and Packing of Meats, &c., for Use as Army Rations (P) ..	169, 169
Warner, H. G. Combined Diester, Drier, Washer, &c. (P) ..	594
Warwick, F. W. Preparation of Ferrous Salts (P).....	275
Warzee, E. Separation and Precipitation of Salts of Iron and Zinc.....	1196
Watel, E. Improving or Preserving Oils and Fats (P) (illus.) ..	531
Watkins, A. Method and Apparatus for Tinting Photographs Development.....	1219
Watkins, F. Coating Metal Sheets with Tin, &c. (P).....	1291
Watson, Eric E. Calculation and Composition of Charges for Smelting Lead and Silver Ores.....	1149
Watson, jun., G. See Mills.....	238
Watson, jun., J. See Seldner.....	533
Watson, M. Preparation of Malted Pulse Food.....	279
Watson, W. A New Medicinal Preparation for Leprosy, Cancer, Eczema, &c.....	417
Watt, G. Report on Indigo Industry in British India.....	994
Wauters, J. Disappearance from Beer of Added Salicylic Acid and Saccharine.....	964
Webb, W. H. Apparatus for Cooling Liquids (P).....	378
Weber. Discussion on the Theory of Dyeing.....	355

	PAGE		PAGE
Weger, C. O. A New Class of Lake Pigments .....	1151	Wilkins, F. R. See Walker .....	529
Discussion on Analysis of India-Rubber Articles .....	485	See Walker, jun. ....	1206
Discussion on India-Rubber .....	476	Wilkinson, T. B. Discussion on Dyeing Leather with Basic Colours .....	487
Discussion on Resist and Discharge Styles with Aniline Black .....	487	Discussion on Indian Hye-stuffs .....	553
Discussion on Substantive Dyes and Dyeing .....	127	Wilks, W. R. See Abbott .....	248
Discussion on Vulcanisation of India-Rubber .....	17	Will, F. See Buttner .....	539
On the Analysis of India-Rubber Articles .....	176	Will, H. Note on "Gluten Particles" in Wort, Beer, and Yeast .....	937
On the Vulcanisation of India-Rubber .....	11	Williams, D. See Wyde .....	1059
The Manufacture of India-rubber Waterproof Cloth .....	33	Williams, M. Manufacture of Cement Mortar (P) .....	1062
See Frankenburg .....	725	Williams, R. G. Colouring Matters (P) .....	724
Substantive Dyes and Dyeing .....	120	Williams, S. D. See Clapp .....	1201
Weber, C. S. S. Automatic Alarm for Water Ovens and Baths (illus.) .....	205	Williams, W. J., and others. Determination of Phosphoric Acid .....	842
Weber, W. Apparatus for Purifying Water (P) .....	169	Williamson, J. A. Determination of Lead in Small Quantities .....	1098
Manufacture of Cement (P) .....	254	Willstätter, R. See Einhorn .....	1217
Treating the Effluent from Purified Sewage (P) .....	169	Wilson, C. W. See McLean .....	888
And Moore, T. Pharmaceutical Preparations for Anaemia and Weak Throats (P) .....	161	Wilson, F. Discussion on Sterilisation of Liquids .....	1193
Wechsler, M. The Separation of Volatile Fatty Acids .....	178	Wilson, W. A. and R. H. C. and Priest, W. B. Manufacture of Paints (P) .....	1071
Welding, M. H. The Alloys of Iron and Nickel .....	955	Wilson, W. V. and Lundy, J. J. Treatment of Lead Sulphate for Use as a Pigment (P) .....	743
Weeks, J. D. The Coke Problem in England and America .....	859	Windeck, L. Apparatus for Pasteurising Beer .....	267
Weerschelder, R. On the Estimation of Copper as Copper Sub- sulphide .....	67	Windisch, Influence of Mashing Process on Attenuation .....	967
Wehner, C. Producing Artificial Citric Acid (P) .....	275	Aluminium Fod as a Beer-Clarifying Agent .....	969
Synthetic Formation of Citric Acid .....	57	A Volumetric Method for Estimating Sulphuric Acid .....	905
Weibull, M. On the Determination of the Fat in Bread .....	905	Windus, W. See Worsley .....	510
Wienberg, E. A. L., and others. Eliminating Oxides of Zinc, Iron, and Copper from Cobalt Oxide, and Apparatus therefor (P) .....	887	Winkler, E. Apparatus for Filtering and Purifying Water (P) .....	577
Wienrich, M. Manufacture or Restoration of Filtering Media (P) .....	249	Winter, A. W. Compound Edible Fats (P) .....	1083
Wier, J. and G. Separating Oily or Fatty Matters from Water (P) .....	504	Wise, G. H. See Aspinall .....	1208
Weiss, F. The Phenylester of Benzamide Phenyl Acetic Acid .....	57	Wiskirchen. The Alcohol Extraction of Beetroot .....	1226
Weldon, L. Dyeing Machines (P) .....	249	Wisse, W. J., and Schneller, A. Electrolytic Decomposition of Hydrocarbons, &c. (P) .....	169
Wells, F. R. Colouring Paper and Cardboard (P) .....	331	Witt, O. N. Die Chemische Industrie auf der Columbischen Weltausstellung zu Chicago, &c. ....	768
Wendringer, M. A Simple Viscometer (illus.) .....	1220	And Johnson, L. S. Azo Derivatives of Hydroquinone .....	28
Wendt, H. O. See Gerold .....	275	Wittman, S. Device for Filtering Water (P) .....	1083
Wendland, A. Removing the Green Colour from Paraffin, Vaseline, and Lubricating Oils (P) .....	829	Wohl, A. Technical Method of Preparing Aniline .....	1188
Wernegreen, J. P. See Hingston .....	1216	The Reduction of Nitro Compounds .....	576
Werner, L. Machines for Removing Stiff Water or Waste Hairs from Belts (P) .....	1209	Wolf, Netter, and Jacobl. Tinning Articles of Cast Iron (P) .....	42
Werner, P. Report on Pokorny's Paper on Synthesis of Azo Colours on Animal Fibres .....	388	Wolfeauer, F. Estimation of the Titre of Fallow .....	998
West, G. N. See Roux .....	725	Valuation of Fallow by Determination of Solidification Point of its Fatty Acids .....	181
Westcott, J. T. Apparatus for Manufacture of Water-Gas (P) (illus.) .....	1051	Wolfenstein, R. Conium Alkaloids .....	1217
Weston, J. Determination of Nickel in Steel .....	669	Wolkowicz, A. Estimation of Carbonic Acid .....	904
Westfälisch-Anhaltischen Sprungstoff Aktien Gesellschaft. Hygroscopic Paint or Coating for Packing Cases (P) .....	276	Wolters, W. Concentrating Sulphuric Acid in Lead Vessels (P) .....	887
Plugging Compound for Blasting Cartridges (P) .....	61	Wood, J. T. Discussion on Fermentation in the Leather Industry .....	222
Weston, D. See Lowe .....	141	Fermentation in the Leather Industry (illus.) .....	218
Westpal, C. Manufacture of Incombustible Burners (P) .....	957	Wood, R. W. Affinity-Constants of Weak Acids .....	826
Whalley, G. Compound for Purifying Sewage, &c. (P) .....	654	The Action of Salts on Acids .....	615
Wheeler, C. Y. See Slocum .....	1071	Woodcock, R. C. Discussion on Detection of Soap in Labri- cants .....	1178
Wheeler, E. H. Electrodes and Cells for Secondary Batteries (P) .....	647	Woodhead, G. Sims. See Irvine .....	131
Wheeler, H. A. The Calculation of the Fusibility of Clays .....	336	Woolley, G. O. Apparatus for testing Paper (P) (illus.) .....	1222
Wheeler, S. Improved Paper or Textile, and Method of Pro- ducing same (P) .....	539	Woosnam, R. W., and The Dairy Supply Co. Apparatus for Estimating Fat in Milk (P) .....	1091
Whitaker, A. Discussion on Substantive Dyes and Dyeing .....	127	Worcester Royal Porcelain Co. and others. Manufacture of Pottery or Earthenware (P) .....	1197
Whitaker, T. Discussion on Substantive Dyes and Dyeing .....	127	Worltitz, E. F. See Berger .....	834
White, J. Barrington. The Manufacture of Prepared Cereals .....	653	Worsley, P. J., and others. Process and Apparatus for Pump- ing and Absorbing Chlorine Gas (P) .....	810
White, W. Preparing Pigment Paper for Photographic Print- ing (P) .....	417	Worsam, H. J. Apparatus for Cooling and Evaporating Brewing Liquids (P) .....	269
Whitehead, W. T. Resists or Discharges for Printing Fabrics (P) .....	320	Wort, G. W. S., and E. W. See Mott .....	153
Resists or Discharges for Printing Fabrics with Aniline Black (P) .....	320	Wotherspoon, J., and Davie, J. Apparatus for Purifying Feed Water (P) .....	505
Whitley, J. See Owens .....	326	Woy. See Seyda .....	981
Whitely, R. L. Discussion on Extinction of Flames by Gaseous Mixtures .....	1156	Wrampelmeyer, E. Valuation of Ground Phosphatic Slags .....	422
Whiting, J. E. See Salmon .....	745	Wright, C. R. Alder. Antiseptic and Disinfectant Material (P) .....	271
Wichelhaus, H. $\alpha$ - and $\beta$ -Naphthalene Indigo .....	29	Certain Ternary Alloys. Part VIII. ....	321
Wichmann, R. Means for Producing Effervescent Beverages (P) .....	898	Composition and Constitution of certain Alloys .....	1014
Wicking, S. G. See Nightingale .....	146	Discussion on Strength of Leather .....	592
Wiernik, J. Concentration of Mother-Liquors from the Manu- facture of Alum .....	731	Discussion on the Colour of Brass .....	594
The Function of Calcium Chloride and the Formation and Constitution of "Stiff Charges" in the Weldon Process .....	806	Wright, F. W. See Gaskell, jun. ....	1196
Wiese, G. E. Manufacture of Liquid Glue (P) .....	405	Wright, R. See Farr .....	983
Wild, H. Improvements in the "Thomas" Process (P) .....	401	Wright, S. H. Method and Apparatus for Exhausting (P) .....	1182
Wiley, H. W. Principles and Practice of Agricultural Analysis .....	289	Production of Butter (P) .....	1083
Wilke, A. Efficiency of Heating by Electricity as Compared with Heating by Combustion .....	790	Wright, W., and Hamond, J. B. Treatment of Zinc Produced by Electrolysis (P) .....	1067
		Wurgart, J. Condensation of Aldehydes with $\alpha$ -Naphtha- quinone and $\alpha$ -Naphthoquinol .....	798
		Wyde, J. R., and Williams, D. Recovery of Copper from Bricks used in the Deacon Chlorine Process (P) .....	1059

## Y

	PAGE
Yagu, N. Apparatus for Sterilising Water (P) .....	113
Yeckney, A. Manufacture or Treatment of Paper (P) .....	302
Young, J. Obtaining Cyanides and Ammonia direct (P) .....	1193
Young, W. Producing Gas from Mineral Oils, Apparatus therefor, and for Producing Water-Gas (P) (illus.)....	934
Production of Gas and Bye-Products, and Apparatus therefor (P) .....	935
Young, W. C. Discussion on Colorimetric Analysis.....	317
Discussion on Dissolved Organic Matter in River Waters .	321
The Natural Diminution of Dissolved Organic Matter in the Water of Rivers .....	318

## Z

	PAGE
Zahn, W. Tanning Hides of Skins (P) .....	822
Zakrzewski, S. von. See Friedländer .....	791
Zaboziecki, R. Terpenes in Petroleum.....	1187
The Waste Liquors from the Refining of Petroleum.....	628
Zawalkiewicz, Z. Estimating Pychnometrically the Densities of Soft Fats (illus.).....	839
Zera, A. Determination of Reducing Action of Organic Substances in Water on Potassium Permanganate .....	427
Ziegler, J. H. Manufacture of New Colouring Matters (P) ...	385
Zikes, H. See Benedikt.....	984
Zimmer, C. Process and Apparatus for Brewing (P) .....	656



# INDEX OF SUBJECTS.

N.B.—In this Index, (P) indicates that the matter referred to is an abstract of a patent; (T.R.) indicates that the matter referred to is in the Trade Report.

A		PAGE
Abrastol, Chemical Action of, on Wine. (Scheurer-Kestner) ..	654	
In Wine, Detection of. (Sangle-Ferrière) ..	177	
In Wines, Detection of. (Briand) ..	1099	
In Wines, Detection of. (Simbaldi) ..	548	
Note on. (Noelting) ..	534	
Acetamide, Volumetric Estimation of. (Kuster) ..	844	
Acenaphthylene, Colour of. (Grabe) ..	27	
Acetamido-Alkylsalicylic Acid, Manufacture of. (P) Abel and Imray. From Kuchler and Bull ..	60	
Acetic Acid, Apparatus for Concentrating or Vaporising. (P) Bonlt. From Peuchen and Clarke ..	251	
Acetone. (Kopp and Grandmouzin) ..	720	
Acetnaphthylene-diamine-sulpho Acids, Production of New. (P) Pitt. From Cassella and Co. ..	801	
Acetol, Manufacture of Salicylic Acid Ester of. (P) Fritsch ..	274	
Acetone and Salicylic Acid in "Sal Acetol." Quantitative Estimation of. (Eckeroth and Koek) ..	288	
And Salicylic Acid in "Sal Acetol." Quantitative Estimation of. (Helling and Passmore) ..	288	
As a Solvent. (Trimble and Peacock) ..	59	
Acetyl, Production of. (P) Von Mering ..	60	
Acetylene, Explosive Nature of. (Meyer) ..	1182	
Metallic Derivatives of. Mercuric Acetylde. (Travers and Plimpton) ..	277	
Acetylde, Mercuric. (Travers and Plimpton) ..	277	
Aceto-4-xtrin. (Lintner and Dull) ..	53	
Acid, Estimation of Free, in Solutions of Per-Salts of the Heavy Metals. (Hoffmann) ..	282	
Acidimetry, Value of Borax as the Basis of. (Salzer) ..	280	
Acidity in Worts, Influence of, on Composition of Low Wines. (Lindet) ..	52	
Acids, Alkalis and Salts. (Class VII.) 34, 151, 249, 394, 513, 634, 729, 805, 884, 947, 1057, 1057, 1195		
Action of Concentrated, on Certain Metals. (Burch and Duleson) ..	525	
Action of Salts on. (Wood) ..	515	
Action of, upon Glass. (Feierstein) ..	1099	
Afinity-Constants of Weak. (Wood) ..	826	
Apparatus for Concentrating Sulphuric, and other. (P) (Dyson) ..	1039	
Apparatus for Recovering and Concentrating Waste. (P) Levinstein ..	152	
Detection of Mineral, in Presence of Organic Acids. (Nickel) ..	423	
Detection of Mineral, in Vinograd. (Grigol) ..	423	
Estimation of the Insoluble Non-Volatile Fatty. (Mellere) ..	983	
Extraction of Pur, from Alkaline Salts. (P) Placet and Bonnet ..	37	
In Alcoholic Solution, Action of Sodium Peroxide on. (Tafel) ..	885	
Manufacture of Various, and Superphosphate of Lime. (P) Prentice ..	250	
Obtaining Sulphonic, free from Inorganic Salts. (P) Helmers ..	508	
Of Beeswax, Extraction of the Free. (Mariet) ..	1207	
Pumps for Raising. (P) Long and Burditt (illus.) ..	930	
Separation of Fatty, from Wool Fats. (P) Krause and Mayer ..	162	
The Separation of Volatile Fatty. (Wechsler) ..	178	
Aconite Alkaloids, On the. Part VIII. On Picroconitine. (Dunstan and Harrison) ..	172	
Alkaloids, On the. Part IX. The Action of Heat on Aconitine. (Dunstan and Carr) ..	172	
Alkaloids, On the. Part X. On the Conversion of Aconitine into Isaconitine. (Dunstan and Carr) ..	172	
Aconitine. (Dunstan) ..	54	
(Freund and Beck) ..	539, 540	
Action of Heat on. (Dunstan and Carr) ..	172	
Conversion of, into Isaconitine. (Dunstan and Carr) ..	172	
Address, Changes of. 3, 94, 197, 397, 447, 573, 690, 784, 868, 927, 1012, 1132		
The Presidential. (Stanford) ..	694	
To Manchester Section. (Carpenter) ..	1026	
To New York Section. (Mason) ..	1167	
To Scottish Section. (Irvine) ..	1038	
To Yorkshire Section. (Smithells) ..	18	
Advice to English Merchants. (T.R.) ..	1109	
Aeration, Effect of, in Brewing. (Briant) ..	534	
Afinity-Constants of Weak Acids. (Wood) ..	826	
Agate, Manufacture of Glass Articles Imitating. (P) Solms-Garuth ..	39	
Agave Americana Fibre ..	802	
Air and Gas, Apparatus for Mixing. (P) Axdorfer and Sass ..	1650	
Apparatus for Enabling One to Breathe Fresh, while in a Noxious Atmosphere. (P) Ash ..	793	
Apparatus for Heating. (P) Fudieker ..	1181	
Apparatus for Netifying Presence of Gases in. (P) Hardy ..	1091	
Bath, A New Form of. (Sauer) (illus.) ..	662	
Filter for. (P) Moeller ..	714	
Hydrogen Dioxide in Atmospheric. (Hosva) ..	753	
Method and Means for Supplying Sterilised, to Rooms. (P) Effront ..	833	
Or Gas, Apparatus for Carburetting or Enriching. (P) Love and Weston ..	141	
Pump, New Form of Mercury. (Kahlbaum) (illus.) ..	760	
Tanks for Storing Compressed. (P) Tiumis ..	798	
Alarm for Water Ovens and Water Baths, An Automatic. (Webster) (illus.) ..	205	
Albumen Derivative, Obtainment of, an Iron, from Animal Organs. (P) Wilcox. From Boehringer and Soehne ..	658	
Albuminoids contained in Worts and Beers. (Wahl and Hantke) ..	893	
Effect of, in Manufacture of Beer. (Wahl and Nilson) ..	896	
Albuminoses, Contribution to the Knowledge of. (Schrotter) ..	273	
Alcohol, Behaviour of Sodium Peroxide towards. (Tafel) ..	1195	
Butylic, Fermentation and the Butyl Ferment ..	969	
Determination of Oil of Turpentine in. (Perl) ..	427	
Estimation of, in Beers by Means of the Ebullioscope. (Torneo) ..	762	
Influence of, on Bactericidal Action of Phenol and Corrosive Sublimate. (Lensi) ..	903	
In Germany. (T.R.) ..	676	
In Germany, The Denaturing of. (T.R.) ..	185	
Manufacture of. (P) Lake. From Tedesco ..	896	
Preparation of, from Apples. (Vivien and Dupont) ..	902	
Production of, in Russia. (T.R.) ..	910	
Rectification of. (Sorel) ..	1081	
Alcohols, Detection of the Higher, in Spirits of Wine. (Bardy) ..	287	
Filters used in the Production of. (P) Iseli ..	377	
The Glucosides of the. (Fischer) ..	167	
Aldehyde, Manufacture of Aromatic Sulphuryl Derivatives of Proteochemie, and Conversion into Vanillin. (P) Pitt. From The Chemische Fabrik auf Aetion vormalis Schering ..	1218	
Of Lemon-Grass Oil, The. (Barbier and Bouvauit) ..	1096	
Preparation of a Crystalline Polymeric Base from Amido-Acetic. (P) Pitt. From The Chemische Fabrik auf Aetion vormalis Schering ..	1218	
Aldehydes, A Reaction of the. (Villiers and Fayolle) ..	1098	
Condensation of, with $\alpha$ -Naphthaquinone and $\alpha$ -Naphthaquinol. (Wurgitt) ..	798	
Aldoses and Cetroses, Differentiation of. (Villiers and Fayolle) ..	1098	
Algiers, Phosphate Deposits of. (T.R.) ..	1108	
Alimentary Substances. See Foods.		
Alizarin Cyanine, Dyeing Wool by Means of Alkaline Solutions of. (Schubel) ..	388	
The Formamides of. (Fruithomme and Rabaut) ..	143	
Pastes, Rapid Method of Determining Shade of. (Perkin) ..	496	
Alkali Cyanides, Production of. (P) Siepermann ..	250	
Electrolytic Production of. (P) Hargreaves and Bird ..	528	
Electrolytic Production of Caustic. (P) Vautin ..	958	
Lands in North China. (T.R.) ..	910	
Metals, Oxidation of the. (Holt and Sims) ..	926	
Method and Apparatus for Manufacture of. (P) Hargreaves and Bird ..	250	



	PAGE
<i>Alkali—cont.</i>	
Production of caustic. (P) Lange and Maxwell Lab.	734
Production of caustic and Lead Chloride. (P) Maxwell-Lange and Lange	734
Recovery of, from a Waste Product. (P) Murray and Baird	1051
Salts, Electrolytic Decomposition of. (P) Sinding Larsen	1206
Test for Kerosene, Naphtha Products, Hydrocarbon Lubricants, &c. (Lassenko and Stanawski)	177
Waste, Manufacture of Cement from. (P) Rigby, Neill, and Carr	638, 948
Works, Thirtieth Annual Report on.	729
<i>Alkalis. (Class VII.)</i>	34, 151, 249, 390, 533, 631, 729, 808, 884, 947, 1057, 1195
Action of, on Paranitrotoluene and its Sulphonic Acid. (Fischer and Hepp)	29
Decomposition of Invert Sugar by. (Prinsen-Geerlings)	119
Minimum Electro-motive Force Required for the Electrolysis of Dissolved Salts of. (Nourisson)	742
<i>Alkaloids. (Class XX.)</i>	57, 170, 272, 415, 533, 657, 755, 831, 902, 970, 1085, 1216
Conium. (Wolffenstein)	1217
From the Seeds of Lappin Albus. (Saldani)	757
German Trade in. (T.R.)	1112
In Alkaloidal Tinctures, Methods for Determination of. (Farr and Wright)	983
In Cinchona Bark, Estimation of Total. (Keller)	1101
In Nux Vomica Seeds, Estimation of. (Keller)	1105
In the Root of Corydalis Cava. (Fremd and Joseph)	59
Of Bark of Pomegranate Root. (Chamian and Silber)	116, 1217
Of Corydalis Cava, The Corydalline. (Dobije and Lauder)	1217
Of Gelsemium Sempervirens. (Cushny)	59
Of Papaveraceae. (Schmidt)	58
Of the Papaveraceae. (König and Tietz)	836
Of the Papaveraceae: Codeine. (Goehlich)	757
The Atropine. (Hesse)	416
The Estimation and Separation of the Cacao. (Kunze)	178
The Pericarp. (Hesse)	416
Allevard, Dressing Spathic Iron Ore at. (Gromier)	1044
<i>Alloy, A New Metallic. (P) Cox</i>	158
A Metallic. (P) Dixon and Skinner	957
An Aluminium. (P) Roman	956
An Improved Metallic. (P) Sage	645
Of Lead and Sodium or Potassium, Electrolytic Production of an. (P) Vautin	741
<i>Alloys, Coating Iron and Steel with. (P) Fenby and Moore</i>	400
Composition and Constitution of Certain. (C. R. Alder Wright)	1014
Electrolytic Production of Chromium. (P) Placet and Bonnet	646
For Manufacture of Wire. (P) Macdonald	523
In a Voltaic Cell, Electro-Motive Force of. (Laurie)	1204
Influence of Aluminium upon the Carbon in Ferro-Carbon. (Hogge) (illus.)	955
Manufacture and Industrial Value of Aluminium. (Daggar)	4
Manufacture of Metallic. (P) Howarth	815
Of Aluminium, Application of. (P) Taylor	42
Of Copper, On New. (Von Knerre)	815
Of Iron and Nickel. (Osmond)	649
Of Iron and Nickel, The. (Wedding)	955
On certain Ternary. Part VIII. (Alder Wright)	321
On the Cupellation of Bismuth-Silver. (Smith)	765
Pickling, to Remove Impurities. (P) Smith	526
Researches on the Properties of. (Roberts-Austen)	154
Separation and Estimation of Tin and Antimony in. (Mengin)	1224
The Ligation of Silver-Copper. (Matthey)	889
<i>Alterations to Bye-Laws.</i>	692
Alum, Concentration of the Mother-Liquors in the Manufacture of. (Wiernik)	731
Alumina, Basic Sulphate of. (Ferguson)	635
Manufacture of Pure, and Bye-Products. (P) Peniakoff	947
Manufacture of Salts or Compounds of. (P) Kessler	38
<i>Aluminium, Action of Iron Sulphide on.</i>	1063
Action of, on Cast Iron	737
Action of, on Sulphates and Chlorides. (Rossel and Frank)	515
Alloy. (P) Roman	956
Alloys, Manufacture and Industrial Value of. (Daggar)	4
And Aluminous Compounds, Manufacture of. (P) Peniakoff	947
Application of. (P) Taylor	42
Bronze "Doctor" for Printing Machines	388
Coating Paper with. (P) Ibbotson	170
Decomposition of Sodium Peroxide by. (Rossel and Frank)	516
Electrometallurgy of	43
Employment of, in Refining Metals	955
Foil as a Beer-Clarifying Agent. (Windisch)	969
Flux for Soldering. (P) Thompson, From Nicolai	890
Impurities of Commercial. (Moissan)	1190
Influence of, on the Carbon in Ferro-Carbon Alloys. (Hogge) (illus.)	955
Manufacture of Salts or Compounds of. (P) Kessler	38
Method for Plating. (Göttig)	1200
Modern Processes for the Manufacture of. (Bucherer)	42
Or its Diluent Alloys, Coating Metals with. (P) Broadwell	815
Preparation of Crystalline Carbide of. (Moissan)	1199
Process for Soldering. (P) Singer, Donat, and Kirchheim	645

<i>Aluminium—cont.</i>	
Qualitative Detection of. (Neumann)	849
Solder for. (P) Nicod	645
Sulphate, Manufacture of. (Jursch)	516
Sulphide, Formation of. (Gorny)	742
Sulphide, Manufacture of. (P) Vautin	178
The Reduction of, as a Scientific Problem. (Bucherer)	522
U.S. Report on the Manufacture of. (T.R.) (illus.)	79
Use of, in Lithography. (T.R.)	201
<i>Amalgam, Influence of Fractional Electricity on Formation of an. (Stuats)</i>	43
Amalgamating Apparatus. (P) Green	102
America, Manufacture of Sulphuric Acid in. (Lange) (illus.)	881
Preparation of Carbon Black from Natural Gas in. (Cibot) (illus.)	128
The Coke Problem in. (T.R.)	859
Water-Gas in. (Bredel)	715
<i>Aminoalkylalcoholic Acid, Manufacture of. (P) Abel and Murray. From Kuehler and Boff</i>	69
Bases, Manufacture of New. (P) Koroff	622
Cresolsulpho Acid, Production of. (P) Lake. From Oehler	723
Naphtholsulpho Acid, Manufacture of a New. (P) Newton. From The Farb. vorm. Bayer and Co.	943
Naphtholsulpho Acid, Manufacture of 2:7. (P) Abel. From The Acten Gesellschaft fur Anilin Fab.	876
Naphtholsulphonic Acid, Manufacture of a New. (P) Abel. From The Acten Gesellschaft fur Anilin Fab.	32
Naphthol Sulpho Acid, Production of. (P) Pitt. From Cassella and Co.	244
Naphthol-Sulpho Acids, Production of. (P) Newton. From The Farb. vorm. F. Bayer and Co.	800
Phenol Acid, Production of. (P) Lake. From Oehler	723
Phenols, Naphthalens of Substituted. (Ulzer and Haller)	730
Sulpho Acid, Production of a New. (P) Johnson. From The Badische Anilin und Soda Fab.	724
<i>Amine Bases, Recovery of Iron from Residue of the Manufacture of. (P) Bromhead. From Landshoff and Meyer.</i>	42
<i>Ammonia, Action of Gaseous, on Magenta. (Prud'homme and Rabaut)</i>	382
Amount of Sulphate of, produced in the United Kingdom. And Cyanides direct from the Air, Obtainment of. (P) Young	729, 1196
And Magenta Colouring Matters, Compounds formed by. (Prud'homme and Rabaut)	382
Apparatus, Construction of. (P) Davis	948
Apparatus for Obtaining. (P) Lake. From Sternberg	887
Decomposition of Chlorides and Iodides of Amines by. (Prud'homme and Rabaut)	382
Defecation of Beetroot Juice with. (Hanus)	263
Estimation of, by means of the Colorimeter. (Hosva)	842
Estimation of, in Liquids containing Sulphur or Cyanogen. (Henry)	761
Explosives from Nitrate of, and Resins. (P) Abel. From König	543
From Fuel, Apparatus for Obtaining. (P) Mond (illus.)	938
-Gas, Action of, on Peroxides. (Michel and Graudmoulin)	37
Influence of Acid Molybdate of, on Rotatory Power of Rhamnose. (Gernez)	1226
Manufacture of Carbonate of. (P) Raspe	395
Method and Apparatus for Producing. (P) Boul. From Cowen (illus.)	119
Production of, and Monocyclic Phosphate. (P) Hurter and Omholt	409
Soda Process, The Commercial Production of Chlorine by the. (Bide) (illus.)	509
Sodium Hydroxide and Chlorine, Process and Apparatus for Manufacturing. (P) Kellner	152
Washers for Separating, from Gas. (P) Cripps (illus.)	379
<i>Ammoniacal Liquor, Apparatus for Distillation of. (P) Pettigrew</i>	734
Liquor, Apparatus for the Distillation of. (P) Spence and Gilchrist	394
Liquor, Valves for. (P) Cripps	508
<i>Ammonio-Cupric Oxide, Stains on Woollen Cloth caused by. (Walter)</i>	946
<i>Ammonium Carbonate, German Trade in. (T.R.)</i>	1111
Formate, Production of, and Hydrocyanic Acid therefrom. (P) De Lambilly	58
Nitrate, Preparation of. (Benker)	515
<i>Ammunition in India. (T.R.)</i>	1115
<i>Amylaceous Materials, Deodorising or Bleaching. (P) Abel. From König</i>	824
Amylodextrin. (Linfner and Dull)	53
Aulene, Isomers and Analogues of. (Vis)	273
(Ortho - E-thoxy - Ana - Mono - Benzoylamido - Quinoline). (Jahn)	1138
<i>Analysis, Employment of "Micro-Chemical" Reagents in. (Leuz)</i>	765
Quantitative Electrolytic. (Classen)	1223
Quantitative Electrolytic. (Rudolf)	1223
Analytical Association in Germany, Proposed. (T.R.)	512
Chemistry and Apparatus. (Class XXIII.) 61, 174, 277, 543, 661, 760, 837, 904, 975, 1090, 1220	418
<i>Andropogon Schoenanthus, Geraniol from Oil of. (Barlier and Bouveault)</i>	1086
Anethol, Transference of Essence of Tarragon into. (Grimaux)	658
Anhydro-ortho-sulphamine Benzoic Acid, Manufacture of. (P) Lang	836

	PAGE
Antides, A.C. Reaction of Acid. (Tafel) .....	177
Aniline and Coal-Tar Colours, German Trade in. (T.R.) .....	1111
Bases, Sulphur Compounds accompanying. (Hantzsch and Piesse) .....	1159
Black, On R. set and Discharge Styles with. (Dreyfus) .....	185
Industry, Preparing Lead Peroxide for the. (Munro) .....	940
Notes on the Oxidation of. (Mills and Macfarlane) (illus.) .....	237
Notes on the Oxidation of. (Mills and Watson, jun.) .....	258
Technical Method of Preparing. (Wohl) .....	1188
Titration of, with Methyl Orange. (Lange) .....	667
Animal Matters, Apparatus for Drying. (P. Currie and Rowlands) .....	714
Substances, Apparatus for Drying. (P. Christy, From Tollner) .....	714
Annual Dinner, The .....	703
Meeting, Proceedings of the Thirtieth .....	621
Anthraquinone, Acid, Manufacture of Monosulphonated. (P. Murray, From La Soc. Anon. des Matieres Colorantes, &c., and Dr. Chapuis) .....	32
Anthraquinolinesquim, Hydroxy-Derivatives of. (Graebe and Phillips) .....	28
Anthraquinone, $\beta$ -, (Lazodius) .....	876
Production of Colouring Matters from. (P. Newton, From The Farb. vorm. Bayer and Co.) .....	723
Antibenzopyrine. (Kissling) .....	944
Antimony and Calcium, Electrolytic Separation of. (Schmucker) .....	280
Blue, (Sehor) .....	40
Discovery of Native, in California. (T.R.) .....	78
In Bolivia. (T.R.) .....	912
Mine in New South Wales. (T.R.) .....	78
Separation and Estimation of, in Alloys. (Mening) .....	1224
Separation of Lead from. (Jannasch and Remmler) .....	66
Antipyretic and Antimorale Preparation. (P. Radlauer) .....	542
Antipyrine, Derivatives of Toly. (Ebert) .....	50
Antiseptic, A New. (P. Pitt) .....	413
Antiseptics and Disinfectants, Manufacture of. (P. Alder Wright) .....	271
Composition of. (P. Hakansson) .....	898
Influence of Mineral on Lactic Fermentation. (Chassevant and Richet) .....	269
Inherent of Ferments to Action of. (Effront) .....	1213
Manufacture of. (P. Newton, From The Farb. vorm. Bayer and Co.) .....	972
Preparation of. (P. Pitt) .....	114
See also under Disinfectants.	
Use of, in the Dyeing. (Class) .....	555
Antitoxin, Behring's Experiments with. (Levinstein) .....	1142
Apparatus. (Class L.) 21, 134, 239, 376, 502, 622, 713, 787, 869, 929, 1017, 1175	
A Simple Extraction. (Buttner) (illus.) .....	118
Construction of Ammonia. (P. Davis) .....	948
Electrolytic. (P. Caldwell) .....	1205
Evaporating. (P. De la Grosse) (illus.) .....	1179
Evaporating Apparatus for Laboratory Use, Vacuum. (Soxhlet) (illus.) .....	977
Evaporating Machine, Hot Air. (P. Newton) .....	871
Evaporators for Salt. (P. Coney) .....	1196
Extraction. (Parker) (illus.) .....	1177
For Amalgamating Metals. (P. Green) .....	902
For Analysis of Gases. (Pfleider) (illus.) .....	63
For Blending Powdered Materials. (P. Detweiler) .....	1181
For Brewing. (P. Zimmer) .....	654
For Burning Cressote or Similar Oil. (P. Allen) (illus.) .....	1050
For Calculating Bicarbonate of Soda into Carbonate. (P. La Soc. Marcheville Dagum of Co.) .....	518
For Cleaning Cotton-Seed. (P. Bibby) .....	1050
For Cleaning Cotton Seeds. (P. Tattersall and Pearson) .....	893
For Collecting Carbonic Acid during Fermentation. (P. Michel and Frank) .....	655
For Concentrating Acids. (P. Dyson) .....	1050
For Concentrating Acids and Liquids. (P. Bradbury) .....	251
For Concentrating Liquids. (P. Kleemann) (illus.) .....	1048
For Concentrating Sulphuric Acid. (P. Levinstein) .....	152
For Concentration or Vaporisation of Acids. (P. Boulton) .....	251
From Penchen and Clarke .....	251
For Condensing Acid and other Vapours. (P. Newton, From Hunt) (illus.) .....	1197
For Continuous Evaporation of Liquids. (P. Harlock) .....	23
For Continuous Refining or Bleaching of Sugar. (P. Prangy) .....	265
For Continuous Sterilisation of Liquids. (P. Herscher) .....	1216
For Controlling Combustion of Solid Fuel. (P. Nobel) .....	874
For Controlling Discharge of Liquids from Tanks. (P. Strype) .....	1180
For Cooling and Forming Glue. (P. Hewitt) (illus.) .....	1075
For Cooling Brewing and Distilling Liquids. (P. Worsum) .....	239
For Cooling, Heating, Drying, &c. (P. Fischer) .....	622
For Cooling Liquids. (P. Webb) .....	378
For Cooling Liquids or Gases. (P. Price) .....	1048
For Cooling or Warming Liquids. (P. Baker) .....	713
For Creating a Vacuum in Vacuum Pans. (P. Pe) .....	624
For Crushing Dry or Wet Materials. (P. Morel and Hempel) .....	623
For Detecting and Estimating Fire-damp in Mines. (P. Fletcher) .....	663
For Detecting and Measuring Inflammable Gases. (P. Tilchman) (illus.) .....	1052
For Determining Density of Gases. (P. Feres and Meslans) .....	378

	PAGE
Apparatus .....	6
For Digesting and otherwise Treating Bones, Fat, Meat, &c. (P. Warner) .....	504
For Disinfecting. (P. Anderson and Sjostrom) .....	900
For Disinfecting. (P. Hermite, Cooper, and Patterson) .....	271
For Disinfecting. (P. Pitt, From La Socie. Pietet) .....	900
For Disinfecting and Fumigating. (P. Crosby) .....	1084
For Distillation of Ammoniacal Liquor, &c. (P. Pettigrew) .....	734
For Distilling. (P. Lavy) .....	751
For Distilling and Breaking up Liquid Hydrocarbons. (P. Clark) .....	351
For Distilling and Gasifying Oils. (P. Caldwell and Tatham) (illus.) .....	936
For Distilling and Sterilising Water. (P. Nagel) .....	623
For Distilling Sea-Water and other Liquids. (P. Fouche) .....	714
For Distilling Spirits and other Liquids. (P. Morris and Edney) (illus.) .....	504
For Distributing and Burning Liquid Fuel. (P. McMurtrie) (illus.) .....	937
For Drying off Heavy Vapours of Solvents. (P. The Parent Syndicate Oil Extractor Co. and Dombain) .....	240
For Drying and Carbonising Textiles. (P. Dasse and Fettweis) .....	1192
For Drying Animal and other Matters. (P. Currie and Rowlands) .....	714
For Drying Animal and Vegetable Substances. (P. Christy, From Tollner) .....	714
For Drying Brewers and Distillers' Grains. (P. Fielder) .....	1214
For Drying Distillers' Washes, &c. (P. Actien Maschinenbau-Ges. vorm. Vemelte and Ellenberger) .....	827
For Drying Gypsum, Clay, Grain, Refuse, &c. (P. Hadden, From Cummer) .....	241
For Drying Materials. (P. Brunton) .....	243
For Drying, Pulverising and Evaporating. (P. Von Podewils) .....	871
For Drying "Ship" and Evaporating Liquids. (P. Peake) .....	39
For Drying Substances Viscous when Hot. (P. Lake, From Schicht) .....	1182
For Dyeing. (P. Gessler) .....	899
For Dyeing. (P. Obermaier) .....	728
For Dyeing and Washing Textiles. (P. Labhardt) .....	391
For Dyeing Fibres. (P. Rhodes and Perkins) .....	888
For Dyeing Hats, Caps, Stockings, &c. (P. Licht) .....	391
For Dyeing Textile Fibres. (P. Lake, From Mattel) .....	728
For Effecting Intimate Mixture of Liquids, &c. (P. Julien) (illus.) .....	757
For Electrically Heating Liquids. (P. Crompton and Dowling) .....	256
For Electrically Heating or Working Metal. (P. Thompson, From Coffin) .....	161
For Electrically Treating Gases. (P. Hall) .....	328
For Electro-Chemical Decompositions. (P. Carmichael) .....	801
For Electro-Deposition of Metals. (P. Alexander) .....	819
For Electrolysis of Alkali Salts. (P. Sanding-Larsen) .....	1206
For Electrolysis of Chlorides and Salts. (P. Hargreaves and Bird) .....	958
For Electrolysis of the Chlorides of Sodium and Potassium. (P. Drake) .....	887
For Electrolysis of Water. (P. Siemens Bros. and Co. and Onch) .....	529
For Electrolytical Decomposition. (P. Lye) .....	958
For Electrolytical Decomposition of Brine and other Liquids. (P. Badly and Guthrie) .....	815
For Electrolytical Decomposition of Metallic Salts. (P. Kellner) .....	404
For Electrolytical Decomposition of Salt Solutions. (P. Kellner) .....	162
For Electrolytical Production of Alkali, &c. (P. Hargreaves and Bird) .....	528
For Electrolytically Dissociating Soluble Salts. (P. Hadden and Co., From Blackmore) .....	257
For Electrolytical Production of Soda and Chlorine. (P. Lake, From the Union Chemical Co.) .....	257
For Electrolytical Separation of Bases of Salts, &c. from Saline Solutions. (P. Vautin) .....	256
For Electrolytic Purposes. (P. Guthrie) .....	1206
For Enabling a Person to Breathe Fresh Air while Surrounded by a Noxious Atmosphere. (P. Ash) .....	793
For Estimating Fat in Milk. (P. Woosnam and the Dairy Supply Co.) .....	1091
For Evaporating. (P. Crance) .....	872
For Evaporating and Cooling Liquids. (P. Scott) (illus.) .....	23
For Evaporating and Drying. (P. Newton) .....	871
For Evaporating Brine and other Liquors. (P. Scott) .....	622
For Evaporating Liquids. (P. Latham and Gayot) .....	713
For Evaporating Liquids and Saccharine Solutions. (P. Morrel and Fromherz) .....	377
For Exhausting. (P. Wright) .....	1182
For Extracting Fat and Glue. (P. Machalski) .....	648
For Extracting Gold and Silver from Ores. (P. Trug) .....	527
For Extracting Oil by Volatile Solvents. (P. MacIwaine and Hardingham) .....	745
For Extracting Oil, &c. from Fibrous Materials. (P. Forles) .....	261
For Extracting Substances by Volatile Solvents. (P. Meikle) .....	622
For Extracting Suint. (P. Richard-Lagerie) .....	962
For Extraction. (Etraix) (illus.) .....	839
For Filtering and Purifying Water. (P. Winkler) .....	377
For Filtering Liquids, or Separating Liquids from Solids. (P. Drummond) .....	377
For Fining Beers and Filling Casks. (P. Crossman) .....	1082
For Fractional Distillation. (Otto) (illus.) .....	837

## Apparatus—cont.

For Fractional Distillation. (Varenne) (illus.)	761
For Fractional Distillation. (Volney) (illus.)	652
For Galvanising Sheet Metal. (P) Laques	178
For Gas Analysis. (Fischer) (illus.)	42
For Gas-making. (P) Hartley	1050
For Gassing Yarn and other Threads. (P) Clouzel	880
For Glazing and Enamelling Heavy Earthenware. (P) Twyford	396
For Generating Gaseous Fuel. (P) Thwait and Threlfall (illus.)	717
For Hardening and Tempering Steel. (P) Fichtax and Beardmore	1067
For Heating Air and Fluids. (P) Fudickel	1181
For Heating and Condensing. (P) Hennebert	713
For Heating, Evaporating, and Condensing. (P) Normandy	29
For Heating or Evaporating Liquids. (P) Morsen	622
For Lifting Blasting-Charges. (P) Roth	690
For Lighting and Heating by Petroleum. (P) De Pass, From Leeumont	141
For Making Bleaching-Powder. (P) Milnes	1039
For Making Vinegar and Maturing Spirits. (P) Lecker	635
For Manufacture and Purification of Iron. (P) Clapp and Williams	1261
For Manufacture of Alkali. (P) Hargreaves and Bird	250
For Manufacture of Alkaline Metals by Electrolysis. (Borchers) (illus.)	159
For Manufacture of Artificial Whalebone. (P) Levy, Rustin, and Boyer	1074
For Manufacture of Beer. (P) Billings	896
For Manufacturing Caustic Soda. (P) Crane (illus.)	636
For Manufacture of Coke, Coal-Gas, &c. (P) Dykowitz	626
For Manufacture of Gas. (P) Dismore	934
For Manufacture of Gas and Bye-Products. (P) Young	1035
For Manufacture of Margarine and Alimentary Fat. (P) Cordewener and de Kunwald	270
For Manufacture of Metallic Pigments or Compounds. (P) Charlier	532
For Manufacture of Paper Pulp. (P) Caldwell	1085
For Manufacture of Pottery. (P) Shack	1062
For Manufacture of Soap Tablets. (P) Finlay	905
For Manufacture of Spirits. (P) Murphy	751
For Manufacture of Sugar. (P) Johnson, From Pellegrini (illus.)	1056
For Manufacture of Sulphocyanides. (P) Brock and others	1195
For Manufacture of Water-Gas. (P) Glasgow	934
For Manufacture of Water-Gas. (P) Paisley	212
For Manufacture of Water-Gas. (P) Westcott (illus.)	1051
For Manufacturing White Sugar from Molasses. (P) Hubner	824
For Measuring Pressure of Gas in Gurs. (P) Castenholz	929
For Mixing and Regulating Paper Stuff. (P) Borland	754
For Mixing Gas and Air. (P) Axdorfer and Sass	1050
For Notifying Presence of Gases in Air. (P) Hardy	1091
For Obtaining Ammonia and Ammonia Salts. (P) Lake, From Sternberg	887
For Obtaining Carbonic Acid. (P) Muller (illus.)	636
For Obtaining Constant Pressure during Estimation of Freezing Point of Oils. (Gantter) (illus.)	1090
For Obtaining Cyanogen, Cyanides, &c. (P) Aiken (illus.)	517
For Obtaining Distilled and Sterilised Water. (P) Nagel	169
For Obtaining Gas from Coal-Tar. (P) Smith	626
For Obtaining Gases, Ammonia and Tar from Fuel. (P) Mond (illus.)	968
For Obtaining Glycerin from Spent Lyes. (P) Ruymbeke and Jobbins	1070
For Obtaining more perfect Combustion of Fuel. (P) Taylor	139
For Oxidising Indigo Blues. (P) Grime	728
For Pasteurising Beer. (Windeck)	267
For Pasteurising Milk, &c. (P) Paaseh	1215
For Pickling Metal Articles. (P) Cowper-Coles	889
For Powdering Roasted Cements. (P) Bollee and Benard	1198
For Precipitating Gold and Silver from Cyanide Solutions. (P) Bell and Carter	816
For Preparation of Malt. (P) Schmiedecke and Gade	827
For Producing and Consuming Gas. (P) Hall	528
For Producing an Improved Paper or Fabric. (P) Wheeler	639
For Producing Artificial Silk. (P) Lehner	1192
For Producing Carbonic Acid Gas. (P) Lane and Fulman	810
For Producing Chlorate of Potash by Electrolysis. (P) Hunter	810
For Producing Pure Castings. (P) Müller	800
For Production of Cranides. (P) Gordon	886
For Production of Metallic Pigments. (P) Charlier	407
For Production of Mineral-Oil Gas and Water-Gas. (P) Young (illus.)	934
For Production of Oxygen from Air. (P) Bria's Oxygen Co. and Murray	856
For Production of Ozone from Phosphorus. (P) Kattenhof	414
For Production of Sulphuretted Hydrogen. (Kuster) (illus.)	278
For Pumping and Absorbing Chlorine Gas. (P) Worsley, Windus, and Bracey	810
For Purification of Gas. (P) Fleischhauer and Bernstein (illus.)	718
For Purifying Feed-Water. (P) Wotherspoon and Davie	505
For Purifying Liquids. (P) Wagner and Marr	751
For Purifying Sewage and Waste Liquids. (P) Adeney and Parry	831

## Apparatus—cont.

For Purifying Water. (P) Maché	841
For Purifying Water. (P) Walker	163
For Purifying Water. (P) Webster	163
For Raising Liquids. (P) Moore	163
For Raising Liquids and Sea Liquids. (P) Taylor	24
For Raising or Aerating Water or other Liquids. (P) Store	964
For Raising or Pumping Liquids. (P) Kestner	625
For Rapid Determination of Carbonic Acids. (P) Pouché (illus.)	241
For Refining Metal by Electrolysis. (P) Hesse and Symon	544
For Refrigerating. (Linder) (illus.)	1068
For Regulating Temp. of Fermenting Liquids. (P) Collins	752
For Regulating Temp. of Fluids. (P) Royle	87
For Removing Fibrous Matter from Cotton-Seed. (P) Thompson, From Stead	1179
For Removing Impurities from Water. (P) Shaddock	820
For Scouring and Degreasing Wool. (P) Boys, From Mahud	23
For Scouring and Washing Fabrics. (P) McNaught	880
For Scouring, Degreasing, and Cleansing Wool. (P) Turney	805
For Scouring Wool. (P) Turney	634
For Separating and Purifying Minerals, &c. (P) Beyer (illus.)	944
For Separating Oil from Feed-water. (P) McDougall	623
For Separating Substances. (P) Anderson, jun.	29
For Separation of Metals, &c. (P) May	871
For Shaking Bottles. (Maill) (illus.)	740
For Sizing Yarn. (P) Maaz	1220
For Smelting and Refining Copper. (P) Vienna	880
For Softening and Purifying Water. (P) Doulton and Manger	644
For Softening and Purifying Water. (P) Smith	832
For Softening Water. (P) Gittins	538
For Sterilising. (P) Hussener	271
For Sterilising Beers, Beverages, &c. (P) May and Newlands	1215
For Sterilising or Disinfecting Liquids. (P) Ottmann and Herbst	771
For Sterilising Water. (P) Yagin	137
For Straining Pulp, &c. (P) Reimcke	416
For Superheating Steam. (P) Musgrave and Dixon	713
For Supplying Carbonic Acid under Pressure. (P) Browne, From Beck	1048
For Supplying Liquid Fuel to Furnaces. (P) Etchells	788
For Testing Gas Cylinders. (P) The Scotch and Irish Oxygen Co. and H. Brier (illus.)	1094
For Testing Paper. (P) Woolley (illus.)	789
For the Continuous Production of Nitric Acid. (Prestice) (illus.)	1222
For the Distillation of Ammoniacal Liquor. (P) Spence and Gilchrist	323
For the Preparation of Malt. (P) Genze	394
For Treating Fibres. (P) Brintons, Lim, and Hansen	412
For Treating Fibres by Liquids. (P) Miller	885
For Treating Powdered Materials. (P) Petwiler	884
For Treating Refuse. (P) Smithson	1181
For Treating Salts Electrolytically. (P) Stormer	271
For Treating Soap. (P) Candy	1067
For Treating Substances containing Fats and Oils. (P) Robbins	1215
For Treating Textiles. (P) Gimes	1070
For Treating Textiles. (P) Jackson and Hunt	248
For Treating Yarns. (P) Jacob	944
For Treatment of Wort, &c. (P) De Geyer (illus.)	596
For using Producer-Gas for Cementing Limestone. (P) Boulton, From Hatch (illus.)	637
For Utilising Lyes of Sulphite Cellulose. (P) Mitscherlich	834
For Valuation of Manganese Dioxide. (Ullmann) (illus.)	879
For Vaporising and Burning Mineral Oil. (P) Grosche	139
For Vaporising Oil. (P) Laird	873
For Washing, Bleaching, Dyeing, and Mordanting. (P) Owens and Whiteley	390
For Washing Fibres. (P) Lazarus and Jefferson	945
For Washing Filtering Media. (P) Thompson, From Arnold (illus.)	25
For Washing Gas. (P) Marshall (illus.)	716
For Washing Photographic Plates and Prints. (P) Thompson	550
For Washing Wool or other Fibres. (P) Dawson	509
For Weighing out Liquids. (Schweitzer and Lunawitz) (illus.)	838
For Working Ores. (P) Allison, From Storer, Martin, and Eaton	816
Hydrostatic, for Treating Liquids and Solutions. (P) Spence	613
Rotary, for Washing and Dyeing. (P) Crabtree	884
Safety Appliances for Use with Electrical Decomposing. (P) Richardson	958
Some Laboratory. (Bidet) (illus.)	621
Apple, Development and Ripening of the Cider. (Lindet)	54
Formation of Cane Sugar during Ripening of the. (Lindet)	750
Study of the Variety of, used for Cider-making. (Truelle)	266
Apples, Preparation of Alcohol from. (Vivian and Dupont)	902
Appliance for Breaking down Crust in Gas-Producers. (P) Hall	627
Aqua Regia, Influence of, on Precipitation of Barium as Sulphate. (Browning)	176

	PAGE
Argentina, New Customs Law of. (T.R.)	182
The Vanadiferous Lignite found in. (Kyle)	738
Arenatics, Production of Substances for Manufacture of. (P)	
Titanium	903
Arsenic, Detection and Separation of. (Gleichen and Hodge)	979
Elimination and Recovery of, from Copper Precipitates. (P) North	1291
In Copper, Modification of Ferric Chloride Process for Estimation of. (Platten)	324
In Sulphides, Determination of. (Jannasch) (illus.)	905
Artichoke, The Carbohydrates of the Jerusalem. (Tauret)	52
Artillery, German Trade in. (T.R.)	1113
Asbestos Industry of Canada, The. (T.R.)	993
Mining in Canada. (T.R.)	674
Baking, Improved Compound. (P) Field	788
Use of, for Filtration of Sugar, Juices, and Syrups. (Maurer)	264
Asphaltic Composition, Production of. (P) Sherer	618
Asphalt Paving, U.S.A. Report on.	726
Asphaltum, Notes on. (Niederlander)	263
Or Pitch, Artificial, and Production thereof. (P) Haddan. From Anderson, Shorler, Kochler, and Dorer.	729
Aspidium Filix, Extract of the Root of. (Ehrenberg)	758
Assam, India-Rubber Production in. (T.R.)	433
Atmosphere, Attempts to Estimate Sulphur Compounds in the. (Oates)	1222
Effect of the, upon Glass. (Foerst)	518
Atropine Alkaloids, The. (Hesse)	416
Auditors, Appointment of.	700
Aver Incandescent Mantle, Is it Poisonous? (Grichant)	1050
Auzsburg, Treatment of Municipal Refuse at.	412
Auramine, Manufacture of. (P) Johnson. From The Badische Anilin und Soda Fab.	247
Auramines, The. (Stock)	244
Aurin. (Herzig and Smoluchowski)	798
Austen Process for Treatment of Pyritic Minerals by Fusion	812
Australia, A Discovery of Coal in. (T.R.)	78
Austria-Hungary, New Patent Law in. (T.R.)	71
Portland Cement Industry in. (T.R.)	1108
The Mineral Production of. (T.R.)	994
Average Prices of General Commodities in England, Course of. (Sauerbeck) (illus.)	Facing p. 672
Azo-Coulour Resists on Steam-printed Indigo. (Schlieper and Bloch and Schwartz)	1193

## B

Bacteria of Cholera and Typhoid, Action of Peat Dust on. (Frankel and Kilpstein)	538
of Hides, Action of Salt on. (Haendlein)	49
Removal of Pathogenic from Drinking-Water. (Fuller)	830
Baking-Powder. (P) Brothers	752
Powder, Means of using. (P) Daim, Brown and Polson	1083
Baku, The Petroleum Industry in, 1893.	875
Balances, Improvements in. (P) Thomson	66
Balanta Production in Surinam. (T.R.)	430
Banana-mel, Production of, in Surinam. (T.R.)	430
Bank Notes, Manufacture of Paper for. (P) Buttner and Will	539
Barium and Calcium, Separation of. (Browning)	282
Estimation of, in Presence of Calcium and Magnesium. (Mar)	282
Influence of Free Nitric Acid and Aqua Regia on Precipitation of, as Sulphate. (Browning)	176
Peroxide, Action of Ammonia-Gas on. (Michel and Grandmaison)	37
Salts, Manufacture of.	392
Barley, Formation of Cane-Sugar during Germination of. (Lander)	750
Influence of Iron on the Vegetation of. (Pott)	632
Production of Saccharose during Germination of. (Lander)	265
Taking Specific Gravity of, by Means of Volumetric Barley-Balances	1078
Bartlett's z-Lead Process, Report on The. (Hawker)	41
Bases, Action of, on Glucoses. (Jessen)	264
In certain Metallic Salts, Volumetric Estimation of. (English)	515
Manufacture of New Amides. (P) Korell	632
Of Salts and Compounds thereof, Electrical Separation of, from Saline Solutions. (Vautin)	256
Titration of Organic, with Methyl Orange. (Lunge)	607
Basic Compounds from Pyrazine, Production of. (P) Newton. From The Farb, vorm. Bayer and Co.	510
Steel Production in 1893. (T.R.)	292
Bath, a New Form of Air. (Sauer) (illus.)	692
Baths for Hardening and Toughening Iron and Steel. (P) Bates	645
Battery, Darrius' Theory of the Lead Secondary. (Schoop)	1293
Batteries, Various:—	
Accumulator, A New. (Leuz)	158
Accumulators, Electric. (P) Ellermann	161

## Batteries—cont.

Accumulators, Electric. (P) Schroeder	800
Dry. (P) Johnson	404
Electrical. (P) Feeny. From Hubers and Mohrdeck	1068
Galvanic. (P) Buell	1068
Galvanic. (P) Boult. From Cudell	647
Galvanic. (P) Schroeder	160, 169, 528
Portable Primary. (P) Morison	1067
Primary Galvanic, and Liquids thereof. (P) Longsdorf. From Hewitt	818
Primary Voltaic. (P) Walker, jun., and Wilkins	529
Primary Voltaic. (P) Walker, jun., and others	1206
Secondary. (P) Doubleday. From Susmann	528
Secondary. (P) Petschel	256
Secondary. (P) Peyrussou	646
Secondary. (P) Schoop	646
Secondary Electric. (P) The Lithanode and General Electric Co. and Niblett	818
Secondary Voltaic. (P) Clulbe and Southey	45
Storage. (P) Johnson. From La Soc. L'Accumulateur Fulmen	819
Storage. (P) Kordon	1206
Storage. (P) Thompson. From Hartung	256
Storage. (P) Usher	162, 162
Storage. (P) Young. From The Hess Storage Battery Co.	1206
Thermo-Electric, and Apparatus for Manufacture thereof. (P) Dion	890
Voltaic. (P) Gregg	1205
Battery Cells, Plates, Elements, Solutions, &c.:—	
Active Material for Accumulators. (P) Mills. From Barbier	1206
Carbon for Electric Batteries. (P) Lake. From Girard and Street	647
Carbon for Electrical Purposes. (P) Acheson	1207
Carbon for Electrical Purposes. (P) Newirth	1206
Carbon for Electrodes. (P) Casner	1067
Cells and Diaphragms, Electrolytic. (P) Hargreaves and Bird	255
Cells, Electrolytic, and Diaphragms thereof. (P) Waite	959
Cells for Electric Batteries. (P) Emery	404
Cells for Electrolytic Apparatus. (P) Hurter, Auer, and Muspratt	958
Diaphragms for Electrolytic Purposes. (P) Kiliani and others	1068
Electrode Plates for Accumulators. (P) Lehmann	1206
Electrodes and Cells for Accumulators. (P) Wheeler	647
Electrodes, Carbon. (P) Barnett	44
Electrodes, Carbon. (P) Livinge	161, 161
Electrodes, Electro-Positive Galvanic. (P) Schroeder	257
Electrodes for Accumulators, Manufacture of. (P) Pollak	44
Electrodes, Platinised. (P) Barnett	44
Electrolytes for Primary Batteries. (P) Longsdorf. From Hewitt	890
Elements for Secondary Batteries. (P) The Lithanode and General Electric Co. and Niblett	256
Elements for Voltaic Batteries. (P) Fitzgerald	743
Elements or Plates for Secondary Batteries. (P) Picard and Thame	256
Plate for Electrical Accumulator. (P) Mills. From Barbier	1206
Plates for Accumulators. (P) Gulcher	741
Plates for Accumulators. (P) Rousseau	744
Plates for Secondary Batteries. (P) Blot	819
Plates for Secondary Batteries. (P) Hough	44
Plates for Secondary Batteries. (P) The Lithanode and General Electric Co. and Niblett	744
Plates for Secondary Batteries. (P) Thompson. From La Soc. Electric Phœbus	161
Plates of Secondary Voltaic Batteries, Treatment of. (P) Epstein	743
Plates or Elements for Secondary Batteries. (P) Hough and March	161
Beakers for Dyeing Tests, Hard Glass. (Lepetit)	978
Bean, The Proteids of the Kidney. (Osborne)	828
Beer, Alleged Disappearance of Added Salicylic Acid and Saccharine from. (Wauters)	969
Apparatus for Pasteurising. (Windeck)	267
Apparatus for Manufacture of. (P) Billings	896
Apparatus for Sterilising. (P) May and Newlands	751
Clarification of. (P) Lake. From Muller	269
Composition for Preserving. (P) Pickles	1083
Effect of Albuminoids in Manufacture of. (Wahl and Nilson)	896
Effect of Alkaline Chlorides on Flavour of	1080
Effect of the Saline Constituents of Water on the Character of. (Readman)	367
Evolution of Carbon Dioxide, and Hydrogen Sulphide in Boiling. (Elion)	267
"Gluten Particles" occurring in. (Will)	967
Influence of Hop-Tannin on. (Hayduck and Goldiner)	967
Influence of Mashing Process on Attenuation of. (Windisch)	967
Manufacture of. (P) Lake. From Tedesco	896
Manufacture of. (P) Pennington	656
Manufacture of. (P) Pkhardt	535
Method and Apparatus for Collecting Carbonic Acid during Fermentation of. (P) Michel and Frank	655
Presence of Boric Acid in. (Brand)	34
Production of Coconut- and Rice. (Calmette)	268
Regulations Affecting the Adulteration of, in Belgium. (T.R.)	290

	PAGE
Beer— <i>cont.</i>	
Some Micro-organisms causing Diseases of. (Fellows).....	1080
Yeast, Chemical Properties of the Alcoholic Extract of. (De Key-Pollander).....	751
Beers, Apparatus for Fining. (P) Grossmann.....	1082
Estimation of Alcohol in, by means of the Ebullioscope. (Torric).....	762
The Albuminoids contained in. (Wahl and Hauck).....	895
Beeswax, Examination of. (Parys).....	1226
Extraction of the Free Acids of. (Mayer).....	1207
Notes on Examination of. (Keldner).....	745
The Analysis of. (Buchner).....	70
Beet Juice, Mucilaginous. (Anderslik).....	719
Juice, Precipitates formed during Heating of. (Herzfeld).....	719
Beetroot Destroying Organisms—Phoma Betæ, (Hollnag).....	264
Destroying Organism, Phoma Betæ, the. (Frank).....	822
Growing Experiments in 1893. (Schuck Sommer).....	213
Juice, Comparative Defecation Experiments with. (Herzfeld).....	823
Juice, Defecation of. (Hanus).....	253
Juice, Evaporation and Boiling of. (Leva).....	823
Juice, Losses by Concentrating. (Dezener).....	1210
Juice, Purification of. (Spunt and Schuchtrupp).....	263
Juice, Purification of. (Bouvier).....	1210
Juice, Purifying and Decolorising. (Steffen and Drucker).....	823
On the Alcohol Extraction of. (Wiskirchen).....	1226
Sugar Industry, Improvements in the. (Jarkovsky).....	965
Sugar Industry of Sweden. (T.R.).....	556
Sugar Manufacture, Control of. (Battuti).....	750
Sugars, Grey Colour of Raw. (Druckmann).....	1210
Beetroots, Determination of Crystallisable Sugar in. (Pellet).....	669
Estimation of Quantity of Juice contained in. (Vivien).....	264
Estimation of Sugar in. (Krocker).....	823
Influence of Fat in Seed of, on Richness in Sugar. (Doerflinger).....	110
Behring's Experiments with Antitoxin. (Levinstein).....	1112
Belgium, Chemical Congress in. (T.R.).....	850
Regulations Affecting the Adulteration of Beer in. (T.R.).....	290
Bell, Presentation to Mr. J. Carter.....	607
Bengal Saltpetre. (T.R.).....	676
Shellac Industry, The. (T.R.).....	774
Benzene, Carburetted Coal-Gas with. (Ries).....	873
Determination of Nitrogen in. (Kruzer).....	425
Benzidine Colours, Cotton Dyeing with. (Mullerus).....	883
Colours, Use of, in Discharging Indigo. (Stein).....	882
Benzine, Electrical Excitation of. (Kissling).....	385
Benzoic Acids, The Odours of. (Lassy).....	670
Benzoylamido Phenyl Acetic Acid, The Phenylester of. (Weiss).....	55
Berlin Experimental Institutes Paper-Testing Department. Expert Opinions from the (T.R.).....	539
The Chemical Industry of. (T.R.).....	912
Bertrams, Visit to Messrs.....	701
Betaine and Choline in Wormseed, Occurrence of. (Jahnst).....	57
Preparation of, from Malt-Combs and Wheat-Germis. (Schulze and Frankfurt).....	51
Betanaphthoquinone, Production of Derivatives of. (P) Newton. From the Farb. vorm. Bayer and Co.....	21
Naphthohydroquinone, Production of Derivatives of. (P) Newton. From the Farb. vorm. Bayer and Co.....	31
Methyl-eurhodine, Manufacture of. (P) Wilcox. From The Badische Anilin und Soda Fabrik.....	878
Beverages, Compounds for Manufacture of Fermented. (P) Heron.....	1082
Means for Producing Effervescent. (P) Wiemann.....	898
Bicelium Phosphate, Action of Water on. (Joly and Sorch).....	634
Binding Medium for Size Colours. (P) Clasen.....	962
Birch-Tar, Distinction of, from Pine-Tar. (Hirschsohn).....	123
Bismit, S. erilisation of. (Balland and Massen).....	169
Bismuth and Silver, Separation of. (Jamnaseh and Lesinsky).....	67
Basic Salicylate of. (Schubardt).....	57
Electrolytic Separation of, from Arsenic, Tin, and Antimony. (Schmuckert).....	280
Gallate, Basic. (Dermatol.) (Caussé).....	171
In Bolivia. (T.R.).....	912
Salts, Preparation of Basic Organic. (Voswinkel).....	273
Silver Alloys, on the Cupellation of. (Smith).....	765
Black-Ash Residue of Wood-Pulp, Treating the. (P) Langville.....	170
Lead, Manufacture of. (P) Johnson.....	821
Lead, Manufacture of. (P) Ripley.....	162
Blat-Furnace, Behaviour of Phosphoric Acid in the. (Kjollberg).....	524
Furnace Charges for Smelting Lead and Silver Ores. (Watson).....	1140
Furnace Slag, Utilisation of. (Elbers).....	643
Furnaces and Chimneys, Construction of, and Bricks therefor. (P) Ingham.....	623
Furnaces, Capacity and Form of. (Hawdon).....	643
Furnace Slag, The Principal Smelting Reactions of. (Elbers).....	598
Blasting and Blasting. (P) McLeod and Davidson.....	70
Curriers, Phlegme Compound for. (P) Auer.....	70
The Westfälische Alkali-Soda-Spinnstoff A.G. Gesellschaft.....	31
Charles, Apparatus for Gasizing. (P) Rother.....	70
Charles in Mines, Typed in. (P) Jarolinek.....	70
Method and Apparatus for. (P) S. H. A. Z.....	70
Blanching. (Class XVIII).....	113
And Decolorisation. (P) Claple.....	113
Lishman.....	113
And Printing, Improvements in.....	84
Cotton Cloths, &c. (P) Schott.....	113
Liquor, Apparatus for Electrolytic Production of.....	113
Kellner.....	113
Mixture, Production of a. (P) Wacht.....	113
Powder, Apparatus for Making. (P) Mihnes.....	113
Powder, Apparatus for Manufacture of. (P) Muspratt, Carey, and Driffield.....	113
Powder, Manufacture of. (P) Gask, & Co., and others.....	113
Powder, Specific Gravities of Solutions of. (Lange.....	113
Solutions, Manufacture of, by Electrolysis. (P) B.....	113
From Knödel and Gehauer.....	113
Blende, Separation of, from Pyrites. (Blaker).....	113
Blow-Lamps for Soldering, &c. (P) Lake. From Horth and Co.....	750
Blue-Pine, An Improved mouth. (P) James.....	758
Blue Coloration Produced by L-ascorbic acid in contact with Acids. (Rosentisch).....	113
For Washing. (P) Annuam.....	113
Board of Trade Returns. 83, 185, 201, 136, 543, 675, 774, 857, 914, 1062, 1119, 1188.....	113
Boghead Canals, General Characters of. (Bertrand and Renault).....	113
Boiler Incrustation, Composition for Preventing and Removing (P) Berneck and Komestik.....	716
Scale, Removal of, by Means of Petroleum.....	113
Shells, The Internal Rusting of. (Pfeifer).....	714
Boilers for Treating Fibres for Paper-Making. (P) Smecher.....	113
Loss of Heat in. (Scheurer-Kestner).....	113
Preventing Incrustation in Steam. (P) Gane.....	113
Prevention of Scale in. (Walters).....	113
Boiling Point, Distillation of Substances having a High. (P) Jaffe and Darmstadter.....	113
Vessels, Improved Mode of Setting up. (Mangoldt).....	113
Bolivia, Antimony and Bismuth in. (T.R.).....	113
Supplies of Caoutchouc in. (T.R.).....	113
Bombax Pentandrum. (Araucaria).....	113
Bones, Production of Glue and Gelatin from. (P) Grilloa and Schroeder.....	113
Bonnet's New Dyeing Process, Report on. Kopp, Neeltz and Grandmoulin.....	726

	PAGE
Calico Printing. (Class VI.)	34, 118, 248, 386, 513, 633, 726, 803, 880, 945, 1057, 1193
Printing. (P) Lang.	150
Printing Machinery. (P) MacPhail	1194
Prints, Production of Fast Reds in. (Dopierre)	1194
California, Discovery of Native Antimony in. (T.R.)	78
Calomet, Manufacture of, in Japan. (Divers) (illus.)	108
Camphele in Spike Oil, Presence of. (Bouchardat)	658
Occurrence of, in Etheral Oils. (Bertram and Walbaum)	756
Presence of, in Essence of Lavender. (Bouchardat)	756
Campholene. (Guerbet)	628
Camphor as a Reagent for Sugar. (Neitzel)	843
Compositions of, with other Substances. (P) Verdun	57
Canada and the American Drug Trade. (T.R.)	995
Chronic Iron in Quebec. (T.R.)	911
Mineral Production of, for 1893. (T.R.)	555
Phosphate and Asbestos Mining in. (T.R.)	674
Tariff Changes in. (T.R.)	672, 769, 846
The Asbestos Industry of. (T.R.)	993
Canaisiere. (Kapff)	946
As a Substitute for Bark in Tanning	650, 894
Canal, the Manchester Ship	11
Cancer, Preparation for Treatment of. (P) Watson	417
Candl's, Rules for the Preparation of Standard Sperm	65
Sulphur, and their Holders. (P) Kingzett	657
Cannabinden. (Kobert)	902
Cannabis Sativa, Occurrence of Trigonellin in Seeds of. (Schulze and Frankfurt)	757
Cannels, General Characters of Boghead. (Bertrand and Renault)	241
Canvass, Rendering, Sensitive to Light. (P) Junk	173
Caoutchouc in Bolivia, Supplies of. (T.R.)	673
Manufacture of Artificial. (P) St. George	48
Membrane, Diffusibility of certain Gases through. (Reyher)	181
Carouel, The Properties of. (Sabanejell and Antouchevitch)	1076
Carbamides, Solutions of Sweet, in Oils, Fats, &c. (P) Sommer	1071
Carbides, The Production of Various. (Muhlhauser)	34
Carbohydrates, Colorimetric Methods of Estimating. (Reitzel)	985
Of the Fruit of the Kentucky Coffee-Nut Tree. (Stone and West)	536
Of the Jerusalem Artichoke. (Tanret)	52
Of Yeast, The. (Salskowski)	411
Pre-existing in Malt. (Jalowetz)	967
Quantitative Colorimetric Estimation of. (Neitzel)	285
Quantitative Estimation of. (Schulze)	985
Carbolic Acid, German Trade in. (T.R.)	1214
Carbon and Iron, Transformations of, in Tempering. (Charpy)	1066
Bisulphide, Manufacture and Refining of, at Zalata. (Farbaky) (illus.)	508
Black, Note on Cabot's Paper on Preparation of, from Natural Gas. (Irvine)	131
Blacks, Preparation of, from Natural Gas in America. (Cabot) (illus.)	128
Dioxide and Nitrogen in Air, Minimum Proportion of, Extinctive of Flames. (Clowes and Feilmann)	1155
Dioxide, Apparatus for Estimating. (Christomanos) (illus.)	1221
Dioxide, Evolution of, in Boiling Beer, Wort and Hops. (Elion)	267
Dioxide, Influence of, on the Growth and Life of Fermentation Organisms.	266
For Electrical Purposes, Manufacture of. (P) Lake, From Girard and Street	647
For Electrical Purposes, Manufacture of. (P) Niewerth	1206
In Ferro-Carbon Alloys, Influence of Aluminium upon (Hogg) (illus.)	955
In Iron and Steel, Determination of. (Hempel) (illus.)	421
In Iron, Examination of Methods for Estimation of. (Ledeber)	281
In Iron, Reliability of Principal Methods for Determining Total. (Gottig)	421
In Iron, Various Methods of Estimating. (Gottig)	1096
In Steel, Determination of. (Koch) (illus.)	979
In Steel, The Conditions in which it Exists. (Arnold and Read)	740
Monoxide and Olefines, Distinction between	841
Testing, Colour Gauzes for. (McMillan)	978
Tetrachloride, Production of. (P) Muller and Dubois	972
Carbonate of Ammonia, Manufacture of. (P) Raspe	394
Carbonates, Extraction of Carbonic Acid from Mineral. (P) Van Berkel	38
Manufacture of Alkaline. (P) Reich	1059
Carbonic Acid and Bye-Products, Manufacture of. (P) Enequist	948
Acid, Apparatus for Obtaining. (P) Muller (illus.)	636
Acid, Estimation of. (Wolkowicz)	904
Acid, Extraction of, from Mineral Carbonates. (P) Van Berkel	38
Acid Gas, Method and Apparatus for Producing. (P) Lane and Pullman	810
Acid, Method and Apparatus for Absorbing. (P) Muller	852
Acid, Method and Apparatus for Collecting, during Fermentation. (P) Michel and Frank	635



	PAGE		PAGE
Carbonic Acid— <i>see oil</i> .		Charcoal, Estimation of Carbon Sulphide in, Annual. H. v.	54
Acid, Pressure Apparatus. (P) Browne, From Beck.	788	Manufacture of Annual. (P) Kriens.	197
Acid, Washers for Separating from Gas. (P) Cripps (illus.)	379	Charges, Apparatus for Loading Blastfurn. (P) Roth.	606
Acid, Utilisation of Waste in Breweries. (Maret) (illus.)	825	Formation and Constitution of "Stiff" in the Weldon Process. (W. v. v.)	809
Carbons for Electric Lamps. (P) Acheson	1297	Chay-Root, The Colouring and Other Principles of. (P) Ben	244
For Electrodes. (P) Custer	1967	and Hummel	245
Carbonyl Chloride, Preparation of. (Erdmann) (illus.)	65	Tinctorial Properties of. (H. v. v.)	245
Carboxylic Acids of Dimethylaniline, The. (Lauth)	629	Chelerythine	836
Carbonyl Derivatives of Dimethylaniline. (Lauth)	214	Cheese, Estimation of Fat in. (Bondzynski)	769
Carboretting. (Hunte)	933	Chemical Action, Influence of Moisture on. (Baker)	767
Cardboard, Process for Colouring. (P) Wells.	391	Congress in Belgium. (T.R.)	839
Carnauba Wax. (Villon)	714	Industry, Depression of, in Germany. (Erdmann)	74
Carpaine. (Van Ryn)	757	Industry of Berlin, The. (T.R.)	912
Castlidges, Improved Blastfurn. (P) Macmah and Dickson.	760	Industry of England and Germany. (T.R.)	771
Casein in Cows' Milk, The Determination of. (Slyke)	181	Industry of Germany since 1885, The. (T.R.)	996
Casks, Apparatus for Priming. (P) Crossman.	1982	Trade of Germany since 1885. (T.R.)	1110
Lac-lined.	265	Industry of the United States. (T.R.)	557
"Cassage" of Wine, On the. (Bouffard)	654	Manures in the United States. (T.R.)	114
Castings, Apparatus for Producing Pure. (P) Muller	800	Properties of the Alcoholic Extract of Beer-Yeast. (P) Rey-Pailhade)	751
Castor-Oil in Balsam of Copaiba, Detection of. (Manly)	981	Works and the Sandy Rest Act. (T.R.)	1234
Oil in Olive Oil, Detection of. (di Vetro)	981	Works, Report of Committee on. (T.R.)	76
Oils, Report on. (Deering and Redwood)	959	Works, Special Rules for, under the Factory and Work-Shop Acts. (T.R.)	77
Cattle-Food, An Improved. (P) Holland, From Holland.	168	Chemicals and Uses on Free List in the Netherlands. (T.R.)	291
Caucasus, Petroleum Discoveries in the. (T.R.)	779	Exports of to British Colonies in 1893. (T.R.)	851
Petroleum Supply of the. (T.R.)	1231	Chemistry of Foods, Sanitary Chemistry, and Disinfectants. (Class XVIII.)	54, 168, 209, 412, 596, 656, 752, 827, 897, 939, 1082, 1215
The Manganese Trade of the. (T.R.)	558	Of Oil-Painting, Importance of Study of. (Petruschewsky)	252
Cells for Batteries. <i>See under Batteries</i> .		Recent Developments in Pharmaceutical. (Erdmann)	549
Manufacture of Porous. (Titaner)	888	Chili, Exemption from Duty of Potassium Cyanide in. (T.R.)	74
Cellulose, Crystallisation of, and Chemical Composition of the Cellular Membrane. (Gibson)	1196	Chimneys and Shafts, Construction of, and Bricks therefor. (P) Incham	623
Derivatives, Commercial. (Beadle)	901	China, Alkali Lands in North. (T.R.)	119
Manufacture of. (P) Kellner	115, 539	And Earthenware, Manufacture, Report of Committee of Enquiry on. (T.R.)	75
Manufacture of Straw. (Beveridge)	101, 213	Clay, Method and Apparatus for Refining. (P) Nicholls.	251
Nitrating. (P) Selphuang.	1220	Connecting Metal to. (P) Drayson.	811
Pulp, Manufacture of. (P) Johnson, From Sobogo	1216	Hermetically Joining the Surfaces of. (P) Doulton and Slater	637
Sulphuric Acid, and Products of its Hydrolysis. (Stern)	1230	Mosaic, Production of Watter. (P) Bale	519
Utilising Lyes of Sulphite. (P) Mitscherlich	834	Opium Adulteration in. (T.R.)	779
Celluloses of the Gramineae. (Smith)	537	Chloralic Acid. (Hanriot and Richet)	755
Cement, Adulteration or Improvement of. (T.R.)	1236	Chloralose. (Hanriot and Richet)	755, 755
Apparatus for Crushing. (P) Morel and Heimpel	623	Chloride of Lime. <i>See under Lime and Bleaching-Powder</i> .	
Chemical and Physical Examination of Portland. (Stillman)	278, 637	Chlorides, Action of, on Nitrosodimethylaniline. (Ehrlich and Cohn)	39
Composition and Treatment of Substances for Use as. (P) Walters	197	Action of Aluminium on. (R. v. v. and Frank)	515
Composition of Materials for. (P) Edwards, From Overton	737	And Mixtures thereof, Electrolysis of. (P) Lyte	404
Effect of Addition of Gypsum to Portland. (Schott)	251	Apparatus for Electrolysis of. (P) Harzeaves and Bird	958
Experiments on Slag. (Matton)	736	Effect of Alkaline, on Flavour of Beer	1089
For Porcelain, Transparent. (Deutsch)	888	Electrolysis of. (P) Harzeaves and Bird	886
From Alkali-Waste, Manufacture of. (P) Rigby, Neill, and Carr	918	Electrolysis of Alkaline. (Ost)	493
Kilns. (P) De Pass, From Lavocat and Candlot	1062	Electrolytical Decomposition of, and Apparatus therefor. (P) Lyte	958
Manufacture and Treatment of Fireproof Plastic Material for. (P) Stone	811	Chlorine and Bromine, Analytical Separation of. (Engel)	1094
Manufacture of. (P) Gui	638	And Bye-Products, Obtainment of. (P) Penikoff.	947
Manufacture of. (P) Webster	254	And Caustic Soda, Production of, by Electrolysis of Salt. (P) Drake	719
Manufacture of. (P) Kilpatrick	158	And Lime, The Interaction of. (Veley)	254
Manufacture of, from Alkali Waste. (P) Rigby, Neill, and Carr	638	And Refined Lead, Production of. (P) Lyte (illus.)	644
Manufacture of Hydraulic. (P) Stein.	529, 1062	And Soda, Apparatus for Electrolytically Producing. (P) Lake, From The Union Chemical Co.	257
Manufacture of Portland, Utilising Waste Carbonate of Lime. (P) Hill	255	Detection of Traces of. (Villiers and Fayolle)	1092
Manufacture of Pulverised Materials for. (P) Patrick	1018	Gas, Method and Apparatus for Pumping and Absorbing. (P) Worsley, Windus, and Incey	810
Mortar, Manufacture of. (P) Williams	1062	In Fats, Estimation of. (Benedikt and Zikes)	984
Portland, Industry in Austria-Hungary. (T.R.)	1198	Liberation of, during Heating of Mixture of Potassium Chlorate and Manganese Peroxide. (McLeod)	272
Quick Setting. (Geyer)	949	Liquid. (Fribourg)	151
Testing Hydraulic. (Prüssing)	949	Manufacture of. (P) Brand.	635
The Characteristics of Portland. (Fresenius)	252	Manufacture of. (P) Gaskell, Jun., and Cary	1196
The Composition of Portland. (Kosmann)	396	Normal, in Spring Waters. (Haines)	828
The Testing of Portland (Giron)	39	Process and Apparatus for Manufacture of. (P) Kellner.	152
Trade of Germany, The. (T.R.)	1237	The Commercial Production of, by the Ammonia-Soda Process. (Bale) (illus.)	206
Use of "Albano" as a. (P) Renison	648	Chloroform, Examination of, for Phosgene	1099
Utilising Precipitated Sewage for Manufacture of. (P) Bruch)	1198	Oxidation of, by Chromic Acid. (Erdmann) (illus.)	65
Cements. (Class IX.)	39, 154, 251, 396, 519, 637, 736, 811, 888, 949, 1062, 1198	Cholesterol, Separation of, from Wool Fats. (P) Krause and Mayer	162
And Cement Work. (P) Ropes and Sellars.	639	Choline and Betaine in Wormseed, Occurrence of. (Jahns)	57
For Leather Belting	164	Preparation of. (Schulze and Frankfurt)	54
The Testing of Hydraulic. (Stanger and Blount) (illus.)	155	Chromic Acid Discharge for Silk. (Kochlin)	737
Transforming Roasted, into Powder, and Apparatus therefor. (P) Belloc and Benard.	1198	Acid, Oxidation of Chloroform by. (Erdmann) (illus.)	67
Centrifugal Machines for Separating Liquids. (P) Ludloff (illus.)	240	Acid, Preparation of. (P) Placet and Bonnet.	57
Cereals, Manufacture of Dough from. (P) Gellinek	56	Chromium, Action of Iron Sulphide on.	1093
Manufacture of Prepared. (White)	635	Carbide, Production of. (Mullhauser)	35
Peptonised Extract of Malted and other. (P) Peptine		Extraction of, by Electrolysis, and Production of Chromium Alloys. (P) Placet and Bonnet.	643
Maltine, Lim., and Bartlett.	1083	In Chromium Steel and Chrome Iron Ore, Estimation of. (Spüller and Kalmann)	281
The Diastase of. (Ezgaroff)	1089	In Steel, Estimation of. (Schneid.)	546
Cetoses and Aldoses, Differentiation of. (Villiers and Fayolle)	1698	Lakes, Dyeing Wool with. (P) Hoffmann	881
Chalybeate Water, Apparatus for Producing Artificial. (P) March	529	Making Green Oxide of. (P) Socum and Wheeler.	1071
Chamber-Kilns, Improvements in. (P) Jung.	154	On the Mordanting of Wool with. (Lieschi and Hummel)	356
Champaecol. (Merek)	831	(Part III.) 222, (Part IV.) 356	
Changes of Address. <i>See under Address</i> .			

	PAGE
Chlorine, Preparation of, from Manganese Dioxide and Hydrochloric Acid, (P. K. Smith).....	381
Chlorine, Properties of, (P. K. Smith).....	509
Chlorine, Uses of, (P. K. Smith).....	757
Chlorine, Manufacture of, and Ripping of the, (Lundell).....	74
Chlorine, Manufacture of, Variety of Applications, (Tunelle).....	266
Chlorine, Back Production of Total Alkaloids in, (Keller).....	1104
Chlorine, Back Production of, (F.R.).....	1118
Chlorine, Back Production of, (F.R.).....	1195
Chlorine, Back Production of, (F.R.).....	907
Chlorine, Back Production of, (F.R.).....	58
Chlorine, Back Production of, (F.R.).....	657
Chlorine, Back Production of, (F.R.).....	78
Chlorine, Back Production of, (F.R.).....	416
Chlorine, Back Production of, (F.R.).....	1115
Chlorine, Back Production of, (F.R.).....	417
Chlorine, Back Production of, (F.R.).....	972
Chlorine, Back Production of, (F.R.).....	184
Chlorine, Back Production of, (F.R.).....	752
Chlorine, Back Production of, (F.R.).....	810
Chlorine, Back Production of, (F.R.).....	676
Chlorine, Back Production of, (F.R.).....	80
Chlorine, Back Production of, (F.R.).....	275
Chlorine, Back Production of, (F.R.).....	57
Chlorine, Back Production of, (F.R.).....	758
Chlorine, Back Production of, (F.R.).....	1216
Chlorine, Back Production of, (F.R.).....	409
Chlorine, Back Production of, (F.R.).....	251
Chlorine, Back Production of, (F.R.).....	1062
Chlorine, Back Production of, (F.R.).....	1198
Chlorine, Back Production of, (F.R.).....	396
Chlorine, Back Production of, (F.R.).....	153
Chlorine, Back Production of, (F.R.).....	33
Chlorine, Back Production of, (F.R.).....	118
Chlorine, Back Production of, (F.R.).....	944
Chlorine, Back Production of, (F.R.).....	78
Chlorine, Back Production of, (F.R.).....	824
Chlorine, Back Production of, (F.R.).....	25
Chlorine, Back Production of, (F.R.).....	689
Chlorine, Back Production of, (F.R.).....	422
Chlorine, Back Production of, (F.R.).....	184
Chlorine, Back Production of, (F.R.).....	773
Chlorine, Back Production of, (F.R.).....	242
Chlorine, Back Production of, (F.R.).....	797
Chlorine, Back Production of, (F.R.).....	1182
Chlorine, Back Production of, (F.R.).....	1106
Chlorine, Back Production of, (F.R.).....	887
Chlorine, Back Production of, (F.R.).....	172
Chlorine, Back Production of, (F.R.).....	416
Chlorine, Back Production of, (F.R.).....	1218
Chlorine, Back Production of, (F.R.).....	1217
Chlorine, Back Production of, (F.R.).....	1218
Chlorine, Back Production of, (F.R.).....	112
Chlorine, Back Production of, (F.R.).....	56
Chlorine, Back Production of, (F.R.).....	1083
Chlorine, Back Production of, (F.R.).....	268
Chlorine, Back Production of, (F.R.).....	757
Chlorine, Back Production of, (F.R.).....	537
Chlorine, Back Production of, (F.R.).....	270
Chlorine, Back Production of, (F.R.).....	536
Chlorine, Back Production of, (F.R.).....	656
Chlorine, Back Production of, (F.R.).....	1083
Chlorine, Back Production of, (F.R.).....	626
Chlorine, Back Production of, (F.R.).....	432
Chlorine, Back Production of, (F.R.).....	243
Chlorine, Back Production of, (F.R.).....	1238
Chlorine, Back Production of, (F.R.).....	379
Chlorine, Back Production of, (F.R.).....	850
Chlorine, Back Production of, (F.R.).....	933
Chlorine, Back Production of, (F.R.).....	1009
Chlorine, Back Production of, (F.R.).....	674
Chlorine, Back Production of, (F.R.).....	428
Chlorine, Back Production of, (F.R.).....	956
Chlorine, Back Production of, (F.R.).....	431
Chlorine, Back Production of, (F.R.).....	871
Chlorine, Back Production of, (F.R.).....	842
Chlorine, Back Production of, (F.R.).....	418
Chlorine, Back Production of, (F.R.).....	308
Chlorine, Back Production of, (F.R.).....	978
Chlorine, Back Production of, (F.R.).....	792
Chlorine, Back Production of, (F.R.).....	423
Chlorine, Back Production of, (F.R.).....	617

	PAGE
Colouring Matters and Dyes. (Class IV.).....	28, 142, 214, 381, 508, 628, 720, 797, 875, 933, 1054, 1155
Colouring Matters and Dyes, Various:—	
Acetone. (Kopp and Gran Inougen).....	729
Aldehydes, Condensation of, (Wurgraft).....	798
Alpha-beta-dimethyl-eurhodines, Manufacture of, (P. Johnson).....	879
Amid-naphtholdisulphonic Acid, Manufacture of a New, (P. Abel).....	32
Amides, Ac., Condensation Products from, (P. Newton).....	801
From The Farb. vorm. Bayer and Co. ....	1184
Aniline, Technical Method of Preparing, (Wohl).....	247
Auranine, Manufacture of, (P. Johnson).....	244
From The Badische Anilin und Soda Fab. ....	798
Aurine, (Herzig and von Smolchewski).....	632
Azo-Colouring Matters from New Amido Bases, (P. Kereff).....	32, 511
Azo-Colouring Matters, Production of, (P. Newton).....	631
From The Farb. vorm. Bayer and Co. ....	1054
Azo-Colours, Production of, (P. Shillito).....	23
Azo-Colours, Production of Insoluble on Fibre, (P. Inray).....	1055
From The Farb. vorm. Meister, Lucius, and Brünig ..... 23	
Azo Derivatives of Hydroquinone, (Witt and Johnson).....	1055
Bases and their Sulpho Acids, Manufacture of New, (P. Abel).....	519
From The Actien-Gesellschaft für Anilin Fab. ....	876
Basic Compounds from Pyrazine, Production of, (P. Newton).....	878
From The Farb. vorm. Bayer and Co. ....	809
Beta-anthraquinone, (Lazodinski).....	797
Beta-methyl-eurhodine, Manufacture of, (P. Wilcox).....	244
From The Badische Anilin und Soda Fab. ....	629
Betanaphthylaminesulphonic Acid, Ac., Preparation of, (P. Tobias).....	581
Brasilin and Haematocrylin, (Herzig).....	599
Carboxyl Derivatives of Dimethylaniline, (Lauth).....	1055
Carboxylic Acids of Dimethylaniline, (Lauth).....	802
Chrysin, (Dunier).....	942
Chrysin, (St. v. Kostanecki).....	1191
Colouring Matters and Condensation Products, (P. Durand and others).....	1190
Matters and Leuco Compounds of the Indigo Series, (P. Wilcox).....	145
From The Badische Anilin und Soda Fab. ....	631
Matters and Materials therefor, (P. Newton).....	946
From The Farb. vorm. Bayer and Co. ....	1189
Matters and Materials therefor, Production of, (P. Newton).....	725
From The Farb. vorm. Bayer and Co. ....	725
Matters and Products Related to the Rhodamine Series, (P. Johnson).....	145
From The Badische Anilin und Soda Fab. ....	631
Matters, Behaviour of Triphenylmethane, to Nascent Bromine, (Vauhel).....	946
Matters derived from Anthraquinone, (P. Newton).....	725
From The Farb. vorm. Bayer and Co. ....	145
Matters derived from Dioxidiphenylmethane, (P. Durand, Huguenin, and d'Andrim).....	631
Matter Derived from Esorine, (Ferreira da Silva).....	946
Matters Dyeing Blue with Mordants, (P. Abel).....	1189
From The Actien-Gesellschaft für Anilin Fab. ....	725
Matters Dyeing with Mordants, Production of, (P. Hays).....	725
From Sandoz ..... 946	
Matters, Effect of Metallic Salts in Development of, certain, on the Fibre, (Carbert and Peco).....	725
Matters for Unmordanted Cotton, (P. Petersen-Müller).....	725
Matters for Unmordanted Fabrics, (P. Inray).....	725
From La Soc. des Matières Colorantes ..... 145	
Matters from Alkylated Hydroxynaphthalene Sulphonic Acids, (P. Newton).....	800
From The Farb. vorm. Bayer and Co. ....	244
Matters from Amid-naphthol-sulpho Acids, (P. Newton).....	723
From The Farb. vorm. Bayer and Co. ....	946
Matters from Amid-naphthol-sulpho Acids, (P. Lake).....	632
From The Farb. vorm. Bayer and Co. ....	723
Matters from Amid-naphthol-sulpho Acids, (P. Lake).....	31
From The Farb. vorm. Bayer and Co. ....	941
Matters from Amid-naphthol-sulpho Acids, (P. Lake).....	723
From The Farb. vorm. Bayer and Co. ....	246
Matters from Amid-naphthol-sulpho Acids, (P. Lake).....	801
From The Farb. vorm. Bayer and Co. ....	722





	PAGE
various Matters and Dyes Various. <i>cont.</i>	
Sulpho Acids of Phenyl Sandoline, and Analogs and Homologues thereof. (P. Johnson. From The Badische Anilin und Soda Fabrik.)	519
Sulpho Acids. Production of. (P. Newton. From The Farb. vorm. Bayer and Co.)	578
Tannin Orange. (Schmidt)	1189
Trihydroxyphenylthale Monosulpho Acid. Production of. (P. Newton. From The Farb. vorm. Bayer and Co.)	1054
As Binding Material for Size. (P. Classen)	902
On a Method of Dyeing Leather with Basic. (H. G. and Procter)	895
Report of Committee on Action of Light. (P. Dyck. Hummel)	843
The Tinting of. (Dyck)	633
The Quinonoxanes in S. and. (Friedlemaier)	148
malin, The New Sodium Nitrate Deposits of. (T.R.)	1001
Castile Composition. (P. Odern)	1957
Position, Cause and Prevention of Spontaneous. (Hapke)	26
Chambers, Controlling Heating. (P. Land and Calkins)	997
Of Lamps. (H. Spontaneous. (Dyck)	670
Of Lamp Shades. Spontaneous. (Dyck)	670
Agency, Proceedings of Chambers of. (T.R.)	1231
Position for Coating. (P. M. and W. W.)	153
For Coating Iron and Steel. (P. Elliott)	992
For Coating Iron and Steel. (P. Walters)	993
For Incubators. (P. H. and P. H.)	793
For Moulds, Elastic. (P. H. and P. H.)	1211
For Preventing and Removing Boiler-Incrustation. (P. Borecki and K. H. and P. H.)	714
For Protecting Lead Surfaces. (P. H. and P. H.)	895
For Uses in. (P. H. and P. H.)	752
For Uses in. (P. H. and P. H.)	648
Improved Antifouling. (P. H. and P. H.)	1216
Positions for Preserving Beer and other Liquids. (P. H. and P. H.)	1083
Pickles. (P. H. and P. H.)	832
For Treatment of Sewage and Foul Waters. (P. H. and P. H.)	326
suitable for Paying. (P. H. and P. H.)	631
suitable, An Electric Insulating. (P. H. and P. H.)	807
For Production of Indigo upon the Fibre. (P. H. and P. H.)	656
For Purifying Sewage and Foul Liquids. (P. H. and P. H.)	260
For Seaming Textiles. (P. H. and P. H.)	661
For Use in the Manufacture of Matches. (P. H. and P. H.)	638
Production of an Asphaltic. (P. H. and P. H.)	759
ounds for Alimentary and Medicinal Purposes. Manufacture of. (P. H. and P. H.)	1082
For Manufacture of Ginger Beer and other Fermented Beverages. (P. H. and P. H.)	1209
For Removing Old Paint. (P. H. and P. H.)	1216
For Treating Sewage. (P. H. and P. H.)	532
For Waterproofing Leather. (P. H. and P. H.)	1216
For Use in Treating Sewage. (P. H. and P. H.)	158
Manufacture of Metallic. (P. H. and P. H.)	754
Manufacture of Oleum or Saponaceous, for Producing Medicinal Emulsions or Disinfectants. (P. H. and P. H.)	797
of Hydrogen Cyanide with the Alkaloids of the Sugars. (P. H. and P. H.)	745
of Iron with Fatty Acids. (P. H. and P. H.)	417
Of the Citral (Geraniol) Group. (P. H. and P. H.)	765
of the Sugars with Polyhydric Phenols. (P. H. and P. H.)	674
pendent General of. (P. H. and P. H.)	708
Concentrating Apparatus. (P. H. and P. H.)	661
Condenser, an Internally Cooled. (P. H. and P. H.)	626
Condensers, Gas. (P. H. and P. H.)	761
For Laboratories. (P. H. and P. H.)	930
Condensing Plates. (P. H. and P. H.)	569
Condensing House and Bradford. (P. H. and P. H.)	767
Condensing and Nitrogen. (P. H. and P. H.)	886
Condensing, Behavior of Woods of the, to Solutions of the Chlorides of Copper. (P. H. and P. H.)	835
Condensing and its Compounds. (P. H. and P. H.)	753
Condensing. (P. H. and P. H.)	1217
Condensing Alkaloids. (P. H. and P. H.)	71
Condensing and Reception. (P. H. and P. H.)	715
Condensing Apparatus. (P. H. and P. H.)	1081
Cop Bottoms, Paste or Size for. (P. H. and P. H.)	957
Copiba, Detection of Castor-Oil in Balm of. (P. H. and P. H.)	1222
Copper, Action of Copper Sulphate and Sulphuric Acid on Metallic. (P. H. and P. H.)	289
Analysis of American Redwood. (P. H. and P. H.)	886
And Lead, Separation of. (P. H. and P. H.)	1992
And Tin, Electrolytic Separation of. (P. H. and P. H.)	817
Behavior of Woods of the Conifer to Solutions of the Chlorides of. (P. H. and P. H.)	957
Compounds, Delicate Reaction for. (P. H. and P. H.)	240
Compounds, Electrolytic, in the Stillberg Lead Works. (P. H. and P. H.)	

	PAGE
Copper— <i>cont.</i>	
Estimation of, as Copper Sub-sulphide. (Wegscheider)	67
Experiments on Analysis of Pig. (De Benville)	667
From Chaldean and Egypt, Composition of Ancient. (Berthelot)	1198
Modification of Ferric Chloride Process for Estimation of Arsenic in. (Platten)	321
Occurrence of, in Extracts. (Holl)	902
On New Alloys of. (Von Knorre)	815
Ores of, Peta, the. (T.R.)	435
Phosphor. (McComb)	1139
Plate Ink, Manufacture of. (Villon)	721
Preparations, Recovery of Arsenic from. (P. North)	1291
Process and Apparatus for Smelting and Refining. (P. Vienna)	644
Production of Japanese. (Gowland) (illus.)	463
Recovery of, from Deacon Process Bricks. (P. Wyle and Williams)	1059
Silver Alloys, Liquefaction of. (Matthey)	889
Simple Method for Determining Foreign Metals in. (Hampel)	421
Sulphate, German Trade in. (T.R.)	1111
Sulphate, Manufacture of. (P. Strawson and Fream)	57
Sulphate, The Electrolysis of. (Chassy)	1204
Solutions, Behaviour of. (Jannasch and Lesnisky)	67
Volumetric Determination of. (Bornträger)	67
Corrections to be Applied in Titrating Liquids containing Suspended Matter. (Lenoble)	762
Corrosive Sublimates, Stability and Preservation of Dilute Solutions of. (Vignon)	171
Sublimates, Stability of Dilute Solutions of. (Vignon)	1087
Sublimates, Stability of Solution of, exposed to Air. (Tanret)	657
Corybulline. (Dobbie and Lauder)	1217
Corydalis. (Dobbie and Lauder)	1217
Corydalis Cava, Alkaloids in the Root of. (Freund and Josephi)	59
Cava, The Alkaloids of. (Dobbie and Lauder)	1217
Coto-bark, A New Derivative from. (Ciancin and Silber)	756
Cotton. (Class V.) 32, 145, 248, 384, 512, 632, 725, 802, 880, 944, 1056, 1192	
Cloths for Filtering Purposes, Treatment of. (P. Newton. From de Haen)	34
Cloths, Method for Bleaching. (P. Schott)	631
Dyeing with Benzidine Colours. (Mullers)	883
Fabrics and Yarns, Dyeing certain Colours on. (P. Hay and Park)	391
Fabrics, Printing on. (P. Jantsch)	513
Fibre, The Tendering of, by Tartaric Acid. (Scheurer)	148
Formation of R. sorcinol-green on. (Pokorny)	727
Goods, Paste or Size for. (P. Brown)	725
Goods, The Tendering of, by Iron Mordants used in Dyeing. (Scheurer)	148
Oil Industry in South Carolina. (T.R.)	992
Producing Fast Colours on. (P. Murray. From the Farb. vorm. Meister, Lucius, and Bruning)	883
Seed, Machinery for Cleaning. (P. Bibby)	1070
Seed Oil, Analysis of Lubricating Oils containing Blown. (Stillman)	286
Seed, Removing Fibre from. (P. Silcock)	945
Seed, The Proteids of. (Osborne and Voorhees)	828
Seed, Treatment of, for Removal of Fibres. (P. Thompson. From Stead)	820
Seeds, Apparatus for Cleaning. (P. Tattersall and Pearson)	803
Spontaneous Combustion of Oils Spread on. (Mackey)	1164
Stearin and Maize Oil. (Hart)	257
The Position of Lardwood on. (Bungert)	389
The Position of Colloid. (T.R.)	674
Council, Annual Report of	691
Coupling for Vessels containing Gases under High Pressure. (P. The Manchester Oxygen Co. and Jackson (illus.))	22
Crape, Artificial Production of Woollen. (Binder)	1192
Crapes, the Glossing of Woollen. (Kopp)	1193
Cream and Butter, Centrifugal Separators for. (P. Wahlin)	56
And Milk, Preserving, for Transit. (P. Casse)	537
Preparation and Employment of. (P. Monfort and Prior)	412
Production of. (P. Julien and Brin)	55
Cresosote, Apparatus for Burning. (P. Allen (illus.))	1050
Producing Esters from. (P. Abel. From von Heyden Nachfolger)	275
Cresosotes from Oak and Beech Wood, Composition of. (Béhal and Coisy)	1187
Qualitative Composition of Official Beech and Oakwood. (Béhal and Coisy)	1087
Cresol as an Anti-septic. (Lacroix-Hunkinbyendian)	273
Producing Esters from. (P. Abel. From von Heyden Nachfolger)	275
Cresols, Volumetric Estimation of the Three Isomeric. (Keppler)	1099
Crimbles, Manufacture of. (P. Digby)	158
Crushing Apparatus for Dry or Wet Materials. (P. Morel and Heimpel)	623
Cupene. Ethyl.	1086
Cupwine. Propyl.	1085

## PAGE

Customs and Excise Regulations in Great Britain, New (T.R.)	818
Decisions in France, (T.R.)	182, 989
Decisions in Russia, (T.R.)	909, 989, 1232
Decisions in Switzerland, (T.R.)	182, 989
Decisions in the United States, (T.R.)	71, 182, 769, 818, 910, 1232
Law, New, of the Argentine Republic, (T.R.)	182
Regulations, ....	71, 182, 289, 430, 559, 672, 769, 816, 909, 989, 1232
Tariff Changes in Canada, (T.R.)	672, 769
Tariff, New, of British India, (T.R.)	559
Tariff, Some Contemplated Changes in the United States, (T.R.)	552
Cyanide Poisoning, Cobalt Nitrate as an Antidote for, ....	1106
Process in the Transvaal Mines, The, (Feldtmann)	951
Process, The, (Feldtmann)	952
Process, The, (Vogel)	739, 950
Use of Ammoniacal Mercuric in Quantitative Analysis, (Schmidt)	548
Works, Notes and Queries on, (Halbeel)	553
Cyanides, Ammonia and Fuel-Gas, Method and Apparatus for Manufacturing, (P) Boul, From Cowen (illus.)	110
And Ammonia, Obtaining of, Direct from the Air, (P) Young	1196
Apparatus for Obtaining, (P) Aitken (illus.)	517
Manufacture of, (P) Gilmour	37
Manufacture of, (P) Gordon	886
Production of, (P) Addie, Cunningham, and Macfarlane	635
Production of Alkali, (P) Siepmann	250
Cyanogen, Amount of, in Gas-purifying Material, and its Influence on Regeneration, (Burschall)	138
Apparatus for Obtaining, (P) Aitken (illus.)	517
Compounds, Manufacture of, (P) Gilmour	37
Compounds, Preparation of, (P) Hood and Salomon	1195
Cylinders, Means for Filling Pressure in Gas, (P) Manchester Oxygen Co., and Jackson (illus.)	1180
Protecting Steel, during Transit, (P) Lea	1180

## D

Damp-resisting Materials, (P) Goddard	1063
Darrieus' Theory of the Lead Secondary Battery, and Life of Plates, (Schoop)	1203
Deacon Process, Utilising the Broken Bricks of the, (P) Wyde and Williams	1059
Deaths, Lists of	1, 95, 197, 308, 447, 691, 927
Deductions from Analysis of India-rubber Articles, (Henriques)	70
Defecation Experiments with Beetroot Juice, (Herzfeld)	823
Densities of Solutions of Soda and Potash, (Pickering)	511
Derivatives of the Oxazine and Eurholine Series, (Lauth)	628
Dervaux Feed-Water Purifier, The, (Simon Thomas)	376
Dessicators, Stop-cock for Vacuum, (Erast) (illus.)	62
Designs on Waterproof Fabrics, Producing, (P) Frankenbure and Weber	725
Desulphurisation: The Action of Iron Sulphide on Various Metals	1063
Detergent Compound for Seaming Textiles, (P) Brothers	260
For Washing Linen, (P) Gross	261
Detonators. <i>See also</i> Fuses and Explosives.	
Developers, Manufacture of Photographic, (P) Abel, From The Actien Gesellschaft für Anilin Fab.	659
Dextrin, German Trade in (T.R.)	1112
Dextrins, Production of, (P) Abel, From Pieper	824
Dextrose, Cause of Biotation of, (Tollens)	105
Fermentation of, by Lavalactic Ferment, (Tate)	268
Diamido base and Tetrazo Colouring Matters therefrom, Manufacture of a, (P) Thompson and Moore	384
Base, Manufacture of a New, (P) Inray, From The Farb, vorm. Meister, Lucius and Bruning	384
Bases, Manufacture of New Unsymmetric, (P) Durand Huguenin and d'Andiran	143
Bases, Manufacture of Unsymmetric and Colouring Matters therefrom, (P) Durand Huguenin and d'Andiran	32
-Dibenzimidazols, Manufacture of, (P) Inray, From The Farb, vorm. Meister, Lucius and Bruning	941
-Naphthalene-Sulpho Acid, Production of, (P) Pitt, From Cassella and Co.	244
Diaphragm for Electrolytic Processes, Materials for, (P) Parker	257
Diaphthol or Quinaseptol, (Guinard)	903
Diastase, Degradation of Starch by the Action of, (Lintner and Düll)	53
Of Cereals, The, (Egaroff)	1080
Reichler's Artificial, (Egaroff)	1080
Utilisation of Flours Rich in, (P) Briant and Walker	55
Diaz Compounds and Nitrosamines, (Schraube and Schmidt)	508
Dibromogallianilide, Some Blue Lakes derived from, (Cazeneuve)	1095
Diffusibility of Certain Gases through Caoutchouc Membrane, (Reychler)	151

## PAGE

Diffusion Battery, Combustible Gases, arising from, (Dewald)	107
In Vacuo by Heckmann's Process, (Manger)	106
Uses, Advantages of Heating, with the Steam from the Diffusion, (Mehcher)	6
Juice, Filtration of, after Heating	107
Juice, Purification of, (Ludke)	107
Report of Polish Commission on, (Muller)	719
Use of Sulphurous Acid in, (Muller)	719
Digester, Ac., for Bones, Fat, Meat, Glue, &c., (P) Warner	94
Digoxine, Preparation of Pure, (Kahn)	84
Dihydroxy-Naphthalene-Sulpho Acids, Manufacture of, (P) Johnson, From The Badische Anilin- und Soda Fab.	512
Dimethylamine, The Carboxyl Derivatives of, (Lauth)	14
The Carboxylic Acids of, (Lauth)	19
Dinner, The Animal	5
Dioxydiphenylmethane, Manufacture of Colouring Matter from, (P) Durand, Huguenin and d'Andiran	143
Dioxy-naphthalene-Mono-Sulpho Acid, Manufacture of a New, (P) Inray, From The Badische Chemical Works	512
Dioxy-naphthalene-Mono-Sulpho Acid, Manufacture of a New, (P) Inray, From The Badische Chemical Works	512
Discharge and Resist Styles with Aniline Black, On, (Peyth)	183
Discharges and Resist on Silk, (Kochlin)	727
On Aniline Black, (Kochlin)	127
On Aniline Black Grounds, Coloured, (Kay)	107
Discolouration of Papers made from Sulphite Cellulose, (Muth)	18
Discussion on Comparative Analysis of Land (Paper by Schweizer and Langwitz)	118
On Comparative Results of Modern Systems of Sewage Treatment	108
Disenses of Wines, (Kays)	70
Disinfectant, A Liquid for Use on, (P) Elliot	152
For Railway Sanitation, Tests of a, (Sodgwick)	7
Disinfectants, (Class XI. H.) 57, 170, 271, 313, 518, 659, 751, 831, 909, 1081, 1116	
Improvements in, (P) McDougall	657
Manufacture of, (P) Alder Wright	171
Manufacture of, (P) Molyneux and Potts	415
Manufacture of Compounds for Preparation of, (Klever)	74
<i>See also under</i> Antiseptics.	
Disinfecting, and Apparatus therefor, (P) Hermite, Cooper, and Paterson	171
And Apparatus therefor, (P) Pitt, From La Société Pictet	900
Apparatus, (P) Andersson and Suström	190
Improvements in, (P) Hermite, Paterson and Cooper	170
Distillation (Destructive), Tar Products, Ac., (Class H.) 27, 219, 350, 507, 627, 719, 791, 871, 938, 1053, 1182	
Apparatus for Fractional, (Volney) (illus.)	102
Apparatus for Fractional, (Otto) (illus.)	837
Method of Fractional, Illustrated by the Investigation of Kerosene, (Wauklyn and Cooper)	627
New Apparatus for Fractional, (Varenne) (illus.)	601
Of Substances having a High Boiling Point, (P) Jaffé and Darmstadter	230
Of Vegetable Substances, Obtaining Products of, (P) Grumbacher	628
Retorts for Obtaining Products of, (P) Bowing	573
Distillers' Washes, Process and Apparatus for Drying, (P) Action Maschinenbauanstalt, vorm. Venneth and Eilenberger	827
Distilling Apparatus, (P) Lavy	751
Pure Cultivation Yeast, and Use of Antiseptics in, (Class)	345
Refrigerator for Use in, (P) Worsam	190
<i>See also under</i> Apparatus.	
"Doctor" for Printing Machines, An Aluminium Bronze	28
Dough, Manufacture of, from Cereals, (P) Gelinek	16
Drainage, Hermet's Process for Purifying, by Electricity	98
Dressing for Boots and Shoes, (P) James	747
Drier for Paints, Manufacture of, (P) Bedford	803
Drug Trade, Canada and the American, (T.R.)	95
Trade of Japan and the New Treaty, (T.R.)	990
Trade of New York and the Tariff, (T.R.)	101
Drying Apparatus. <i>See under</i> Apparatus.	
Dubbing or Dressing for Leather, (P) Emden	117
Dulcin, Obtaining of, (P) Wetter, From Riedel	42
Duties, The New Spirit, (T.R.)	140
Duty on Sulphate of Potash and Soda in Finland, (T.R.)	290
Dyeing, Calico Printing, Paper Staining, and Bleaching, (Class VI.) 34, 118, 218, 350, 513, 633, 729, 893, 880, 1045, 1057	
Apparatus, (P) Gessler	100
Apparatus, (P) Obermayer	128
Improvements in Turkey-red, (Eben and Specht)	110
Improvements in Turkey-red, (Muller)	63
Indigo Blues, Vats for, (P) Grune	143
Machines, (P) Weldon	243
Machines, Rotary, (P) Crabtree	284
Note on the Different Theories relating to, (Vignon)	220
On Substantive, (Weber) theories relating to, (Vignon)	120
Process, A New, (Bannett)	729

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## E

	PAGE
Electro-plating, Process and Apparatus for. (P) Thompson. From Barber.....	959
Electrolysis, Apparatus for Producing Chlorate of Potash by. (P) Hunter.....	810
Extraction of Chromium by. (P) Placet and Bonnet.....	646
Manufacture of Bleaching-Solutions by. (P) Boulé. From Knechtel and Gelauner.....	250
Manufacture of Colouring Matters by. (P) Goebel.....	876
Of Alkaline Chlorides. (Oettlé).....	403
Of Chlorides and Mixtures thereof. (P) Lyte.....	404
Of Saline Solutions. (P) Lignard.....	818
Quantitative Analysis by. (Classen).....	547
Quantitative Analysis by. (Piloty).....	545
Quantitative Analysis by. (Rudorff).....	1223
Use of, in Technical Chemical Processes. (Linsley).....	645
Electrolytes, Electrical Transport of Heat in. (Bagard) (illus.) See under Batteries.	71
Electrolytic Analysis, Quantitative. (Classen).....	1225
Analytical Estimation and Separation. (Vortmann) (illus.).....	68
Electrolytic Apparatus. (P) Andreoli.....	160
Apparatus. (P) Castner.....	529
Apparatus. (P) Crancy.....	819, 819
Apparatus. (P) Hambury.....	45
Apparatus. (P) Kellner.....	161
Apparatus, Making. (P) Hargreaves and Bird.....	528
Corner Deposition in the Stollberg Lead Works. (Cohen).....	817
-Plating, Improvements in. (P) De Pass. From Thofehrn.....	45
Elements. See under Batteries.	
Physical Influence of, on Iron. (Arnold).....	612
Embroidery, Obtaining a Substitute for. (P) Lake. From The Fratelli Mora.....	1216
Emetics, The. (Adam).....	46
Emulsion, Production of Fat. (P) Ekenberg.....	46
Emulsions, Manufacture of. (P) Hebling.....	754
Manufacture of Compounds for Preparation of Medical (P) Kleyer.....	417
Silver Haloid, for Photography. (P) Prestwich.....	1198
Enamel Glass Letters, &c. Manufacture of. (P) Böhm.....	154
Lining Pipes with. (P) Herbert.....	39
Enamelled Goods, Manufacture of. (P) Claus.....	39
Enamels. (Class VIII.).....	39, 153, 251, 395, 518, 637, 735, 811, 888, 948, 1060, 1197
Manufacture of. (P) Thompson. From Leuchs.....	519
Engelmann's Process for Decoration of Glass. (Appert).....	1197
England and America, The Coke Problem in. (T.R.).....	850
and Germany, The Chemical Industries of. (T.R.).....	771
Eosin in Coloured Tomatoes, Detection of. (McElroy and Biczewski).....	284
Eremophila maculata.....	897
Eriodendron anfractuosum. (Ariandon).....	147
Errata, Table of. See Special Index, page lx.	
Erythroextrin. (Lüthner and Dull).....	53
Eserine, A New Test for and Green Colouring Matter Derived from the Alkaloid. (Pereira da Silva).....	68
A Study of. (Pittit and Polonovsky).....	758
Essences. (Class XX.).....	57, 170, 272, 415, 539, 657, 755, 834, 902, 970, 1085, 1216
German Trade in. (T.R.).....	1111
Esters from Creosote, Guaiacol or Cresol, Producing. (P) Abel. From von Heyden Nachfolger.....	275
Manufacture of. (P) Krafft and Roos.....	759
Ether Motor of P. de Susini. (Fregtag) (illus.).....	135
Ethers, Manufacture of Phenol and other. (P) Krafft and Roos.....	759
Of Sulphuric Acid, Production of. (P) Newton. From The Farb. vorm. F. Bayer and Co.....	60
Of the Alcohols, Manufacture of. (P) Otto and Verley.....	1219
Ethyl Alcohol, Chlorination of. (Altshul and Meyer).....	415
Phenols, The. (Behal and Choy).....	794
Ethylene, Action of Heat on. (Lewes).....	505
Action of Heat upon. (Lewes).....	873
Eucalyptol, Extraction and Estimation of. (P) Scammell.....	1106
Euphorbia Demmunda.....	897
Eurhodine, Manufacture of Beta-methyl. (P) Willcox. From The Badische Anilin und Soda Fabrik.....	878
Series on some Derivatives of the. (Lanth).....	628
Eurhodines, Manufacture of Alpha-beta-dimethyl. Johnson. From The Badische Anilin und Soda Fabrik.....	879
Evaporating Apparatus. See Apparatus.	
Exhausting, Method of, and Apparatus therefor. (P) Wright. Without an Air or Water-Air Pump. (Schall) (illus.).....	1182, 1221
Expenditure and Revenue for 1893, Statement of.....	574
Explosion Caused by Sodium Peroxide. (Dupré).....	108
Explosive, A New. (P) Boyd.....	1219
Composition. (P) Maurette.....	620
Compounds. (P) Newton. From Nobel.....	760
Compounds. (P) Buchert.....	1090
Improve Safety, and Manufacture of Same. (P) Evelyn-Lardet.....	61
Substances, The Agglomeration of. (Vieille).....	1089
Explosives, Matches, &c. (Class XXII.).....	61, 173, 276, 417, 543, 659, 760, 837, 973, 1083, 1219
Eighteenth Annual Report of H.M. Inspectors of, for 1893.....	837

Explosives—cont.	
Experiments on. (Biltz).....	173
From Nitrate of Ammonia and Resins. (P) Abel. From König.....	543
German Trade in. (T.R.).....	1113
Improvements in. (P) Edmunds.....	1099
Manufacture of. (P) Kabin.....	659
Manufacture of. (P) Lake. From Paper.....	975
Manufacture of. (P) Ritter von Dahmen.....	1219
On the Manner of Combustion of. (Vieljeux).....	679
Preparation of. (P) Maxim.....	1219
Researches on Modern. (Mannaband Ristori).....	973
Smokeless.....	1088
The Gelatin. (Sanford).....	276
Export of Tinctures and Essences, Revenue Concessions on. (T.R.).....	772
Exports of British Chemicals to British Colonies for 1893. (T.R.).....	851
Of British Chemicals to Foreign Countries for 1893. (T.R.)	852
Of Produce and Manufactures for 1892 and 1893, Abstract of. (T.R.).....	856
Extraction Apparatus. (P) Meikle.....	622
Apparatus. (Itani) (illus.).....	839
Apparatus. (Parker) (illus.).....	1177
Apparatus. A Simple. (Buttner) (illus.).....	418
Apparatus for Liquids. (Kurbatow) (illus.).....	978
Apparatus for Organic Chemistry. (Hagemann) (illus.).....	64
Mdl. (Lehmann) (illus.).....	950
Extracts. (Class XX.).....	57, 179, 272, 415, 539, 657, 755, 834, 970, 1085, 1214
Examination of Liquid. (Linde).....	909
From Malted Grain, Production of. (P) Faulkner and Johnson.....	1214

F

Fabric for Garments. (P) Luncheon.....	802
Method and Apparatus for Producing an Improved. (P) Wheeler.....	538
Fabrics, Bleaching or Cleansing Vegetable. (P) Riley and The Thornliebank Co.....	728
Dyeing certain Colours on. (P) Hay and Park.....	391
Dyeing, Mordanting, and Treating. (P) Reiffert.....	513
Machine for Dyeing various. (P) Liehtl.....	391
Machines for Washing, Bleaching, Dyeing, or Mordanting. (P) Owens and Whiteley.....	390
Manufacture of Rubber-Coated Waterproof. (P) Abbott and Wilks.....	248
Marking indelibly on. (P) Cross and Bevan. From Beadle.....	1191
Printing and Dyeing on. (P) Ostertsetzer.....	391
Printing on Cotton. (P) Iantsch.....	513
Production of Many-coloured, by Mordanting Varn before Weaving. (Lange).....	387
Rendering various, Sensitive to Light. (P) Junk.....	173
Resists or Discharges for use in Printing or Dyeing. (P) Whitehead.....	390
Resists or Discharges for use in Printing, with Aniline Black. (P) Whitehead.....	390
See also Fibres and Textiles.	
Fat and Glue, Apparatus for Extracting. (P) Machalski.....	648
Apparatus for Manufacture of Alimentary. (P) Corde- weener and de Kunwald.....	270
Emulsion, and Method of Producing Same. (P) Ekenberg In Bread, Note on the Determination of the. (Weibull).....	908
In Cheese, Estimation of. (Bondzynski).....	766
In Milk, Apparatus for Estimating. (P) Woosnam and The Dairy Supply Co.....	1091
In Milk, Soxhlet's Method of Determining. (Timpe).....	1101
Resist for Silk. (Koechlin).....	727
The Examination of Wool. (Herbig).....	1068
Fats, Oils, and Soap-making. (Class XII.) 45, 162, 257, 401, 529, 617, 744, 819, 891, 959, 1068, 1207	
And Oils, Improving or Preserving. (P) Watel (illus.).....	531
And Oils, Method and Apparatus for Deodorising. (P) Bull (illus.).....	531
And Oils, Producing Consistent and Semi-consistent, without Heat. (P) Pommerhauz.....	531
And Oils, the Methods of Testing. (Milliau).....	181
And Resins, New Method of Analysing. (Mellinney).....	658
Apparatus for Treating Substances containing. (P) Robbins.....	1070
Bleaching, Sweetening, and Purifying. (P) Aspinall. Hear, and Wise.....	1208
Compound Edible. (P) Winter.....	1083
Detection of Disguising and Perfuming Media in. (Holde) Ekenberg's Process for Refining. (Benedikt).....	891
Estimating Pyknometrically the Densities of Soft. (Zawal- kiewicz) (illus.).....	859
Estimation of Chlorine in. (Benedikt and Zikes).....	984
Freed from Odour or Flavour. (P) Abel. From Fabriques de Produits Chim. de Thann et de Mulhouse.....	406
Method of Separating. (P) Ekenberg.....	46
On the Analysis of. IV. Colour Reactions. (Lewkowitsch) Treatment of Alimentary. (P) Thompson. From Artus.....	917
See also Greases.	

Fatty Acids, Manufacture of Compounds of Iron with. (P) Huntington.....	746
Acids, the Separation of Volatile. (Wechsler).....	178
Matters, Separation of, from Water. (P) Weir.....	504
Feeding Stuffs Act and Regulations, Notes on. (Carter Bell).....	116
Fellowships, Foundation of, by City and Guilds of London Institute.....	1232
Felt, Sanitary. (P) Quilham.....	831
Fermentation, Action of Calcium Sulphite and Potassium Bisulphite on Alcohol. (Ravizza).....	236
And "Frets," Studies on Secondary. (Van Lee).....	575
Influence of Mineral Antiseptics on Lact. (P) Cassava and Reibel.....	269
In Tanning Liquors, Origin of the. (Henderson).....	659
In the Leather Industry. (Wood) (illus.).....	218
Organisms, Influence of Carbon Dioxide on the Growth and Life of.....	269
Sealed Seum.....	965
Fermentations, Boiling.....	968
Boiling. A Criticism on Hesse's Results. (Heimshamm) Treatment of Boiling (Feynman). (Hesse).....	967
Ferments, Increment of, to Action of Antiseptic. (Edmont).....	1213
Ferrie Compounds, Treatment of Sewage by. (P) Tanner.....	578
Oxide, Production of. (P) Lunge and Maxwell-Lyte.....	734
Ferrihydride Salts, Preparation of. (Kassner).....	392
Ferro Boron, Manufacture of. (P) Leiller.....	1201
Fertiliser, A Liquid. (P) Elliott.....	652
Fertilisers and Feeding Stuffs Act and Regulations, Notes on. (Bell).....	116
Manufacture of. (P) Seldner and Watson.....	573
See also under Manures.	
Fibre from Cotton-Seed, Removing. (P) Silcock.....	945
Of the Acave Americana.....	802
Rooting, Manufacture of Vegetable. (P) McTeer.....	737
The Dyeing of. (P) Lake. From Waldstein, Peter, and Spott.....	947
Fibres, Apparatus for Dyeing. (P) Rhodes and Perkins.....	883
Apparatus for Treating. (P) Bruntson, Linn, and Hanson.....	883
Apparatus for Treating, by Liquids. (P) Miller.....	884
Apparatus for Washing. (P) Dawson.....	802
Apparatus for Washing. (P) Lazarus and Jefferson.....	945
Dyeing, Sizing, Bleaching, &c. (P) Smith.....	513
Extracting and Preparing Vegetable. (P) Fellows, Crozier, and Ferguson.....	632
Influence of Structure of, on Attraction for Colouring Matters. (Von Georgievics).....	945
Method and Apparatus for Bleaching and Treating. (P) McMeekin.....	219
Method and Apparatus for Washing. (P) Kelly.....	34
Preparing, for Reception of Aniline Black. (P) Clapham, Lishman, Picard, and Vilechou.....	728
Process and Apparatus for Extracting Oil, &c., from. (P) Forbes.....	261
Process and Apparatus for Treatment of Textile Vegetable. (P) Gomes.....	218
Production of Colours on. (P) Newton. From The Farb, vorm. Bayer and Co.....	722
Production of Dyestuffs, Fast to Light and Washing on the. (P) Pitt. From Cassella and Co.....	390
Scouring, Bleaching, and Disinfecting. (P) Hughes.....	728
Synthesis of Azo-Colours on Animal. (Pokorny and Werner).....	888
The Retting of. (P) Pennington and Allison.....	725
To be used for Medical and Surgical Purposes, Rendering, Antiseptic. (P) Clutterbuck.....	833
Treatment of Vegetable Textile. (P) Gomes.....	944
See also Fabrics and Textiles.	
Films for Photographic Purposes, Manufacture of. (P) Blair.....	659
For Photographic Purposes, Manufacture of. (P) Pettit.....	659
Imitating Mother of Pearl. (P) Hahn.....	1072
Manufacture of Photographic. (P) Reissner and Hauser.....	375
Production of Continuous Cellulose. (Beadle).....	901
Separating Photographic Gelatin, from Supports. (P) Barrat and Hill.....	1088
Filter Beds, Construction of. (P) Cosham.....	1181
Cloths, Treatment of. (P) Newton. From de Haen.....	34
For Air, Gas, and Steam. (P) Moeller.....	714
For Diffusion Juice. (Bouvier).....	167
Presses. (P) Seitz (illus.).....	714
Presses. (P) Critchlow.....	929
Filtering Apparatus. (P) Jensen (illus.).....	239
Apparatus. (P) Allen.....	378
Apparatus. (P) Bristowe.....	639
Apparatus. (P) Oliphant.....	1049
Apparatus for Water. (P) Winkler.....	377
Material, Apparatus for Cleansing. (P) Reiser.....	378
Media, Apparatus for Washing. (P) Thompson. From Arnold (illus.).....	25
Media Manufacture or Restoration of. (P) Wetter. From Weinrich.....	240
Method and Apparatus for. (P) Prat.....	117
Filters for Purification of Water and Sewage. (P) Candy.....	169
Improvements in. (P) Delhotel and Joride.....	156
Improvements in. (P) Williams. From Spalding.....	137
Manufacture of. (P) Lake. From Bird and Co.....	872
Manufacture of Earthenware and Porcelain. (P) Prado and Medina-Santurio.....	811
Used in the Manufacture of Alcohols, Oils, Greases, &c. (P) Iseli.....	377

	PAGE
Fine Chemicals, Alkaloids, Essences, and Extracts. (Class XX.)	1216
Freeclays. Determination of the Refractoriness of. (Hofman and Demm).....	638
Free-damp in Mines. Apparatus for Detecting and Estimating. (P) Fletcher.....	603
Freeze-fracture pumps. Lechtun.....	512
Freeze Method for Extinguishing. (P) Vicerie.....	168
Freeze. See under Explosives.	
F. Needles, Composition of Essences of. (Bertram and Walker).....	750
Fish and Fish Oil. Treatment of. (P) Stanley.....	165
Extract or Essence, and Utilising Waste Products of Producing same. (P) Knebel and Sahlfeld.....	829
Livers, Furnace Pan and Concentrator for Boiling. (P) Lotherington and Spencer.....	162
Tallow, Refined. (Eitner).....	841
Flames, Minimum Proportion of Carbon Dioxide and Nitrogen in Air Extinctive of. (Clowes and Fehmann).....	1155
Flashing-Point of Mineral Oils. (Levinstein).....	116
Flask, a Modification of the Litre. (Giles) (illus.).....	277
Pyroping, for Standard Solutions. (Vanderpool) (illus.).....	692
Flax. Connection between Tenacity of, and Moisture in. (Lohry de Bruyn).....	385
Method and Apparatus for Bleaching and Treating. (P) McMeekin.....	240
Routine, Manufacture of. (P) McTeir.....	797
Floorecloth, Manufacture of. (P) Farmer and Storey.....	33
Flours. Rich in Dextrose, Utilisation of. (P) Briant and Walker.....	55
Fluids, Apparatus for Heating. (P) Fudicker.....	1181
Apparatus for Regulating Temperature of. (P) Royle.....	1179
Apparatus for Sterilising. (P) May and Newlands.....	751
See also: Liquids and Solutions.	
Fluorine, Influence of Compounds of, on Beer Yeast. (Effront).....	1079
Influence of Compounds of, on Beer Yeast. (Effront).....	1079
Flux for Soldering Aluminium and other Metals. (P) Thompson. From Nicolai.....	890
Fodder Plants, Poisonous. Jackson.....	897
Production of, from Residue of Oil Production. (P) Spindler and Stautz.....	893
Fohr's Methods of Estimation of Sulphur. (Marchlewski).....	283
Food and Oil from Peanuts, Manufacture of.....	530
An Improved. (P) Morit.....	898
An Improved Cattle. (P) Holland. From Holland.....	168
Cakes, Manufacture of. (P) Rehnstrom.....	1215
Composition for Use as, and Preparation of same. (P) Taffin.....	752
Injection of Salicylic Acid in. (McElroy).....	679
For Horses, Manufacture of. (P) Lake.....	752
For increasing the Production of Milk by Animals. (P) Klutentrichter.....	898
In Soils, Determination of probably "Available" Mineral Plants. (Dyer).....	288
Method and Means for Preserving. (P) Lillont.....	833
Preparations. (P) Moelson.....	1215
Preparation of, from Animal and Vegetable Substances. (P) Tippet.....	752
Preparation of Malted Pulse. (P) Watson.....	279
Preparations, and their Manufacture. (P) Clark.....	199
Preservation of. (P) Paulsen.....	370
Substances, Preservation of. (P) Sahlstrom.....	36
Treatment and Packing of, for Army Use. (P) Pfleiderer. From Warden and Billings.....	169
Foods, Manufacture of Invalids' and Children's. (P) Briggs. Method for Preserving Animal or Vegetable. (P) Vicerie. Preparation and Treatment of. (P) Maris.....	828 168 898
Ford and Co's Holyrood Glass Works. Visit to.....	702
Formaldehyde, Action of, on Vegetable Tissues. (Holfert).....	909
Dose and Estimation of. (Trillat).....	177
Formamides of Alizarin, the. (Prud'homme and Rabaut).....	143
Formic Acid, Determination of. (Lieben).....	288
Forth Bridge. Visit to the.....	703
France, Bougies on Manufacture of Shell Oil in. (T.R.).....	183
Customs Decision in. (T.R.).....	182
Manufacture of Hydraulic Mortar in. (Hauenschield).....	19
Margarine Production in. (T.R.).....	292
Margarine Trade in. (T.R.).....	1233
New Deposit of Phosphates in the South of. (T.R.).....	976
The Match Monopoly in. (T.R.).....	770
Franklin Institute, Rewards for Discoveries offered by the.....	73
Frost, Effect of, on Building Construction.....	949
Execution of Masonry and Brickwork during. (Greil).....	713
Fruit Industry of Portugal, The Preserved. (T.R.).....	956
Fuchsian, Constitution of. (Fischer and Jennings).....	112
Fuel, Gas and Light. (Class II.)..... 25, 138, 241, 378, 505, 625, 715, 789, 872, 931, 1049, 1182	715
A New, and Production thereof. (P) Scott.....	871
An Improved. (P) Nightingale and Wicking.....	140
Apparatus for Distributing and Burning Liquid. (P) M. Murrie (illus.).....	937
Apparatus for Obtaining more Perfect Combustion of. (P) Taylor.....	139

## Fuel—cont.

Apparatus for Supplying Liquid. (P) Etchells.....	934
Blocks, Manufacture of Artificial. (P) De Velum.....	243
Burner, The Holden Liquid. (illus.).....	932
Consumed in India. (T.R.).....	1116
Liquid. (Stockfleth).....	625
Liquid. (Hornwood).....	792
Manufacture of. (P) Hartridge.....	873
Manufacture of Artificial. (Kirbis).....	1182
Manufacture of Block. (P) Stanley.....	242
Method and Apparatus for Controlling Combustion of Solid. (P) Nobel.....	571
Obtaining Complete and Smokeless Combustion of. (P) Armstrong (illus.).....	1051
Obtaining Gases, Ammonia, and Tar from. (P) Mond (illus.).....	938
Fuels, Valuation of. (Fischer) (illus.).....	429
Fumigant for Use in Horticultural Buildings. (P) Smithson.....	960
Fumigants. (P) McDougall.....	657
Furfural, Detection of Sesame Oil by. (Villavechia and Fabris).....	69
Furnace and Apparatus for Working Ores. (P) Allison. From Storer, Martin, and Eaton.....	816
An Improved. (P) Dempster.....	871
A Recuperative Gas. (Bigot) (illus.).....	174
A Useful Electric for Laboratory Work. (Readman).....	375
For Burning Petroleum. (Crane) (illus.).....	21
Pan and Concentrator for Boiling Fish Livers. (P) Lotherington and Spencer.....	162
Furnaces, Electric. (P) Lake. From Girard and Street.....	626
Electric. (P) Chaplet.....	996
For Burning Refuse. (P) Elliott and McC. Paton (illus.).....	898
For Distilling Zinc, Cadmium, &c. (P) Golby. From Francis.....	402
For Drying and Coking Peat Briquettes. (P) Stauber (illus.).....	715
For Electric Smelting. (P) Newerth.....	169
For Obtaining Smokeless Combustion. (P) Armstrong (illus.).....	1051
Melting. (P) Bantzenberg, jun.....	816
Metallurgical. (P) Torres.....	1067
The Capacity and Form of Blast. (Hawdon).....	643
Furniture Polish. (P) Horn.....	803
Fusel Oil in Spirit, Sources of Error in Roes's Method of Estimating. (Glaserapp).....	982
Fusibility of Clays, The Calculation of the. (Wheeler).....	396
Of Mixtures of Isomorphous Salts. (Le Chatelier).....	634
Fusion Tests, Determination of High Temperature by. (Schneider).....	702
The Austin Process for Treatment of Pyritic Minerals by.....	812
Fuses, Electric. (P) Brain.....	769

## G

Galena, Treatment of, for Production of Lead, Lead Sulphate, and Lead Oxide. (P) Macdonald.....	491
Gallianilide, On the Homologues of. (Cazeneuve).....	755
Or Gallanol, Germicidal Action of. (Cazeneuve).....	755
Oxidophenolic Colouring Matters from. (Cazeneuve).....	720
The Metallic Compounds of. (Cazeneuve).....	58
Galloparatolide. (Cazeneuve).....	755
Oxidophenolic Colouring Matters from. (Cazeneuve).....	720
Galvanic Batteries. See Batteries.	
Garden Party at Messrs. Flemings.....	703
Gardenia Resin. (Heckel and Schlagdenhauffen).....	47
Gas. (Class II.)..... 25, 138, 241, 378, 505, 625, 715, 789, 872, 931, 1049, 1182	715
Analysis Apparatus. (Fischer) (illus.).....	62
And Air, Apparatus for Mixing. (P) Axdorfer and Sass.....	1050
A New Compound, Produced Electrically. (P) Hall.....	328
Apparatus, Difficulties in Working Parallel Series of. (Leybold).....	1049
Apparatus for Manufacture of Water. (P) Glasgow.....	934
Apparatus for Manufacture of Water. (P) Paisley.....	212
Apparatus for Manufacture of Water. (P) Westcott (illus.).....	1051
Apparatus for Washing and Purifying. (P) Marshall (illus.).....	716
Burners, Hoods for Incandescent. (P) Böhm and Crawford.....	627
Burners, Incandescent. (P) Murray. From Medler.....	242
Carburetted Coal, with Benzene. (Ries).....	873
Composition and Origin of Natural. (Phillips).....	790
Condensers. (P) Cutler.....	626
Conditions for Production of Generator, observed during Combustion of Carbon in Air. (Ernst).....	379
Cylinder Connection. (P) The Manchester Oxygen Co. and W. M. Jackson (illus.).....	22
Cylinders, Apparatus for Testing. (P) The Scotch and Irish Oxygen Co. and H. Brier (illus.).....	789
From Coal-Tar, Apparatus for Obtaining. (P) Smith.....	626
Filter for. (P) Medler.....	714



	PAGE		PAGE
<b>Gas—cont.</b>		<b>Gelsemium Semipervians.</b> Alkaloids of. (Cushny).....	59
From Oil, Processes for Manufacturing. (Macdonald).....	378	Genstein, Synthesis of. (Kostanetski and Tambor).....	507
From Paraffin Oils, and from Pure Members of Paraffin and Terpene Series. (Tschier) (illus.).....	931	Geraniol. (Barbier).....	60
Furnace, A Recuperative. (Bigot) (illus.).....	174	From Oil of Andropogon Schimmbus. (Rubio and Bouvenant).....	178
Heat of Combustion of Coal. (Agnittou).....	26	Manufacture of, from Citronella Oil. (P. Bertram).....	178
Hydraulic Mains. (P) Dunsmore.....	624	Germany, Alcohol in. (T.R.).....	654
In America, Water. (Bredel).....	715	And Russia, New Commercial Treaty between. (T.R.).....	551
In Guns, Apparatus for Measuring Pressure of. (P) (Casteholz).....	929	Chemical Industry of, since 1855. (T.R.).....	966
Lamps, Incandescence Bodies for. (P) Schneider.....	715	Chemical Trade of, since 1855. (T.R.).....	1110
Lighting, Incandescence. (P) Bernstein and Silbermann.....	793	Denaturing Alcohol in. (T.R.).....	184
Lightning, Manufacture of Bodies for Incandescence. (P) (Laughaas).....	141	Depression of the Chemical Industry in. (T.R.).....	551
Liquor, Treatment of Waste from. (P) Crowther.....	1050	Drying Potato Pulp for Cattle Feeding. (Dehne).....	975
Making Apparatus. (P) Hartley.....	1050	Electro-Chemical Society of. (T.R.).....	125
Manufacture, Commercial Position of Bye-Products of. (Mallet).....	719	Imports and Exports of Dyeing Materials for 1892 and 1893. (T.R.).....	913
Manufacture of. (P) Mansfield.....	871	Imports and Exports of Dyes by. (T.R.).....	1109
Manufacture of, and Apparatus therefor. (P) Dunsmore.....	934	Manufacture of Hydraulic Mortar in. (Hünnerschild).....	11
Manufacture of Coal, and Apparatus therefor. (P) Dvorkovitz.....	626	Mineral Production of, for 1893. (T.R.).....	554
Manufacture of, from Refuse Material. (P) Stanley.....	242	New Table Oils in. (T.R.).....	89
Manufacture of Illuminating. (P) de Verna.....	243	Production of Zinc in. (T.R.).....	71
Manufacture of Oil. (P) Modler.....	379	Proposed Analytical Association in. (T.R.).....	912
Manufacture of Water, in New York. (Lange).....	872	Proposed Nationalisation of Potash and Magnesium Deposits. (T.R.).....	558
Method and Apparatus for Generating Fuel. (P) Thwaite and Threlfall (illus.).....	717	Sugar Beet Returns of. (T.R.).....	292
Method and Apparatus for Producing. (P) Kitson (illus.).....	213	The Cement Trade of. (T.R.).....	1237
Method and Apparatus for Producing Fuel. (P) Boulton From Cowen (illus.).....	149	The Chemical Industry of. (T.R.).....	771
Or Air, Apparatus for Carburetted or Enriching. (P) Love and Weston.....	141	The Foreign Trade of, in 1893. (T.R.).....	290
Preparation of Carbon Black from Natural, in America. (Cabot) (illus.).....	128	The Iron-making of, and its Residual Products. (T.R.).....	558
Process and Apparatus for Using Producers, for Calcining Limestone. (P) Boulton, From Hatch (illus.).....	636	The Mining Industry of. (T.R.).....	1063
Producers. (P) Abel, From The Gas Motoren Fabrik Deutz.....	937	The Nitre Question in. (T.R.).....	559
Producers, Appliance for Breaking Down Crust in. (P) Hall.....	627	Ginger Beer Compounds for Manufacture of. (P) Heron.....	1082
Producers, Improvements in. (P) Duff.....	110	Glass, Pottery, and Enamels. (Class VIII.).....	39, 153, 251, 395, 518, 637, 735, 811, 888, 948, 1069, 1107
Producers, Improvements in. (P) Meldrum (illus.).....	242	Action of Acids upon. (Foerster).....	518, 1069
Production of, and Bye-Products, and Apparatus therefor. (P) Young.....	335	Articles Imitating Agate. (P) Solms-Baruth.....	39
Production of, from Mineral Oils, and Apparatus therefor. (P) Young (illus.).....	934	Decorating. (P) Shelley.....	919
Production of Illuminating. (P) Smith.....	873	Effect of the Atmosphere and Water upon. (Foerster).....	518
Production of Water, and Apparatus therefor. (P) Young (illus.).....	934	Engelmann's Process for Decorating of. (Appert).....	1197
Products of Combustion of, from Argand and Anker burners. (Grehant).....	1215	Letters, Manufacture of Enamel. (P) Bohm.....	1198
Purification of. (P) Fleischhauer and Bernstein (illus.).....	718	Lining Pipes with. (P) Horbert.....	154
Purifying Material, Amount of Cyanogen in. (Burschall).....	138	Making Transparent Photographs on. (P) Allison, From Forster and Rackwood.....	117
Purifying Material, Estimation of Sulphur in. (Meyer) (illus.).....	283	Manufacture of Tubes and Vessels of Covered. (P) Browne, From Kastner.....	949
Tanks for Storing Compressed. (P) Tammis.....	788	Method for Joining to Metals. (P) Boulton and Slater.....	637
Washers for Extraction of Tar, &c. from Coal. (P) Cripps (illus.).....	379	Ornamentation of. (P) Gibbs.....	888
<b>Gases, Apparatus and Method for Determining Density of. (Meslans) (illus.).....</b>	<b>64</b>	Process for Etching on. (P) Retzsch.....	154
Apparatus for Cooling. (P) Price.....	1048	Producing Pictures, Inscriptions, &c. on. (P) Gortitz.....	811
Apparatus for Rapid Determination of Combustible. (Pond) (illus.).....	544	Production of Enamelled Letters and Designs on. (P) Rodan.....	811
Combustible, Arising from the Diffusion Battery. (Dewald).....	749	The Chemical Behaviour of. (Foerster).....	518
Diffusibility of certain, through Caoutchouc Membranes. (Reyher).....	181	The Sealing of Wires in. (P) Lake, From Tibbits.....	545
Enclosed in Coal and Coal-dust. (McConnell).....	25	The Weathering of, and its Decomposition by Water. (Foerster).....	1069
For Carbonic Acid, Oxygen, and Carbon Monoxide, Apparatus for Analysis of. (Pfeiffer) (illus.).....	63	Glaze, Laundry. (P) Conroy.....	531
Heating, by Electricity. (P) Donaldson.....	529	Glazes, Manufacture of. (P) Thompson, From Leuchs.....	519
In Ducts and Towers, Motion of. (Jurisch).....	513	Glove-making Industry in Spain.....	821
In Air, Apparatus for Notifying Presence of. (P) Hardy.....	1091	Glucose, Cause of Bloatation of. (Tollens).....	165
Means for Destroying Obnoxious. (P) Baker.....	1084	Determination of. (Gaud).....	1229
Method and Apparatus for Detecting and Measuring Inflammable. (P) Tilghman (illus.).....	1052	Gravimetric Estimation of. (Gaud).....	1227
Method and Apparatus for Determining Density of. (P) Freres and Meslans.....	378	Syrups, Characteristics of. (Stone and Dickson).....	178
Preventing Intermingling of, passing through Liquids. (P) Hilliard.....	1196	Glucoses, Action of Bases on. (Jessen).....	264
Process and Apparatus for Electrical Treatment of. (P) Hall.....	528	Glucosides and Rhamnosides, Dyeing with. (Schmuck and Marchlewski).....	633
Pumps for Pumping or Compressing. (P) Murray (illus.).....	377	Of the Alcohols, The. (Fischer).....	167
Researches on the Phenomena of Oxidation and Chemical Properties of. (Phillips).....	790, 840	Glue. (Class XIV.) 49, 164, 498, 532, 619, 746, 821, 894, 964, 1072, 1209	
The Qualitative Reactions of. (Phillips).....	840	And Fat, Apparatus for Extracting. (P) Machalski.....	648
Treating Blast-Furnace, for Extracting Alkaline and other Compounds. (Murray) From Gredt.....	816	And Gelatin from Bones, Production of. (P) Grillo and Schroeder.....	408
Utilising the Pressure in Gas-Cylinders for Employing the. (P) Manchester Oxygen Co. and Jackson.....	715	Examination of, for Manufacture of Coloured Papers. (Schlossmann).....	424
<b>Gastrolabium.....</b>	<b>897</b>	Estimation of.....	424
Gauge, The Standard Wire.....	929	German Trade in. (T.R.).....	1112
Gelatin, Application of Bichromated. (Izarn).....	1106	Manufacture of Liquid. (P) Wiese.....	498
Explosives, The. (Sanford).....	276	Process and Apparatus for Cooling and Forming. (P) Hewitt (illus.).....	1075
From Bones, Production of. (P) Grillo and Schroeder.....	408	Putty and Cement, Composition and Treatment of Substances for Use as. (P) Walters.....	407
Manufacture of. (P) Spencer.....	897	Removing Impurities from. (P) Hewitt.....	1075
German Trade in. (T.R.).....	1112	Glue-stuffs, Valuation of. (Gantler).....	285
Gelsemine. (Cushny).....	59	"Gluten Particles" occurring in Wort, Beer, and Yeast. (Will).....	967
Gelsemine. (Thompson).....	59	Glycerin as an Agent for Softening Leather.....	164
		Process and Apparatus for Obtaining, from Spent Lyes. (P) Ruymbeke and Jobbins.....	1070
		Process for Distilling, and Treating the Residue. (P) Van Ruymbeke.....	1208
		Glycerol, Influence of, on Bacterioid Action of Phenol and Corrosive Subimate. (Lousi).....	403
		Inversion of Sugar by. (Borcht).....	1211
		Invertive Action of. (Donath).....	828
		Study of the Methods of Estimating. (Sulzer).....	287
		Valuation of Crude. (Heller).....	908
		Gold and Silver, Extracting, from Solutions thereof. (P) Raleigh.....	402
		And Silver, Extraction of, from Ores. (P) MacArthur, Ellis, and The Cassel Gold Extracting Co.....	526

	PAGE		PAGE
Gold— <i>contd.</i>		Hay, Heating and Spontaneous Combustion of. (Berthelot) ..	1108
And Silver, Method and Apparatus for Extracting, from		Heat, Action of, on Ethylene. (Lewes) .....	505, 873
their Ores. (P. Trug) .....	527	Allotropic Transformation of Iron under Influence of	
Condition of in Quartz and Calcite Veins. (Liversidge) ..	398	(Chapoy) .....	737
Electrolytic Separation of, from Silver. (Schubel) .....	743	Developed by Italian Ballistite. (Macnab and Ristori) ..	974
Extraction of, from Ore of Matte. (P. Moersch) .....	1067	Developed by Modern Smokeless Powders. (Macnab and	
Extraction of, from Ores, &c. (P. Montz) .....	956	Ristori) .....	973
Suggestions On the Origin of. (Liversidge) .....	398	Developed by Nitro-glycerin and Nitro-cellulose. (Macnab	
On Treatment of, at the Witwatersrand Goldfields. (De		and Ristori) .....	973
Meunthals (illus.) .....	326	Effect of, on Iodates and Bromates. (Cook) .....	734
Ores, The Chlorination of. (Goldshall) .....	155, 157	Electrical Transference of. (Houllevigne) .....	72
Precipitating, from Cyanide Solution. (P. Bell and		Electrical Transport of, in Electrolytes. (Bazard) (illus.)	71
Carter) .....	816	In Boilers, Loss of. (Scheurer-Kestner) .....	1049
Treating Zinc-Lead-Sulphide Ores containing. (P. Fell		In Combustion Chambers, Method of Controlling. (P)	
From The Elements Zinc C) .....	402	Land and Caulkins) .....	937
Tests on Extraction of, by Dilute Potassium Cyanide.		Of Combustion of Coal-Gas. (Agaitton) .....	26
Lloyd) .....	723	Loss of in Steam Engines of a Sugar Factory. (Classen) ..	748
Volumetric Estimation of. (Frammeschi) .....	204	Heating Apparatus. See Apparatus.	
Goldobium uncinatum) .....	897	By Electricity .....	789
Grain for Brewing, Method of Treating. (P. Bayley) .....	1082	By Electricity, Efficiency of, compared with Combustion.	
Obtaining Matte for Brewing from. (P. Baum) .....	268	(Wilke) .....	790
Process of Malting. (P. Blake) .....	268	Electrically by Induced Currents. (Illus.) .....	816
Production of Extracts from Malted. (P. Faulkner and		Heckmann's Process for Diffusion in Vacuo. (Muzge) .....	1210
Johnson) .....	1214	Hemp, Connection between Tenacity of, and Moisture in.	
Raw Conversion in the Brewery. (Salomon) .....	510	(Lobry de Bruyn) .....	385
Ores, Drying, Browsers' and Smelters', and Apparatus there-		Method and Apparatus for Bleaching, &c. (P) McMeekin.	249
for. (P. Fielder) .....	1214	Hempel's Experiments on Zinc Smelting in Blast Furnaces ..	811
Gumminee, Cellulose of the. (Smith) .....	537	Hempel's Process for Purifying Drainage by Electricity .....	898
Group Aine, A Colouring Matter in the Leaves of the.		Hides, Action of Salt on the Putrefying Bacteria of. (Haen-	
Schmuck, Knecht, and Marchlewski) .....	909	lein) .....	49
Graphite in Pig-Iron, Estimation of. (Crobauch) .....	665	Action of Tannin on. (Passler) .....	964
Manufacture of. (P. Johnson) .....	821	And Skins, Tanning. (P) Durio. ....	1074
Gases, Extracting and Preparing the Fibres of. (P)		Colouring Tanned, by Means of Smoke. (P) Hermann ..	964
Tellers, Crozier, and Ferguson) .....	632	Continuously Treating, in Tanning Liquor. (P) Brunning.	408
Grease, Recovery of, from Waste Suds. (P) Inston) .....	1070	Decomposition of, during Process of Tanning. (Von	
Greses, Bleaching and Purifying Refuse. (Virolleand) .....	1068	Schroeder and Passler) .....	49
Filters used in the Production of. (P) Iseli) .....	377	Does Tannin Tan? (Procter) .....	747
Treatment of Wool, for Separation of their Constituents.		Or Skins, Tanning. (Zahn) .....	822
(P) Hutchinson) .....	260	Softening and Cleansing. (P) Jackson) .....	1075
See also Fats.		Treatment of. (P) Lake. From Strauss) .....	51
Grease, The Mineral Industry of. (T.R.) .....	431	See also under Skins.	
Grey, A New Direct. (Report by Baumann in Paper by		Holden Liquid Fuel Burner. The. (Illus.) .....	932
Pitzold) .....	113	Homochelidonine, $\gamma$ - and $\beta$ - .....	836
Grids. See Batteries.		Homopyrocatechol, Action of Sulphuric Acid on. (Cousin) ..	60
Greiser's Method for Estimation of Sulphur. (Marchlewski) ..	283	Honey-Dew, Composition of. (Von Raumer) .....	1076
Guanoel, Crystallised. (Preyest) .....	972	Honey, Influence of Abundant Honey-dew on Character of.	
Manufacture of. (P) Thompson, From von Heyden		(Von Raumer) .....	1076
Nachfolger) .....	275	The Composition of Ling-tree. (Maquenne) .....	52
Producing Esters from. (P) Abel, From von Heyden		The so-called "Turkish." (Fajano) .....	652
Nachfolger) .....	275	Hoods for Incandescent Gas Burners. (P) Bohm and Craw-	
Guatemala, Tariff Changes in. (T.R.) .....	989	ford) .....	627
Gum. (Class XVI.) .. 52, 165, 263, 410, 533, 652, 718, 822, 965,		Hop-Tannin. (Hayduck and Goldiner) .....	966
1210		Hops. (Briant and Meacham) .....	534
Hazon of Siam (Ludy) .....	821	Composition of. (Anbry) .....	895
Bleaching Starch. (P) Siemens Bros. From Siemens and		Composition of, and their Influence on Wort. (Anbry) 411,	1077
Halse) .....	34	The Chemistry of. (Lintner and Buegener) .....	52
Manufacture of, from a Waste Product. (P) Hanson,		Treating Brewers' Spent (P) Adkins) .....	168
Castle, and Morrison) .....	410	Treatment of. (P) Connell) .....	896
Yeast. (Salkowski) .....	1213	Utilising Spent, for Food and Litter. (P) Greening) .....	655
Gums, On the Solution of Indian, by Hydrogen Peroxide.		Horn, Production of Substitute for. (P) Hartmann) .....	1075
(Koechlin) .....	653	Hungary, Commercial Museum of, at Budapest. (T.R.) .....	1235
Gunpowder. See also under Explosives.		Hydraulic Gas Mains. (P) Dunsmore) .....	626
An Improved. (P) Leonard) .....	61	Mortar. See under Mortar.	
Manufacture of. (P) Schneider) .....	975	Hydrazine Sulphate, Colour Reaction of, with Lignin, &c.	
Guns, Apparatus for Measuring Pressure of Gas in. (P)		(Nickel) .....	423
Castenholz) .....	929	Hydrobromic Acid, Testing for. (Villiers and Fayolle) .....	1092
Gutta-Percha and India-Rubber, Manufacture of Elastic		Hydrocarbon Burners. (P) Kiesel) .....	718
Material from. (P) Desprez) .....	1269	Lubricants, The Alkali Test for. (Lissenko and Stepanow)	177
A Substitute for. (P) Le Brocqay) .....	964	Oils, Burners for. (P) Lehning) .....	793
Note on. (Lazarde) .....	746	Hydrocarbons, Combinations of, with Picric Acid, &c. (Tilden	
Preparing for Various Purposes. (P) Hutchinson) .....	619	and Forster) .....	172
Gymnocladus Canadensis, Carbohydrates of the Fruit of.		Decomposition of, for obtaining various Products. (P)	
Stone and Test) .....	536	Wisse and Scheller) .....	160
Glasses and other Materials, Machinery for Drying. (P)		Discussion on Production of Oil-Gas from Terpene Series of	501
Haidan, From Gummer) .....	241	Distillation and Breaking up of Liquid. (P) Clark) .....	381
Effect of Addition of, to Portland Cement. (Schott) .....	251	Method and Apparatus for Burning. (P) Hill and Brett	
		(illus.) .....	139
		Method and Apparatus for Converting Non-Gaseous, into	
		Combustible Gas. (P) Siemens (illus.) .....	139
		Mixing for use as Heating or Lighting Agents. (P) Sahl-	
		strom and Parr) .....	139
		On Essence of Valerian, The Volatile. (Oliviero) .....	758
		Preventing Spontaneous Combustion of Liquid. (P)	
		Richter) .....	1192
		Hydrochloric Acid, Action of Gaseous, on the Platinum	
		Metals. (Dadley) .....	255
		Acid, Detection of. (Villiers and Fayolle) .....	1691, 1691
		Acid, Estimation of Sulphuric Acid in Commercial.	
		(Rurup) (illus.) .....	904
		Acid, Manufacture of. (P) Prentice) .....	250
		Acid, Results of Lunge-Rohrmann Plate-Towers for Con-	
		densing. (Lunge) .....	1034
		Hydrocyanic Acid, Detection of. (Autenrieth) .....	843
		Acid, Estimation of. (Gregor) .....	176
		Acid, Production of. (P) De Lambilly) .....	58
		Hydrocyanosulphide and Fuchsin, Constitution of. (Eiseher	
		and Jennings) .....	112



	PAGE		PAGE
Hydrofluoric Acid, Capacity of Vessels to Live in Media containing. (Sareb).....	673	India-Rubber— <i>cont.</i>	
Hydrogen Cyanide, Compounds of, with the Amides of the Sugars. (Von Miller, Floch and Strauss).....	767	Rolls of Machinery for Mixing and Calendering. (P) Brooks, From Davis.....	678
Dioxide in Atmospheric Air and in Aqueous Deposits therefrom. (Llova).....	753	Substitutes. (Henriques).....	47
Dioxide, On the Question of Atmospheric. (Schone).....	773	Substitutes, Analysis of. (Henriques).....	48
Oven for Drying. (Morse) (illus.).....	171	Waterproof Cloth, Manufacture of. (Webster).....	43
Peroxide, On the Solution of Indian Gums by. (Koechlin).....	653	Indian Dyestuffs, Timetorial Properties of Some. (Hummel and Perkin).....	313
Peroxide, Separation and Estimation of Metals in Alkaline Solution by. (Jannasch and Lesinsky).....	518	Indigo, $\alpha$ - and $\beta$ -Naphthalene. (Weichmann).....	29
Peroxide, Separation of Metals by. (Jannasch and Rose).....	1224	Blue, the Dyeing of, combined with other Colouring Matters. (Polonovsky and Nitzberg).....	149
Production of Pure, from Sodium. (Rosenfeld).....	249	Blues, Method and Apparatus for Dyeing. (P) Crane.....	728
Sulphide, Evolution of, in Boiling Beer, Wort and Hops. (Elion).....	267	Compound for Production of, upon the Fibre. (Fischer).....	847
Sulphuret Apparatus. (Kuster) (illus.).....	278	Cultivation in the Punjab. (T.R.).....	747
Hydrometers, Improvement in. (P) Keating.....	849	Dyeing, Hawking Machines for. (P) Stahley.....	728
Table for Comparison of.....	976	Estimation of Indigotine in. (Donath and Strasser) (illus.).....	426
Hydroquinone, Azo Derivatives of. (Witt and Johnson).....	28	German Trade in. (T.R.).....	1111
Hydrosulphites, Manufacture of. (P) Grossmann.....	1060	Industry in British India, The. (T.R.).....	994
Separation of. (P) Grossmann.....	1136	Manufacture of. (P) Lake, From Kalle and Co.....	145
Hydrosulphurous Acid, Manufacture of. (P) Grossmann 1060	1136	Manufacture of Refined. (P) Simpkin.....	382
Hydroxyanthraquinones, Production of Derivatives of. (P) Newton. From The Farb. vorm. Bayer and Co.....	144	Preparation, New Process of.....	741
Hydroxyamine, Preparation of Free. (Brühl).....	835	Salt: A New Dyeing Preparation. (Fischer).....	74
Quantitative Precipitation in Presence of. (Jannasch and Mai).....	176	The Electrolytic Reduction of. (Muller).....	387
Hydroxyl-Derivatives of Anthraquinolnquinone. (Graebe and Phillips).....	28	Use of Benzidine Colours in Discharging. (Stein).....	882
Hydroxynaphthalene Sulphonic Acids, Production of Alkylated. (P) Newton. From the Farb. vorm. Bayer and Co.....	115	Vat, Production of an. (P) Read, Holiday, and Sons, and Bindschadler.....	349
Hygroscopic Properties of Textiles, Research on the. (Schloessing, jun.) (illus.).....	146	Indigotine, Estimation of, in Indigo. (Donath and Strasser) (illus.).....	426
Hyponitrous Acid. (Thum).....	35	Reduction of, to Indigo White. (Coppelsroeder).....	388
Hyoscine. (Hesse).....	115	Indulines and Safranines, Relation between the. (Fischer and Hepp).....	30
		Ink, Manufacture of Copper-plate. (Villon).....	721
		Manufacture of Printing. (P) Babbly.....	145
		Inks, Treatment of. (P) Nienstaedt and Goldmark.....	511
		Insecticide, a Liquid. (P) Elbott.....	652
		Insecticides and Fungoid Extremators. (P) McDougall.....	657
		Improvements in. (P) Boulton. From Haase.....	833
		Production of. (P) McDougall.....	833
		Insects, Preservation of Timber from the Attacks of. (Dehe-ram).....	49
		Insulating Compound, an Electric. (P) Baarnhielm and Jer-nander.....	531
		Material, and Production of same. (Geitzsch).....	521
		Inventor, the Invasion of the. (Carey).....	1024
		Iodates and Bromates, Effect of Heat on. (Cook).....	734
		Iodide, Mercurous, obtained Crystallised by the Wet Process. (François).....	755
		Of Nitrogen. (Szuhay).....	61
		Iodides and Iodine, Extraction of, from Blast Furnace Gases. (P) Murray. From Great.....	816
		Electrolysis of. (P) Hargreaves and Bird.....	886
		Iodine, Estimation of. (Villiers and Fayolle) (illus.).....	1005
		Estimation of, in Presence of Bromine and Chlorine. (Groeger).....	420
		Extraction of, from Blast Furnace Gases. (P) Murray. From Great.....	816
		Luminescent. (P) Annine.....	836
		Number of Rosin, the. (McIlhenny).....	668
		Iodoform, Moist. (Vulpis).....	962
		Iodometry, Sodium Thiosulphate for. (Meineke).....	420
		Ipecacuanha, Note on. (Moeller).....	903
		Ireland, Coal in. (T.R.).....	773
		The Peat Bogs of. (Dvorkovitz).....	597
		Iridin, the Glucoside of the Iris Root. (De Laure and Tie-mann).....	272
		Iridium, the Electro-deposition of. (Dudley).....	255
		Iridol, A New Colour Reaction of. (Nickel).....	906
		Iron, Action of Aluminium on Cast.....	737
		Albumen Derivative, Obtaining, from Animal Organs. (P) Willeox. From Boehminger and Schuler.....	678
		Allotropic Transformation of, under Influence of Heat. (Charpy).....	737
		And Carbon, Transformations of, in Tempering. (Charpy).....	1066
		And Nickel, Alloys of. (Osmond).....	649
		And Nickel, the Alloys of. (Wedding).....	955
		And Steel, Coating, with Brass and other Alloys. (P) Fenby and Moore.....	499
		And Steel, International Standards for the Analysis of. (T.R.).....	185
		And Steel, International Standards for Analysis of.....	127
		And Steel, Manufacture of. (P) Sattmann and Homatsch.....	601
		And Steel, On the Oxidation and Corrosion of. (Thom-son).....	118
		And Steel Plates, Pickling. (P) Kirkman.....	645
		Baths for Toughening, Strengthening and Hardening. (P) Bates.....	645
		Carbide, Production of. (Muhlhauser).....	35
		Changes produced in, by Permanent Deformation in the Cold. (Charpy) (illus.).....	43
		Compounds for Treating Sewage, &c. (P) Levinstein.....	1216
		Compounds of Sugars with. (Evers).....	415
		Conversion or Purification of. (P) Boulton. From Kneuman, Singer, and Hatch.....	42
		Determination of Carbon in. (Hempel) (illus.).....	421
		Elimination of Sulphur from Molten. (P) Phillips.....	42
		Estimation of Graphite in Pig. (Crobauget).....	665
		Estimation of Phosphorus in Pig. (Spuller and Kaimann).....	175
Igniting Composition for Matches. (P) Meissner.....	417		
Imperial Institute Research Committee. (T.R.).....	293		
Imports during 1893, Analysis of Value of. (T.R.).....	853		
Of Foreign and Colonial Merchandise for 1892 and 1893. Abstract of. (T.R.).....	854		
Incarescent Bodies for Gas Lighting, Manufacture of. (P) Langhans.....	141		
Gas Lighting. (P) Bernstein and Silbermann.....	793		
Incarescences, Composition for. (P) Medhurst. From Chalmor.....	793		
Incrustation in Steam Boilers, Preventing. (P) Gane.....	504		
India as a Field for Industrial Enterprise. (T.R.).....	1115		
Botanical Experiments in. (T.R.).....	849		
Cinchona Cultivation in British. (T.R.).....	995		
Consumption of Chemicals in. (T.R.).....	1117		
Consumption of Copper in. (T.R.).....	1117		
Consumption of Manures in. (T.R.).....	1118		
Exports of, for 1892-3. (T.R.).....	1117		
Hide and Skin Industries of. (T.R.).....	1118		
Manufacture of Soda Water in. (T.R.).....	1119		
New Customs Tariff of British. (T.R.).....	550		
Oil Production of. (T.R.).....	1118		
Opium Industry of. (T.R.).....	1118		
Production of Glass in. (T.R.).....	1117		
Production of Paper in. (T.R.).....	1117		
Production of Salt in. (T.R.).....	1117		
Seed Industry of. (T.R.).....	1118		
Sugar Trade of. (T.R.).....	1119		
The Indigo Industry in British. (T.R.).....	994		
The Shellac Industry of. (T.R.).....	1236		
Trade with the United Kingdom. (T.R.).....	1117		
India-Rubber. (Class XIII.) 45, 162, 262, 406, 532, 648, 746, 821, 896, 962, 1071, 1209			
India-Rubber. (Christy).....	1138		
A Criticism. (Thomson).....	473		
Analytical Control of Vulcanisation of. (Henriques).....	987		
And Gutta-Percha, Manufacture of Elastic Material from. (P) Desprez.....	1209		
Articles, Deductions from The Analysis of. (Henriques).....	70		
Articles, On the Analysis of. (Weber).....	476		
Articles, The Analysis of. (Henriques).....	986, 987, 988		
Articles, The Analysis of. (Lobry de Bruyn and Van Leent).....	963		
Artificial. (P) Newton. From Nobel.....	1072		
A Substitute for. (P) King and Jelliffe.....	962		
A Substitute for. (P) Le Brocqy.....	964		
Devulcanising. (P) Gerber.....	964		
Goods, Analysis of. (Holde).....	425		
Goods Methods for Valuation of. (Lobry de Bruyn).....	986		
Industry in 1892, Report on the. (T.R.).....	81		
On the Vulcanisation of. (Weber).....	11		
Preparing, for various Purposes. (P) Hutchinson.....	649		
Production in Assam. (T.R.).....	433		
Regenerated. (Henriques).....	407		



Liquids—*cont.*

The Technical Estimation of, (Low).....	250
Volumetric Estimation of, in Tin Solutions or Platings, (Bayne).....	420
Leadon Pipes in Vitriol Manufacture, (Lambert).....	733
Leather, (Class XIV.) 49, 164, 408, 532, 619, 716, 821, 894, 963, 1072, 1209	
And Glove-making Industry in Spain.....	821
Articles, Treatment of, with Fatty Matters, (P) Gillibrand.....	651
Belted, Cements for.....	164
Dubbing or Dressing for, (P) Emden.....	717
Glycerin as an Agent for Softening.....	164
Industry, Fermentation in the, (Wood) (illus.).....	218
Industry of Mexico, The, (T.R.).....	992
In India, (T.R.).....	1116
Manufacture of, (P) Collin.....	894
Manufacture of a Substitute for, (P) Kumagaya.....	754
Manufacture of a Substitute for, (P) Lake, From Sachsenroder.....	164
Method for Disinfecting, (P) Vigerie.....	968
Obtaining a Substitute for, (P) Lake, From the Fratelli Mora.....	31
On a Method of Dyeing, with Basic Colours, (Hummel and Procter).....	496
Process and Compounds for Waterproofing, (P) Lewis.....	532
Rendering Colours Fast on, (P) Hardy, Brownword, and Bradley.....	894
Rendering Soft, Durable, and Waterproof, (P) Becker.....	164
Some Experiments on the Strength of, (McMillan) (illus.).....	585
Sugar-Content of, (Schroeder and others).....	1227
Sugar Content of Weighted, (Von Schroeder and others).....	1101
Treatment of, (P) Löbl and Atlas.....	51
Water contained in Air-dried Bark-tanned, (Von Schroeder).....	1073
Waterproofing, (P) Lewis.....	532, 532
Legality of Laboratory Stills, (T.R.).....	78
Lefcocones, Production of, (P) Abel, From Pieper.....	824
Lemon-Grass Oil, A Natural Unsaturated Ketone in, (Barbier and Bouveault).....	1086
-Grass Oil, The Aldehyde of, (Barbier and Bouveault).....	1086
Le-prosy, Cancer, &c., Preparation for Treatment of, (P) Watson.....	417
Lence-auramine, Blue Coloration Produced by, in Contact with Acids, (Rosenstiehl).....	629
Compounds, Manufacture of New, (P) Newton, From The Farb. vorm. Bayer and Co.....	722
Compounds Related to the Indigo Series, Production of, (P) Wilcox, From The Badische Anilin und Soda Fabrik.....	802
Le-vulin, Crystalline, (Schulze and Frankfurt).....	533
Light, (Class II.) 25, 138, 241, 378, 505, 625, 715, 759, 872, 931, 1049, 1182	
Action of, on Metastable State of Soda and its Applications, (Schoen).....	388
Action of, on Oxalic Acid, (Richardson).....	516
Action of, on Phenol, (Bach).....	968
Action of, on Textile Dyes, (Kischelt).....	939
Action of, upon Dyed Colours, Report of Committee on, (Hummel).....	803
Measurement of Radiation of, by the Radiometer.....	428
Rendering Various Fabrics Sensitive to, (P) Junk.....	173
Lignin, Colone Reaction of Hydrazine Sulphate with, (Nickel).....	423
Lignite, Vanadiferous, found in Argentina, (Kyle).....	738
Lime, Action of, in Rendering Clay Flocculent, (Sachse and Becker).....	409
And Chlorine, The Interaction of, (Veley).....	250
Cultivation in the West Indies, (T.R.).....	819
Defeating Diffusion Juice with Pulverised Slaked, (Mittelmann).....	106
Juice and Citric Acid, Production of, (T.R.).....	184
Kilns, (P) Dawson, From Quarez Freres.....	1062
Manufacture of Oxygen from Plumbate of, (Le Chatelier).....	27
Manufacture of Superphosphate of, (P) Prentice.....	250
Residue, Manufacture of Plaster of Paris from, (P) Kerr Salts, German Trade in, (T.R.).....	1111
Use of Caustic, in the Blast Furnace, (Bell).....	954
Utilising Waste Carbonate of, in Manufacture of Portland Cement, (P) Hill.....	255
Limestone, Process and Apparatus for Using Producer-Gas for Calcining, (P) Boulton, From Hatch (illus.).....	636
Lime-tree Honey, Composition of, (Maquenne).....	52
Lime-glass, The Porcelain Industry at, (T.R.).....	558
Linen, Detergent for Washing, (P) Gross.....	261
Linoleum, Manufacture of, (P) Farmer and Storey.....	33
See also Flocculth.	
Linseed Cakes, Determining Moisture and Oil in, (Aitken) (illus.).....	689
Oil, Examination of, (Filsinger).....	892
Oil, Note on Oxidised, (Reid).....	1020
Oil, Treatment of, (P) Bedford.....	406
Liquid for Use as a Fertiliser, Insecticide and Disinfectant, (P) Elliott.....	652
Refrigerating, (P) Warner.....	1181
Liquids and Solutions, Hydrostatic Apparatus for Treating, (P) Spence.....	713
Apparatus for Controlling Discharge of, from Tanks, (P) Strype.....	1180

Liquids—*cont.*

Apparatus for Cooling, (P) Price.....	1048
Apparatus for Cooling, (P) Waddy.....	578
Apparatus for Cooling and Warming, (P) Baker.....	715
Apparatus for Continuous Concentration of, (P) Harlock.....	23
Apparatus for Distilling, (P) Fouché.....	711
Apparatus for Distilling, (P) Morris and Edley (illus.).....	504
Apparatus for Effecting Intimate Mixture of, (P) Julien, (illus.).....	787
Apparatus for Electrically Heating, (P) Crump and Dowling.....	256
Apparatus for Electrolytical Decomposition of, (P) Bailey and Guthrie.....	818
Apparatus for Evaporating, (P) Latham and Gayot.....	713
Apparatus for Evaporating, (P) Morrell and Froehner.....	377
Apparatus for Evaporating and Cooling, (P) Scott (illus.).....	23
Apparatus for Filtering, or Separating Liquids from Solids, (P) Drummond.....	577
Apparatus for Heating or Evaporating, (P) Morison.....	622
Apparatus for Heating or Vaporising, (P) Heineberg.....	713
Apparatus for Raising, (P) Macer (illus.).....	23
Apparatus for Raising, &c., (P) Taylor.....	931
Apparatus for Regulating Temperature of Fermenting, (P) Collins.....	806
Apparatus for Sterilising, (P) Ortmann and Herbst.....	157
Apparatus for Treating Fibres by, (P) Miller.....	884
Apparatus for Weighing out, (Schweitzer and Lungwitz) (illus.).....	838
Centrifugal Machines for Separating, (P) Ludloff (illus.).....	240
Centrifugal Separators for, (P) Lundstrom and Burrell, (illus.).....	21
Charging and Sealing Vessels with Volatile, (P) Macnab and Dickson.....	929
Clarification and Preservation of Alcoholic and other, (P) Evans.....	827
Compound for Purifying Foul, (P) Whalley.....	656
Containing Suspended Matter, Corrections to be Applied in Titrating, (Lenoble).....	762
Decomposition of, by Contact with Powdered Silica, (Gore).....	428
Extraction Apparatus for, (Kupatow) (illus.).....	978
Prevention of Bumping in Boiling or Distilling, (Gerhardt).....	838
Process and Apparatus for Concentrating, (P) Klemmann (illus.).....	1048
Process and Apparatus for Purifying, (P) Wagner and Marr.....	751
Purification of Impure, (P) Melldrum.....	413
Self-Acting Apparatus for Raising or Forcing, (P) Kestner.....	241
Sterilisation of Alimentary, (Kuhn).....	1133
Tanks for Storing Volatile, (P) Tammis.....	788
Vaporisation of, (P) Boulton, From Penchen and Clarke.....	340
See also under Fluids and Solutions.	
Liquor, Defecation and Filtration of Diffusion, (Mittelmann).....	965
Liquors, Apparatus for Aerating or Raising, (P) Stone.....	655
From Petroleum Refining, Examination of Waste, (Zaloziecki).....	628
Method and Apparatus for Evaporating, (P) Scott.....	622
Obtaining Useful Products from Sulphite Process, (P) Ekman.....	1085
Litharge, Manufacture of, from Galena, &c., (P) Kilpatrick.....	158
Lithium, Preparation of Metallic, (Guntz).....	255
Lithography, Use of Aluminium in, (T.R.).....	291
Litter, Utilising Spent Hops as, (P) Greening.....	655
Liver, Formation of Sugar in the, (Cavazzani).....	845
Locust Bean, Treatment of, and Products from, the Seed of the, (P) Pearson and Tamton.....	1209
Logwood Extract, (Donath).....	982
The Dyeing of, on Cotton, (Banzeet).....	389
London Section: Opening of Session 1891 5.....	1013
Loom-pickers, Treatment of, with Fatty Matters, (P) Gillibrand.....	651
Lothian Coal Co., Visit to the.....	703
Louisiana, Sulphur Mining in, (T.R.).....	995
Lubricants, Manufacture of Pressure, (Villon).....	745
New Reaction for Detection of Soap in, (Schweitzer and Lungwitz).....	1178
Lucifer Match Works, Report of Committee of Enquiry on, (T.R.).....	75
Lumber, Artificial, (P) Fell, From Smith.....	811
Luminosity and Ozone, Production of, Electrically, (P) Andreoli.....	162
Luncheon at Messrs. Pullar's.....	706
Lunge-Rohrman Plate-Towers for Condensing HCl, Results of, (Lunge).....	1094
Lupinus albus, Alkaloids from the Seeds of, (Soldani).....	757
Lyes of Sulphite Cellulose, Utilising, (P) Mitscherlich.....	831

	PAGE	
Magnolia Root and Sap. (Rohr.) Red, Fish and Hepar.....	29	
Magnetic Deposits in Germany, Proposed Nationalisation of.		
(T.R.).....	578	
In Great Britain (T.R.).....	632	
Magnesium and Zinc Action of, on Metallic Solutions. Vill-		
berger and Buzig.....	68	
Chloride Separating Salts of Soda and Potassium Solu-		
tions. (P. Bell).....	517	
Pyrophosphate, Reliability of, for Estimating Phosphoric		
Acid. (Neubauer).....	1002	
Sulphate, Production of. (P. Piquet).....	248	
Maze Oil and Cotton Seaming. (Hart).....	257	
Malic Acid in Wines, Determination of. (Meke).....	284	
Malt, Apparatus for Preparation of. (P. Schickelschee and		
Gade).....	82	
Estimation of the Acidity of. Prior to Mashing.....	906	
Estimation of the Ready-formed Sugars in. (Jais).....	53	
Kilning of, in Relation to the Colour of the Malt Germ.		
Corn.....	267	
Preparation of, and Apparatus therefor. (P. Gengen)		112
Respiration of, on the Malt Kiln. Jaeschke (illus.).		1078
The Carbohydrates Pre-existing in. (Jakowetz).....		967
The K Lachryzogen. Corn.....		845
The Ready-formed Sugars of. Ehrlich.....		845
Maltizing, Employment of Sulphuric Acid in. (Kukla).....		968
Influence of Ferruginous Water in. (Kukla).....		1080
Maltol. Brand.....		1213
Composition of. Brand.....		806
Malts, Influence of Cane Sugar on Valuation of. (Pier)		1080
Manchester Chamber of Commerce Proceedings of. (T.R.)		1234
"Ship Canal" The.....		11
Manganese, Treatment for Production of Permanganates. (P)		
Metzgers.....		38
Manganese, Action of Iron Sulphide on.....		1063
Carbide, Production of. (Mullhauser-Pf.).....		35
Dichloride, Apparatus for Valuation of. (Thümann) (illus.).		979
in Minerals and Metals, Review of Methods for Estimation		
of. Saunero.....		112
In Steel, Estimation of. (Schneider).....		546
Oxide in Vases. (T.R.).....		772
Ores, Apparatus for Valuation of. (Christomanos) (illus.).		1221
Peroxide, Action of Ammonia Gas on. (Michel and Grand-		
montagne).....		37
Peroxide, Manufacture of. (P. Albright and Hood).....		517
Steel, Le Chatelier's.....		1198
Steel, Manufacture of. (P. Hadfield).....		741
Steel, Results of High Treatment on and their Bearing on		
Carbon Steel. (Hadfield).....		644
Trade of the Caucasus, The. (T.R.).....		558
Mang-Kashy, Colouring and other Principles contained in.		
Perkin and Hummel).....		910, 1054
Timctoral Properties of. (Hummel and Perkin).....		352
Mannte, Formation of, in Wines. (Shabot).....		750
Mannit, Fermentation of, by Lactobacetic Ferment. (Tater)		268
Optical Titration of. (Mullen).....		797
Mantle, Is the Auer Incandescent, Poisonous? (Cochran)....		1050
Mantles, or Hoods for Incandescent Gas Burners. (P. Bohm		
and Crawford).....		627
For Incandescent Gas Lights. (P. Blucher).....		1182
Mantua, Drinking Water obtained from Artesian Wells at.		
Bonato.....		752
Maure, An Improved. (P. Brothers).....		169
Manufacture of, from Sewage and Sludge. (P. Carter)....		964
Manufacture of Mineral. (P. Rodrum). From Heusel and		
Spethoff.....		1075
Potassum Phosphate as. (Beyer).....		469
Manures. Class XV. .... 165, 264, 400, 534, 652, 738, 804, 141,		1075,
1210		
(Colonial) in the United States. (T.R.).....		434
Comparative Value of Various Phosphatic. (Von Delan)		263
Examination of Artificial.....		980
Monometry of. (P. Newton). From Seybold and Hoeder		748
The Valuation of. (T.R.).....		434
Analysis of Artificial. (Hühfeldt).....		1224
Natural Fertilisers.		
Marble, Decaying Material in Titulation of. (P. Geze and		
Falk).....		638
Manufacture of Artificial. (P. Baumer and Peck).....		639
Marbles, Manufacture of Artificial. (P. Grand'Montagne)....		254
Marcari, Apparatus for Manufacture of. (P. Cordweener		
and De Kunwald).....		270
Compared with Natural Butter. (Jellies).....		827
In Butter, Comparison of Methods for Detection of. (Scyda		
and West).....		584
In Butter, Modifications of Reichert-Meisßl Method for		
Detecting. (Bunter).....		427
Production of France. (T.R.).....		292
Rendered Conspicuous by Addition of Phenolphthalein.		
(Hart).....		412
Trade in France. (T.R.).....		1254
Markets, Securing the Foreign. (T.R.).....		1169
Marking Inks and Pencils, Manufacture of Coloured. (P)		
Hickson.....		216
Martius Yellow, Poisonous Action of, and its Detection.		
(Vitali).....		423
Masonry and Brickwork, Execution of, during Frost. (Gail).		
		729

	PAGE
Masons in India. (T.R.)	1116
Massenette, Yield of Sugar from. (Claisen)	1210
Massenettes, Estimating Quantity and Purity of. (Hanns)	165
Estimation of Crystalline Sugar contained in. (Vivien)	1210
Match Monopoly in France. (T.R.)	779
Matches. (Class XXII. .... 61, 173, 275, 417, 543, 659, 769, 837, 975, 1088, 1219	
And Striking Surfaces therefor. (P) Herz	1090
Compound for Use in the Manufacture of. (P) Priester	661
German Trade in. (T.R.)	1113
Igniting Composition for. (P) Meissner	417
Manufacture of. (P) Thompson, From Lagneau	61
Non-Poisonous. (P) Simonet	173
The Inventor of Phosphorus. (Jensch)	837
Mc Ewan and Co's Brewery, Visit to Messrs.	709
Metal of Sunflower Cake. (Theodor)	99
Meat Extract, An Improved. (P) Macloskey, From Niemann	537
Treatment and Packing of, for Army Use. (P) Pfeleiderer, From Warden and Billins	169
Meats, Fluid, and Manufacture thereof. (P) Clark	168
Medicines, Importation of Patent into Turkey. (T.R.)	558
Meeting, Proceedings of the Thirteenth Annual	691
Melting-Point, Note on an Abnormal. (Byan)	60
Point of Corone Hydrochloride. (Hesse)	172
Point of Spermaceti, Method of Determining	65
Members Elected, Lists of. 2, 94, 196, 307, 447, 573, 690, 936, 1012	1132
Mercuric Acetylde. (Travers and Plimpton)	277
Chloride, Stability of Aqueous Solutions of. (Burcker)	1057
Cymide, Use of Ammoniacal in Quantitative Analysis. (Schmidt)	518
Phenolates, and Derivatives thereof. (Desesquelles)	835
Mercury Air Pump, New Form of. (Kahlbaum) (illus.)	760
Electrolytic Separation of, from Tin, Antimony, and Arsenic. (Schmucker)	280
Gallate: a New Antisyphilitic. (Brousse and Gay)	171
Thermometer for High Temperatures. (Mahlke)	62
Thermometer for High Temperatures. (Recklinghausen)	61
Meta-amidophenolphthaleines, Production of Colouring Matters from. (P) Imray, From the Farb. vorm. Meister, Lucius and Bruning	790
Metal, Apparatus for Galvanising Sheet. (P) Lagness	158
Apparatus for Refining, by Electrolysis. (P) House and Symon	1068
Articles, Method and Apparatus for Pickling. (P) Cowper-Coles	889
Coating Non-metallic Articles with. (P) Ash, Gill, and Green	401
Connecting, to Earthenware. (P) Drayson	811
Method and Apparatus for Electrically Heating or Working. (P) Thompson, From Collin	161
Plates, Improvements in Coating with Metals. (P) Watkins	1261
Plates, Wear of, by Action of Steam Jets. (Walter)	134
Powders for Bronzing. (P) Imray, From Sachs	805
Protection of, by Coating. (P) Cowper-Coles and Walker	1067
Varnishing, The Electro-Nickelling of. (P) Perle and Herrmann	1072
Metallic Powders, Manufacture of Coated. (P) Imray, From Sachs	958
Salts, Reactions of Acid Potassium Arsenite with. (Reichard)	732
Surfaces, Plating. (P) Marino	958
Metallurgy. (Class X.). 40, 154, 255, 308, 520, 639, 737, 812, 889, 959, 1063, 1198	
Metals, Action of certain on Acid Solutions of their Chlorides. (Ditte and Metzner)	43
Action of Concentrated Acids on certain. (Burch and Dodson)	525
Action of Gaseous HCl and Oxygen on the Platinum. (Dudley)	255
And Compounds, Position of. (P) Cowper-Coles and Walker	1067
Apparatus for Amalgamating. (P) Green	402
Apparatus for Electro-Deposition of. (P) Alexander	819
Apparatus for Manufacture of Alkaline, by Electrolysis. (Borchers) (illus.)	139
Apparatus for Production of, from Ors. (P) Berner	889
Coating, with Aluminium or its Diluent Alloys. (P) Broadwell	815
Effect of Mechanical Stress on Electrical Properties of. (Gray and Henderson)	957
Effect of Stress on Corrosion of. (Andrews)	1209
Electro-deposition of. (P) Rudholzner	529
Electrolytic Production of. (P) Hoepfner	714
Employment of Aluminium in Refining	955
Estimation of Free Acid in Solutions of Per-Salts of the Heavy. (Hollmann)	282
Extraction of, by Electricity. (P) Tauszig	403
Flux for Soldering. (P) Thompson, From Nicolai	890
From Complex and Refractory Ors. Obtaining. (P) Hall	741
In Alkaline Solution, Separation and Estimation of. (Jannasch and Lesinsky)	548
In Copper, Simple Method for Determining Foreign. (Hampel)	421
Influence of Temperature on the Tensile Strength of. (Rudolphi)	522

	PAGE
Metals—cont.	
Klein's Method of Shaping Electro. (Langbein).....	1205
New Method of Depositing. (Gottig).....	1200
Obtaining and Depositing, by Electrolysis. (P) Steuls.....	358
On the Second Group. Electrolytic Separation of. (Schmucker).....	280
Oxidation of the Alkali. (Holt and Sims).....	536
Pickling, to Remove Impurities. (P) Smith.....	536
Preclipping Precious, from Solutions. (P) Moldenhauer.....	815
Process and Apparatus for Separation of. (P) Mays.....	710
Process for Surfaceing. (P) Thompson. From Kiesel.....	12
Production of, with Nitric Acid, from Nitrates. (P) Darling and Forrest.....	617
Purification of, in their Moulds. (P) Taussig.....	101
Quantitative Separation of. (Jannasch and Lesnisky).....	67
Recovery of, from Ores. (P) Fuller.....	816
Separating and Purifying. (P) Mays.....	816
Separating, from Ores and Mixtures. (P) Besenfelder.....	816
Separating, from Scraps or Chippings. (P) de Fursac, Moisset, and Verry.....	109
Separation of, by Hydrogen Peroxide. (Jannasch and Rose).....	1221
Separation of, from their Dilute Solutions. (Mylus and Fromm).....	527
Sweep Smelting and Refining of. (Bettel).....	953
The Metallurgy of Sulphates of. (P) Mills.....	711
The World's Production of Noble. (T.R.).....	676
Treatment of Slimes containing Precious. (P) Raleigh.....	101
Metamargate of Soda, Action of Light on, and its Applications. (Schoen).....	388
Methyl Orange, Titration of Aniline and other Organic Bases with. (Lunge).....	667
Mexico, Discovery of Salines in. (T.R.).....	210
The Dyewood Industry of. (T.R.).....	431
The Leather Industry of. (T.R.).....	232
"Micro-Chemical" Reagents, Employment of, in Analytical Chemistry. (Lenz).....	766
Micro-organisms causing Diseases of Beer. (Fellowes).....	1080
Mignouette, Localisation of the Active Principle of. (Guignard).....	59
Milk, An Abnormal. (Greig Smith).....	613
Analysis, Centrifugal Methods of. (Scott).....	719
And Cream, Preserving, for Transit. (P) Casse.....	597
Apparatus for Estimating Fat in. (P) Woosnam and The Dairy Supply Co.....	1091
Compositions for Preserving. (P) Pickles.....	1083
Determination of Butter Fat in. (P) Tichborne and Thompson.....	663
Enriching, and Producing Cream and Butter. (P) Julien and Brin.....	55
Manufacture or Preservation of Condensed. (P) Ekin and Thew.....	828
Preparations, Manufacture of Condensed. (P) Brizes.....	828
Skin Milk and Whey, Their Comparative Composition and Specific Gravity. (Cookran).....	239
Soxhlet's Method of Determining Fat in. (Tunpe).....	1104
-Testers. (P) Pedersen.....	56
-Testing, with Rennet. (Leze and Hilsout).....	1099
The Curdling of. (Iles).....	536
The Determination of Casein in Cows'. (Slyke).....	181
Milk, Extraction. (Lehmann) (illus.).....	959
Mineral Acids, Detection of, in Presence of Organic Acids. (Nickel).....	423
Acids in Vinegar, Detection of. (Grizen).....	123
Antiseptics, Influence of, on Lactic Fermentation. (Chassevant and Riehet).....	269
Industries of South Russia. (T.R.).....	819
Industry of Greece, The. (T.R.).....	131
Lubricating Oils, Solubility Tests of Dark. (Hilde).....	663
Oil and Sperm Oil, Analysis of Mixtures of. (Lobry de Bruyn).....	120
Production of Austria, The. (T.R.).....	994
Production of Canada for 1893. (T.R.).....	553
Production of Germany for 1893. (T.R.).....	556
Production of the United States. (T.R.).....	181
Production of the United States for 1892 and 1893. (T.R.).....	622
Oils on Ships in the Tropics. (Lobry de Bruyn).....	67
Oils, Removal of Sulphur from, at Chicago. (Kiehe and Roume).....	257
Production of the United Kingdom for 1893. (T.R.).....	719
Statistics for 1893. (T.R.).....	551
Waters, Report of French Committee on Natural. (T.R.).....	776
Minerals, Apparatus for Separating and Purifying. (P) Beyer (illus.).....	832
Disintegration of Agglomerated. (P) Lockhart.....	88
Dissolving, Leaching, and Filtering. (P) Murray. From Lacy and Storer.....	711
The Austin Process for Treatment of Pyritic, by Fusion... ..	813
Miners' Squibs. (P) Powell.....	766
Mines, Apparatus for Detecting and Estimating Fire-damp in (P) Fletcher.....	66
Coal Dust in. (T.R.).....	18
Exploding Blasting Charges in. (P) Jaroljnek.....	973
Mining Industry of Germany, The. (T.R.).....	998
Moisture and Oil in Linseed Cakes. (Aitken) (illus.).....	668
Influence of, on Chemical Action. (Baker).....	76
In Wood Pulp, On the Estimation of. (Carter Bell).....	1

Molasses, Augmentation of the Fermentable Capacity of, (Greger).....	28
Manufacture of White Sugar from, (P) Habner.....	874
On the Fermentation of, (Edmond).....	333, 824
Molybdenum Method, Determination of Phosphorus by the, (Mylidertz).....	67
Mordant Dyes, The Theory of, (Loeberman).....	28
Mordants, Resists with Tannin, (Chasnovat).....	882
Tendering of Cotton Goods by Iron, used in Dyeing, (Scheurer).....	448
Morinda-root, Tinctorial Properties of, (Hummel and Perkin).....	348
Morphine, Reactions and Constitution of, (Voss).....	284
Sulphate, Manufacture of, (Calvert).....	172
Mortar, Manufacture of Cement, (P) Williams.....	196
The Manufacture of Hydraulic in France and Germany, (Hansenschild).....	94
Mortars, (Class IX.),... 30, 151, 251, 396, 519, 637, 736, 811, 888, 949	1062, 1198
Mosaic, Production of Waifer China, (P) Gale.....	549
Mother-Liquors of the Manufacture of Alum, Concentration of the, (Wiernik).....	731
Mother-of-Pearl, Filus imitating, (P) Hahn.....	1072
Motor, De Susini's Ether, (Frestag) (illus.).....	135
Mould, Changes produced in Bread by, (Hebebrand).....	412
Moulds, An Elastic Composition for, (P) Herisse.....	1211
For Terra-Cotta Blocks, (P) Boulton and Carr.....	918
Muffle for Sugar Assay, (Schweitzer and Langwitz) (illus.).....	858
Museum, Hungarian Commercial, at Budapest, (T.R.).....	1245
Musk, Investigation of Artificial, (P) Millman.....	1105
Manufacture of Artificial, (P) Millman.....	659
Production of Artificial, (P) Baur.....	903, 906
Musts, Removal of Lead from, after Clarification, (Born-trager).....	1230
Myrabolans, Extraction of Tannin from, (Von Schroeder).....	1073

# N

Naphtha Products, The Alkali Test for, (Lissenkow and Stepanow).....	177
Naphthalenes of Substituted Annaphenols (Ulzer and Holler).....	769
Naphthalene, Acidimetric Estimation of, (Kuster).....	844
Derivatives, Production of New Sulpho Acids of certain, (P) Johnson, From The Badische Anilin und Soda Fab.,.....	144
Derivatives, Some Sulphonations of, (Dressel and Kothe).....	875
Exemption of, from Coal Tar, &c., (P) Clark.....	847
(T.R.).....	847
Extraction of, from Coal Tar, &c., (P) Clark.....	797
Production and Application of New Dyes from, (P) Johnson, From The Badische Anilin und Soda Fab.,.....	63
Series, Production of Sulpho Acids of the, (P) Newton, From The Farb. vorm. Bayer and Co.,.....	246
Naphthaquinol, Condensation of Aldehydes with $\alpha$ , (Wurgaft).....	798
Naphthaquinone, Condensation of Aldehydes with $\alpha$ , (Wurgaft).....	798
Naphthol, Acidimetric Estimation of $\alpha$ and $\beta$ , (Kuster).....	844
And Naphthylamine Sulphonic Acids, New Method of Estimating, (Vaubel).....	548
Compounds of Mercuric Phenolates, (Desesquelles).....	835
Sulphonic Acid, Preservation of Wines by Means of $\beta$ , (Simibaldi).....	501
Naphthylamine, Preparation of $\beta$ , (P) Tobias.....	890
-Sulpho Acids, Production of, (P) Newton, From the Farb. vorm. F. Bayer and Co.,.....	890
-Sulphonic Acid, Preparation of a New $\beta$ , (P) Tobias.....	890
-Sulphonic Acid, Substantive Azo-Colouring Matters from 1-4-, (Bonal).....	947
Naphthykne Diamene, 2:3-, (Friedlander and von Zakrzewski).....	727
Naphthyl Red and Magdala Red, (Fischer and Hepp).....	29
Naphthylsulfamine, Preparation of Salts of $\beta$ , (P) Tobias.....	890
Narcene, Investigations on, (Martin, Freund, and Frankfurter).....	708
Nelson and Sons' Works, Visit to.....	701
Netherlands, Dyes and Chemicals on the Free List in the, (T.R.).....	231
Exemption of Naphthalene from Import Duty in the, (T.R.).....	847
New South Wales, Anthimony Mine in, (T.R.).....	78
York Drug Trade and the Tariff, (T.R.).....	1067
York, Manufacture of Water Gas in, (Lange).....	872
Zealand, Kauri Copal Drizzling in.....	47
Niagara, The Power of.....	1176
Nickel, Action of Iron Sulphide on.....	1067
And Iron, Alloys of, (Osmond).....	640
And Iron, The Alloys of, (Wedding).....	255
And Lead, Separation of, (Jannusch and Lesinsky).....	67
And other Metals, Electrolytic Production of, (P) (Heppner).....	701

	PAGE		PAGE
Nickel		Oil, Adulteration of Olive. (Oliveri)	45
History, Uses, and Distribution of. (Charleton)	640	Analysis of Mixtures of Sperin and Mineral. (Lobry de Bruyn)	426
Industry, The. Visit to	400	Analysis, Studies in. (Bullantyne)	1100
For Nickel Sulfate, Determination of. (Campbell)	666	And Food from Peanuts, Manufacture of	530
For Nickel Sulfate, Determination of. (Weston)	666	And Moisture in Lined Cakes. (Aitken) (illus.)	660
Plating, Preparation of Iron or Steel Plates for. (P)	814	Apparatus for Vaporising. (P) Lard	873
Quantitative Estimation of. (Schmidt)	1225	Apparatus for Vaporising and Burning Mineral. (P)	139
Qualitative Estimation of Common Impurities in. (Heitmann)	1066	Composition of Rape-seed. (Ponzo)	257
Sulphide, Oxidation of. (De Clement)	151	Cotton Stearin and Maize. (Hart)	257
Sulfate and Concime. (Hout)	767	Detection of Sesame Oil in Olive. (Musset)	423
Extraction of, from Tobacco Juice. (T.R.)	850	Examination of Linseed. (Filsinger)	892
Technical Products of, Bochesheim. (Schlossing, jun.)	1087	Extraction of, from Plants and Fruits. (P) Spindler and Stautz	893
Nitrate Discoveries in Colombia. Alleged. (T.R.)	993	Gas from Paraffin Oils, &c., Postponed Discussion on Production of	501
Fields of South Africa. (T.R.)	993	Industry, Improvement in the Mineral. (Mann)	875
Of Soda in Egypt. (MacKenzie)	611	Inspissated, for Electrical Insulating. (P) Gentzsch	820
Of Soda Market, The. (T.R.)	773	Rapeseed. (Henriques)	258
Of Soda, Statistics of, 1886-1892. (T.R.)	81	Manufacture of Garamol from Citronella. (P) Bertram	758
Nitrates, Electrolysis of. (P) Hargreaves and Erd.	886	New Constituents of Wood. (Looff)	1188
Electrolytic Production of Nitric Acid and Metals from. (P) Darling and Forrest	617	Note on Oxidised Linseed. (Reid)	1020
Estimation of, by means of the Nitrometer. (Henry)	764	Of Roses. (Bertram and Gildemeister)	834
In Egypt, Report of Commission on. (T.R.)	1236	Of Roses, Bulgarian. (Markownikoff and Reformatsky)	272
For Potable Water, Determination of. (Gill)	663	Of Rowan Berries (Parasorbic Acid) and its Isomerism with Sorbic Acid. (Doehner)	539
In Waters, Estimation of. (Fraser)	1095	Of Spike (Lavandula Spica). (Bouchardat)	60
Recovery of, in Sugar Factories.	749	Of Tea-Seed. (T.R.)	79
Nitre Question in Germany, The. (T.R.)	359	Of Turpentine in Alcohol, Determination of. (Perl)	427
Nitric Acid, An apparatus for the Continuous Production of. (P) Prentice (illus.)	329	Of Vitriol, Methods for Rectification of. (Tate)	206
Acid and Nitrous Acid, Action of, on Silk. (Vignon and Lisey)	32	Painting, Importance of Study of Chemistry of. (Petruschelsky)	262
Acid, Apparatus for Condensation of. (P) Newton. From Hart (illus.)	1137	Presence of Camphene in Spike. (Bouchardat)	458
And, Influence of Free, on Precipitation of Barium as Sulphate. (Browning)	176	Press, Hydraulic. (P) Greenwood, Batley and Lambert	406
Acid, Manufacture of. (P) Cartway	735	Presses. (P) Johnson. From Gonnelle	1048
Acid, Manufacture of. (P) Prentice	250, 250	Presses, Plates for. (P) Leechman	377
Acid, New Method of Performing Schloesing's Process for Estimating. (De Koninck) (illus.)	763	Process and Apparatus for Extracting, by Volatile Solvents. (P) McIlwaine and Hardingham	745
Acid, Production of. (P) Lunge and Maxwell-Lyte	731	Processes for Manufacture of Gas from. (Macadam)	378
Acid, Production of, from Nitrates Electrically. (P) Darling and Forrest	617	Properties of Sunflower. (Holde)	892
Nitrication, Influence of Potassium Salts on. (Dumont and Crochetelle)	652	Separator for Feed-Water Apparatus. (P) McDougall (illus.)	22
Nitrates in Waters, Estimation of. (Fraser)	1065	The Analytical Constants of Seal. (Chapman and Rolfe)	845
Nitrocellulose in Filter-Paper. (Cramer)	833	Treatment of Linseed. (P) Bedford	406
Nitro Compounds, Securing the Chemical Stability of. (P) Schuphaus	171	U.S. Report on Pea-Nut	819
Compounds, The Reduction of. (Wohl)	876	Oils. (Class XII.)	45, 162, 257, 404, 529, 647, 744, 819, 891, 950, 1068, 1207
Nitrogen and Carbon-Dioxide in Air, Minimum Proportion of. (Extinctive of Flames. (Clowes and Teilmann)	1155	Analysis of Lubricating. (Stillman)	286
Determination of, by Kieldahl's Method. (Krug)	425	And Fats, Bleaching, Sweetening and Purifying. (P) Aspmann, Hoar and Wise	1208
Determination of, in Nitrates, &c. (Krug)	1224	And Fats, Improving or Preserving. (P) Watel (illus.)	531
Estimation of Secondary Products containing. (Desva)	722	And Fats, Method and Apparatus for Deodorising. (P) (illus.) Ball	261
Isolde of. (Schubert)	61	And Fats, The Methods of Testing. (Milliau)	181
Nitro-Glycerine. See Explosives.		Apparatus for Distilling and Gasifying Hydrocarbon. (P) Caldwell and Tatham (illus.)	936
Nitrohydroxy and Nitro-amino-azo Compounds. (Tanber)	28	Apparatus for Obtaining Constant Pressure during Estimation of Freezing Point of. (Gantter) (illus.)	1090
Nitrometer, Estimation of Nitrate by means of the. (Henry)	764	Apparatus for Treating Substances containing. (P) Robbins	1070
Nitronaphthalene, Detection of, in Mineral Oils. (Leonard)	69	A Viscosimeter for Lubricating. (Kunkler) (illus.)	543
In Oils and Fats. (Holde)	906	Compounds of Blown, with Pyroxylin. (P) Fairfax. From Field	164
Nitrosamines and Diazo Compounds. (Schrambe and Schmidt)	508	Continuous Purification of. (Villon)	745
Nitrosodimethylamine, Action of Acid Chlorides on. (Ehrlich and Cohn)	30	Detection of Disguising and Perfuming Media in. (Holde)	906
Nitrous Acid and Nitric Acid, Action of on Silk. (Vignon and Lisey)	32	Detection of Nitronaphthalene in Mineral. (Leonard)	69
Oxide, Manufacture and Compression of. (Thilor (illus.)	470	Effect of Electrical Treatment in Improving Quality of. (Levat)	258
Oxide, Physical Properties of Pure. (Villmatt)	1087	Ekenberg's Process for Refining. (Benedikt)	591
North British Distillery Co., Visit to the	701	Fats, &c., freed from Odour or Flavour. (P) Abel. From Fabriques de Produits Chim. de Than et de Mulhouse	406
Norway, Land Changes in. (T.R.)	290	Filters used in the Production of. (P) Iseli	377
The Wood-Pulp Industry in. (T.R.)	671	Influence of Fatty, on Bactericidal Action of Phenol and Corrosive Sublimite. (Leust)	903
Notes, Analytical and Scientific	71, 181, 289, 327, 670, 767, 845, 903, 1106, 1239	Mixing, for use as Heating or Lighting Materials. (P) Sahlstrom and Parr	139
General Trade	71, 182, 291, 331, 537, 678, 770, 849, 910, 963, 1108, 1234	New Table, in Germany. (T.R.)	80
Laboratory. (De Benneville)	667	Occurrence of Camphene in Ethereal. (Bertram and Walbaum)	756
Notes, General	1, 193, 305, 347, 415, 572, 689, 783, 867, 925, 1041, 1131	On Ships in the Tropics, Mineral. (Lobry de Bruyn)	257
New Vanner Series, Estimation of Alkaloids in. (Koller)	1105	On the Flashing Point of Mineral. (Levinstein)	116
		Oxidation of Fatty. (Fahriou)	404
		Producing Consistent and Semi-consistent, without Heat. (P) Pommethanz	531
		Production of Gas from Paraffin. (Tocher) (illus.)	231
		Refining, Bleaching and Boiling. (P) Hartley and Blenkinsop	617
		Removing the Green Colour from. (P) Wendland	820
		Report on Castor. (Deering and Redwood)	950
		Solubility Tests of Dark Mineral Lubricating. (Holde)	698
		Spontaneous Combustion of, on Cotton. Part I. (Mackey)	1164
		Technical Analysis of Ethereal. (Klimont)	985
		The Aerial Oxidation of Essential. (Kingzett)	273
		The Clarification of. (P) (illus.)	1207
		The Constitution of Turkey-Red. (Juillard)	820
		The Oxygen Absorption and Acid and Iodine Numbers of Various Drying. (Lettenmayer)	258
		The Properties of Essential, and their Employment in Painting. (Lettenmayer)	273
		Treating and Purifying Soda and other. (P) Turney	16
Oak Tannins, Preparation of. (Trumble and Peacock)	59		
Obituary Notices:—			
Cornelberg, Dr. H. J.	786		
Jay, Alfred	133		
Wright, C. R. Alder	785		
Oils of Benzene Acids and other Bodies. (Parr)	670		
Oil of Glycer, Election of	691		



	PAGE		PAGE
Olefines and Carbon Monoxides, Distinction between.....	841	Packings, Improved Compound Asbestos, (P) Field.....	783
Olive Oil, Adulteration of, (Oliveri).....	17	Paint, An Improved, (P) Edwards, From Oxidation.....	167
-Oil, Detection of Castor-Oil in, (Di Vetre).....	981	A New or Improved, (P) Ward and Walbe.....	167
-Oil, Detection of Sesame Oil in, (Musset).....	423	A New Protective, (P) John.....	166
-Oil Industry in Spain, (T.R.).....	910	Hypocroscopic, for Packing Cases, (P) Abel, From 1 to 2.....	166
Opium Adulteration in China, (T.R.).....	770	Westfälisch-Anhaltischen Sprengstoff Actien-Gesellschaft.....	167
Ore containing Precious Metals, Treatment of Finely-Divided, (P) Raleigh.....	601	Manufacture of, (P) Hyatt.....	618
Manganese in Virginia, (T.R.).....	772	Manufacture of Preservative, (P) Grote.....	167
Reducing Sulphuretted, into Oxides and Sulphur, (P) Heilmann.....	153	Or Paint-like Composition, (P) Nangle.....	618
Roasting with a Blast, (Kloz).....	953		
Scandinavia as a Source of Irons, (T.R.).....	675	Paints, Pigments, Resins, &c., Class XII.....	16, 162, 262, 403, 522, 618, 746, 821, 893, 962, 1071, 1210
Ores, Calculation and Composition of Charges for Smelting Lead and Silver, (Watson).....	1110	Drying Medium for, (P) Bedford.....	814
Dissolving, Leaching, and Filtering, (P) Inray, From Lacy and Storer.....	741	For Preserving Ships' Bottoms, (P) Baker.....	648
Extracting Gold and Silver from, (P) MacArthur, Ellis, and The Cassel Gold Extracting Co.....	526	From Iron Protosulphate, Manufacture of, (P) D'Andrea.....	814
Furnace and Apparatus for Working, (P) Allison, From Storer, Martin, and Eaton.....	816	Heating Surfaces for Treating, by Means of Electricity, (P) Crompton and Dowling.....	168
Method and Apparatus for Extracting Gold and Silver from, (P) Irng.....	527	Manufacture of, (P) Wilson and Priest.....	1071
Method and Composition for Treating, (P) Kendall.....	890	Manufacture of Oil, (P) Anzell.....	823
Preparation of Pulverised, for the Blast Furnace, (P) Thomlinson.....	889	Manufacture of Red Oxide, (P) D'Andrea.....	262
Preparing Reagents for Working Gold and Silver, (P) Lake, From Noriega.....	41	Pans for Evaporating Brine, (P) Parks (illus.).....	171
Processes and Converters for Smelting and Refining, (P) Allen.....	1206	Papaveraceae, Alkaloids of, (Schmidt).....	168
Purchasing Silver, Gold, and Lead, (T.R.).....	559	Alkaloids of the, (König and Tietz).....	818
Separating Metals from, (P) Besenfelder.....	815	The Alkaloids of the Codeine, (Goehle).....	777
Testing Zinc, (Meyer).....	1225	Paper, Pasteboard, &c., Class XIX.....	170, 414, 538, 657, 754, 833, 906, 1087, 1216
The Chlorination of Gold, (Godshall).....	155, 157	Standing, (Class VI.).....	34, 119, 218, 386, 513, 633, 726, 803, 880, 955, 1057, 1113
Treating Auriferous, with Bromine, (Lossen).....	1198	A Finely Granulated, and Production of same, (P) Thompson, From Schaffellen.....	513
Treating Complex and Refractory, and Obtaining Metals and Pigments therefrom, (Hall).....	741	And Boards Imitating Natural Woods, (P) Cornell.....	1216
Treating Refractory, (P) Ashcroft.....	957	And Book Industries International Exhibition at Paris, (T.R.).....	111
Treatment of, and Recovery of Metals therefrom, (P) Fuller.....	44	A New Photographic Printing, (Artigues).....	770
Treatment of Lead, (P) Fell.....	880	And Cardboard, Process for Colouring, (P) Wells.....	391
Treatment of Metallic, (P) Richardson and English.....	401	Apparatus for Testing, (P) Woolley (illus.).....	1222
Treatment of Zinc-Lead-Sulphide, (P) Fell, From The Emmeus Zinc Co.....	402	Brown, from Wood.....	119
Orris-root, Manufacture of a New Chemical Substance from, (P) Tiemann.....	542	Causes of Difficulty of Sizing, in Hot Season, (Muth).....	167
Orthohalogen-phenol, Manufacture of, and Production of Pyrocatechine therefrom, (P) Baum.....	658	Coating, with Aluminium, (P) Abbott.....	171
Orthotolidine, Some Reactions of, (Schiff and Ostrogovich).....	797	Decorative, (P) Vander Hoop.....	1071
Otto of Roses, (Bertram and Gildermeister).....	834	Dyeing in Pulp, (Falke).....	789
Oven, A Disinfecting, (P) Baumbach.....	413	For Bank Notes, &c., Manufacture of, (P) Buttner and Will.....	514
An Aluminium Dryer, (Meyer) (illus.).....	1220	-Making in India, (T.R.).....	1216
For Drying Hydrogen, (Morse) (illus.).....	174	-Making Materials, Bolders for Treating, (P) Sinclair.....	756
Ovens, Comparative Working of Coke, (T.R.).....	1238	Manufacture of a Substitute for Ordinary, (P) Kumazawa.....	754
For Ceramic Ware, (P) Hanquiaux.....	1018	Manufacture or Treatment of, (P) Lusby.....	932
For Drying and Coking Peat Briquettes, (P) Stauber (illus.).....	715	Manufacture or Treatment of, (P) Yeckley.....	762
See also Furnaces and Kilns.....		Marking Indelibly on, (P) Cross and Bevan, From Beadle.....	1114
Oxalic Acid, Action of Light on, (Richardson).....	516	Method and Apparatus for Producing an Improved, (P) Wheeler.....	514
Oxazine and Euxhodine Series, On some Derivatives of the, (Lauth).....	628	Mills of Cowan and Sons and Brown and Co., Visit to.....	760
Oxidation, Researches on the Phenomena of, (Phillips).....	810	Nitrocellulose in Filters, (Chamer).....	815
Oxide of Iron, Process and Apparatus for Manufacture of, (P) Hemingway.....	163	Par. lignit (illus.).....	414
Of Zinc Pigments, Preparation of, (P) Laurie.....	262	Preparing Pigment for Photographic Printing, (P) White.....	177
Paints, Manufacture of Red, (P) D'Andrea.....	262	Presence of Residual Chemicals in, (Irvine and Woodhead).....	161
Oxides and Compounds, Manufacture of Metallic, (P) de Pass, From Thofehn.....	158	Printing Various Designs on, (P) Vogel, junr.....	391
Oxindophenolic Colouring Matters from Gallanilide and Galloparatoluide, (Cazeneuve).....	720	Pulp, Apparatus for Manufacture of, (P) Caldwell.....	1085
Oxyaldehydes Compounds of Aromatic and p-Amidophenol Ethers, (P) Inray, From the Society of Chemical Industry of Basle.....	271	Pulp, Bleaching, (P) Abom.....	884
Oxy-Anthraquinone, Production of, (P) Inray, From the Farb. vorm. Meister, Lucius and Brünig.....	878	Rendering, Sensitive to Light, (P) Junk.....	173
Oxycelluloses, Natural, I. Celluloses of the Gramineae (Smith).....	337	Sanitary, (P) Quilliam.....	869
Oxygen, Action of, on the Platinum Metals, (Dudley).....	253	Sensitised, for Photographic Purposes, (P) Heschel.....	171
Action of, on Yeast, (van Laer).....	1078	Spots in, (P) Berland.....	758
And the Halogens, Electrolytic Preparation of, (P) Cechu From Calcium Plumbate, Manufacture of, (Kassner).....	415	-Stuff, Apparatus for Mixing and Regulating, (P) Berland -Testing Department of Berlin Experimental Institutes, Expert Opinions from the, (T.R.).....	779
Manufacture of, (Le Châtelier).....	27	Treatment of, for Receiving Printed Impressions, (P) Berger, Waldbaur, and Worlitzer.....	814
Production of, from Air, and Apparatus therefor, (P) Brin's Oxygen Co., and Murray.....	836	Papers, Examination of Glee for Manufacture of Coloured, (Schlossmann).....	424
Oxy-hydrogen Light, Saturators and Jets for Producing, (P) Suiter.....	874	Made from Sulphite Cellulose, Discoloration of, (Muth).....	169
Oxy-Oil Gas, and its Use for Enrichment Purposes, (Thorne).....	138	Para-Amidophenol Ethers and Aromatic Oxyaldehydes, Manufacture of Compounds of, (P) Inray, From the Soc. of Chem. Industry, Basle.....	276
Ozone and Luminosity, Production of, Electrically, (P) Andreoli.....	162	-chloralose and Para-chlorallic Acid, (Hanriot and Richer).....	775
Formation of, at High Temperatures, (Brunck).....	170	-ethoxy- and Para-methoxy-phenyl succinimides, Production of, (P) Inray, From the Farb. vorm. Meister, Lucius und Brünig.....	810
From Phosphorus, Production of, (P) Kattenhoy.....	411	Paraffin and "Lubricating Oil," Detection of, in Distillate from Tram Oil, (P) Engler and Singer.....	5
		And Mineral Oil Industries, Improvements in the, (Mann).....	875
		Oils, Discussion on Production of Oil-Gas from.....	211
		Oils, Production of Gas from, (Tischer) (illus.).....	211
		Wax, United States Exports of, (T.R.).....	201
		Paraffins, The Estimation of, (Holland).....	196
		Parahydroxyphenylurethanes, Production of Propionyl Compounds of, (P) von Mering.....	40
		Paranitrophenol as an Acid Indicator in Testine Feed-Water, (Goldberg).....	419
		Paranitrotoluene, Action of Alkalis on, (Fischer and Hepp).....	29

	PAGE		PAGE
Para-phenetolecarbazole, Obtainment of. (P) Wetter. From Rochelle. ....	542	Petroleum—cont.	
Pararosaniline and its salts. (Molitor) .....	143	Transport of, in Bulk. (Redwood) (illus.) .....	1182
Parasitic Methyl for Dyeing. (P) Vigerio. ....	168	Vaporisation of. (P) Boulé. From Penchen and Clarke. .	380
Parasorbic Acid, and its Isomerism with Sorbic Acid. (P) Schner .....	539	Pharmaceutical Chemistry, Recent Developments in. (Erdmann) .....	549
Paratoluidine, The Oxidation of. (Green) .....	113	Preparations, Manufacture of. (P) Webster and Moore. .	161
Paraldehyde, Manufacture of. (P) Brand. ....	1085	Phenanthrene Series, New Dyes of the. (P) Wilcox. From The Badische Anilin and Soda Fab. ....	912
Paper (illus.). ....	114	Phenyl Action of Light on. (Bach) .....	938
Paper, International Book and Paper Industries Exhibition. (T.R.) .....	240	Compounds of Mercuric Phenolates. (Desesquelles) .....	835
Paste, A Cleansing. (P) Baerlein and Proxvus. ....	939	Etters, Manufacture of. (P) Kraft and Roos .....	759
Or Size for Cop Bottoms and Cotton Goods. (P) Brown. .	725	Influence of Alcohol, Ac., on Bactericidal Action of. (Leasi) .....	905
Paste-board. Class XIX. 1176, 114, 538, 657, 754, 833, 900, 1085	1216	Phthalin in Margarin rendered Conspicuous by Admixture of. (Hart) .....	412
Pasteurising Apparatus. (P) Pausch. ....	1215	Phenols, Compounds of the Sugars with Polyhydric. (Fischer and Jennings) .....	765
Patent Law in Austria-Hungary, New. (T.R.) .....	74	Obtaining Aqueous Solutions of. (P) Hilmers. ....	900
Law of the United States, Proposed Changes in. (T.R.) ..	183	The Ethyl. (Behal and Choay) .....	794
Office Rules, Changes in the United States. (T.R.) .....	433	Phenomena accompanying Dialysis of Beer Yeast Cells. (Omnus) .....	1213
Patents, Monthly Lists of. 187, 205, 438, 594, 681, 776, 859, 916, 1004, 1121, 1210	81	Phenylmethylglypazalone $\beta$ -resorcylate. (Petit and Fèvre) .	902
In Russia, the Granting of. (T.R.) .....	736	Phenylphosphor, Comparison between Coloured and Colourless Derivatives of Di- and Tri-. (Rosenstiehl) .....	798
Pavements, Modern Street. (Gunn) .....	736	Phenylproindoline, Manufacture of Sulpho Acids, Analogous, and Homologues of. (P) The Badische Anilin and Soda Fab. ....	510
Paving Blocks, Cement, Ac., Composition of Materials for. (P) Edwards. From Overton .....	737	Phoma Betæ: the Beetroot Destroying Organism. (Hollrung) ..	264
Manufacture of a Composition suitable for. (P) Sykes .....	396	Betæ: the Beetroot Destroying Organism. (Frank) .....	822
Peanuts and Pea-Nut Oil, U.S.A. Report on. ....	539	Phosgene, Examination of Chloroform for. ....	1099
Manufacture of Oil and Food from. ....	897	Phosphate and Asbestos Mining in Canada. (T.R.) .....	674
Peas, Indian. ....	602, 605	Deposits of Algiers. (T.R.) .....	1108
Peat Analyses of. (Dvorkovitz) .....	243	Manufacture of Monocyclic. (P) Hurter and Omholt. ....	394
Apparatus for Drying. (P) Brimton. ....	797	Preparation of Bi-Calci. (P) Chan telon .....	748
Peas of India, Extent of. (Dvorkovitz) .....	715	Production of Monocyclic, and Ammonia. (P) Hurter and Omholt .....	409
Braquettes, Ovens for Dyeing and Cooking. (P) Stauber (illus.) .....	715	Phosphates, Agricultural Value of certain Natural. (Paturel) ..	1210
Pest, Action of, on the Bacteria of Cholera and Typhoid. (Frankel and Kipstein) .....	538	An Automatic Extractor for. (Horne) .....	278
Preparing, for Production of Heating Gas, Ac. (P) Dillon	242	Experiments on Fertilising Effect of Various. (Kellner, Kozai, Mori, and Nagaoka) .....	409
The Distillation of. (Dvorkovitz) (illus.) .....	56	In South of France, New Deposit of. (T.R.) .....	910
Pelargonium, The Rhodinol of the Essence of. (Barbier and Bonvault) .....	1085	Phosphoric Manures, Comparative Value of Various. (Von Dohn) .....	263
Pelts, Removing stiff Water or Waste Hairs from. (P) Werner .....	1209	Phosphor Copper and Bronze. (McCombie) .....	
Pemberton's Method of Phosphoric Acid Determination. On. (Day and Bryant) .....	667	Phosphoric Acid contained in Bone Meals, Solubility of. (Gehele) .....	894
Pentosans in Vegetable Substances, Estimating. (Hötter) ..	324	Acid, Determination of. (Pemberton) .....	176, 667
Pepper, Treatment of. (P) Kirchberger. ....	537	Acid, Determination of. (Williams, Bergami, and Terme) ..	842
Percarbonate in Potassium Nitrate, Occurrence of. (Haußermann) .....	947	Acid, Determination, On Pemberton's Method of. (Day and Bryant) .....	667
Perochromic Acid, Sodium Salt of. (Haußermann) .....	259	Acid, Estimation of, by the Citrate Method. (Glaser) .....	1225
Peroxy Alkaloids. (Hesse) .....	416	Acid in the Blast-Furnace, Behaviour of. (Kjellberg) .....	524
Perfumery, Modern. (Vonger) .....	1477	Acid, Reliability of Magnesium Pyrophosphate for Estimating. (Neubauer) .....	1062
Production of Substances for Manufacture of. (P) Thiemann .....	903	Acid, Volumetric Estimation of. (Holleman) .....	843
Perfumes, German Trade in. (T.R.) .....	1111	Acid, Volumetric Estimation of Soluble, in Superphosphates. (Kalman and Meissels) .....	812, 905
Pernanganate of Soda or Potash, Manufacture of. (P) Brock and Harter. ....	394	Acid, Volumetric Method of Estimating. (Holleman) .....	763
Pernanganates, Production of, from Manganates. (P) Menzies	38	Phosphorus, Apparatus for Production of Ozone from. (P) Kattenhoy .....	411
Peroxides, Action of Ammonia Gas on. (Michel and Grandmouzin) .....	37	Determination of, by the Molybdenum Method. (Myhlertz) ..	67
Per-Salts of the Heavy Metals, Estimation of Free Acid in Solutions of. (Hollmann) .....	282	Estimation of, in Steel containing Silicon, and in Pig-iron. (Spuller and Kalman) .....	175
Persa, Petroleum in. (Stahl) .....	243	Formation of, from the Phosphates of Alkalis. (Rossel and Frank) .....	515
The Copper Ores of. (T.R.) .....	435	Gazette Trade in. (T.R.) .....	1110
Persian Coal. (Stahl) .....	242	In Coal and Coke, Determination of. (Jynchenheim) .....	422
Perth, Visit to. ....	705	Influence of, on the Composition of Pig-iron. (Kjellberg) ..	524
Petroleum, a Crucible Finance for Burning. (Crane) (illus.) .	21	In Steel, Determination of. (Doodittle and Eavenson) .....	666
American and Russian. (Becker) .....	872	In Steel, Determination of. (Dudley and Pease) .....	665
Apparatus for Lighting and Heating by Means of. (P) De Pass. From Leumann .....	191	In Steel, Determination of. (Handy) .....	666
Crocks, occurring in Trenton Limestone. (Muhlhauser) ..	105	Matches, The Inventor of. (Jensch) .....	837
Discoveries in the Caucasus. (T.R.) .....	779	New Method of Estimating Yellow. (Toth) .....	420
Distillation of. (P) Wanklyn and Cooper. (illus.) .....	720	Preparation of. (Rossel) .....	845
Examination of Waste Liquors from Refining of. (Zaloziecki) ..	628	Photographic Materials and Processes. (Class XXI.) 173, 276, 417, 543, 659, 759, 973, 1088, 1219	
Industry and Output of Russia, in 1893. (Thiess) .....	1275	Developers, Manufacture of. (P) Abel. From the Actien Gesellschaft fur Anilin Fab. ....	659
Industry in Baku, 1893. ....	875	Development, Method and Apparatus for Timing. (P) Watkins .....	1219
Industry, Present State of the. (De Bousien) .....	794	Exhibition at the Imperial Institute. (T.R.) .....	1115
In Persia. Stahl. ....	243	Fibres, Manufacture of. (P) Blair .....	659
In Sumatra. ....	76	Pictures, Development of. (P) Andersen .....	1219
Preliminary Examination of Canadian Sulphur. (Mabery) ..	707	Plates and Films, Manufacture of. (P) Pettit .....	659
Process for Purifying. (P) Schrader. ....	1054	Plates and Prints, Apparatus for Washing. (P) Thomson ..	659
Production and Consumption of, in India. (T.R.) .....	1116, 1117	Plates, Manufacture of. (P) Magerstedt .....	276
Removal of Boiler Scale by Means of. ....	13	Printing in Bromide of Silver. (P) Norden .....	276
Report of Select Committee on. (T.R.) .....	999	Printing Paper, A New. (Artigues) .....	759
See also under Asphaltum, Naphthalin, Mineral Oil, Paraffin, and Kerosene. ....		Plates, Production of. (P) Packham .....	1219
Sludge, Spontaneous Ignition of Dried. (Kissling) .....	874	Photographing in Colours. (P) Jock .....	1088
Spirit Electrical: excitation of. (Kissling) .....	385	Photographs, Colouring, and Colours for. (P) Beacham .....	759
Spirit Fires in Chemical Cleaning, Prevention of. (Gohring)	385	On Glass, Making Transparent. (P) Allison. From Forster and Rockwood .....	417
Sulphur Compounds in Ohio. (Mabery and Smith) .....	507	On State, Preservation of. (P) Jones .....	659
Supply of the Caucasus. (T.R.) .....	1234	Physical Influence of Elements on Iron. (Arnold) .....	642
Tar, Products of Distillation of. (Tisitschenko) .....	1053	Piccin. (Farré) .....	1217
Terpenes in. (Zaloziecki) .....	117	Picene. (Bamberger and Chattaway) .....	27
The Origin of. (Phillips) .....	799		
The Solidification of. (T.R.) .....	9		



	PAGE
Pernicoline, On. (Danstan and Harrison) .....	172
Peric Acid, Combinations of Hydrocarbons with. (Tilden and Forster) .....	172
Pne-copper, Experiments on Analysis of. (De Benneville) .....	657
Pig Iron, Estimation of Graphite in. (Crobaugh) .....	665
Pigment for Coating Iron and Steel. (P) Elliott .....	962
Manufacture of Sulphate of Lead. (P) Macdonald .....	893
Paper for Photographic Printing, Preparing. (P) White .....	117
Production of Iron Oxide for Use as a. (P) Lyle .....	962
Treatment of Lead Sulphate for Use as a. (P) Wilson and Landy .....	746
Pigments. (Class XIII.) 46, 162, 262, 106, 532, 618, 713, 821, 895, 962, 1071, 1269	
And Compounds, Manufacture of Metallic. (P) Coarlier .....	732
A New Class of Lake. (Weber) .....	1151
For Dyeing, Painting, or Drawing. (P) Niemann, Goldmark, and Hermann .....	618
Manufacture of. (P) Imray. From Sachs, Meier, and Gerstendörfer .....	532
Manufacture of Lead. (P) Gregory .....	618
Manufacture of Pulverised Materials for. (P) Pattee .....	1018
Obtaining, from Complex and Refractory Ores. (P) Hall .....	711
Preparation of Oxide of Zinc. (P) Laurie .....	262
Production of Metallic, and Apparatus therefor. (P) Charlier .....	407
Pine Tar. (Renard) .....	1188
Tar, Distinction of, from Birch-Tar. (Hirschsolm) .....	623
Pinus Maritima, Tar of. (Renard) .....	1188
Pipe Columns for Sulphuric Acid Chambers. (Gilchrist (illus.) .....	1142
Pipe-Joints, Improved Earthenware and other. (P) Ewing .....	788
Piperazine and its Salts, Manufacture of. (P) Newton. From The Farb. vorm. Bayer and Co. .....	275
Pipes, Manufacture of Stone-ware Sanitary. (P) Tushy .....	735
Method of Lining, with Glass, Enamel, &c. (P) Herbert .....	154
Or Tubes for Heating, Cooling, and Evaporating Apparatus. (P) Thompson. From Dumas .....	715
Porous Material suitable for Tobacco. (P) Smith .....	395
Pisum Sativum, Occurrence of Trigonellum in Seeds of. (Schulze and Frankfurt) .....	757
Pitch, German Trade in. (T.R.) .....	1113
Production of Artificial. (P) Haddan. From Anderson, Shutter, Kochler, and Dower .....	720
Stearin. (Donath and Strasser) .....	359
Pitches, Distinction between Coal-Tar and Blast-Furnace. (Buchanan) .....	1098
Place of Meeting for 1895, Decision on. .....	700
Plant (General), Apparatus, and Machinery. (Class I.) 21, 131, 239, 376, 502, 622, 713, 787, 869, 929, 1017, 1179	
Plants, Poisonous Fodder. (Jackson) .....	897
Plastering for Ceilings and Partitions. (P) Jung .....	1063
Plaster of Paris from Lime Residue. (P) Kerr .....	888
Plate-Towers for Condensing HCl, Results of Lunge-Rohrmann. (Lunge) .....	1064
Plates, Darrius' Theory of the Life of Lead Secondary Accumulator. (Schoop) .....	1203
For Batteries. See under Batteries.	
For Condensing Acids, &c. (P) Mackenzie. From Brante and Co. .....	930
For Oil and other Presses. (P) Leechman .....	377
Platinum, Constitution of the Rock Matrix of. (Meunier) .....	639
In Iron Solutions, Effect of. (Mahon) .....	516
Metals, Action of Gaseous Hydrochloric Acid and Oxygen on the. (Dudley) .....	255
Output of the Ural Mines. (T.R.) .....	435
The Ural Deposits of. (T.R.) .....	995
Plugging Compound for Blasting Cartridges. (P) Abel. From the Westfälische Anhaltischen Sprengstoff Actien Gesellschaft .....	61
Plumbago, Manufacture of. (P) Johnson .....	821
Poisons, Bottles for Containing. (P) Reed .....	788
Bottles for Holding. (P) Quine .....	713
Polish for Furniture. (P) Horn .....	896
Polishing Medium. (P) Theil and Schmitz .....	497
Polypheols, some Blue Reactions of the. (Cazeneuve) .....	1098
Pomegranate Root, Alkaloids of the Bark of. (Ciancian and Silber) .....	416, 1217
Ponceau-Red Dye, a Japanese. (Scheurer) .....	598
Populus Tremula, The Tar of the Bark of. (Pharmakofsky) .....	794
Porcelain Industry at Limoges. (T.R.) .....	558
Process for Etching on. (P) Retzlaff .....	151
Producing Enamelled Letters and Designs on. (P) Hodan .....	884
Transparent Cement for. (Deutsch) .....	888
Portugal, the Preserved Fruit Industry of. (T.R.) .....	895
Potash and Soda, Densities of Solutions of. (Pickering) .....	514
Apparatus for Making Caustic. (P) Petrie .....	38
Deposits of Germany, Proposed Nationalisation of. (T.R.) .....	558
German Trade in Chromate of. (T.R.) .....	1110
German Trade in Prussiate of. (T.R.) .....	1111
Influence of Substances on Estimation of. (Dods) .....	1095
Manufacture of Caustic. (P) Garway .....	735
Manufacture of Peroanganate of. (P) Brock and Hunter .....	394
Potash—cont.	
Separation of Salts of, from Solutions of Magnesium Chloride. (P) Bell .....	9
Yellow Prussiate of, and Detection of Hydrocyanic Acid (Autenrieth) .....	814
Potashes, On the Valuation of. (Bendheim) .....	705
Potassium and Lead, Electrolytic Production of an Alloy of. (P) Vautin .....	714
And Sodium, Utilisation of Chlorides of. (P) Parker and Robinson .....	250
Arsenic, Reactions of Acid, with Metallic Salts. (Rochard) .....	732
Bisulphite, Action of, on Alcoholic Fermentation. (Bazzani) .....	266
Borohydroxide, Volumetric Determination of. (Schlotheim) .....	280
Carbide, Production of. (Mullhauser) .....	73
Chloride, Apparatus for Manufacture of, by Electrolysis. (P) Harter .....	810
Chlorate, Electrolytic production of. (Haussmann and Naschold) .....	885
Chloride, Apparatus for Electrolysis of. (P) Drake .....	887
Chloride, German Trade in. (T.R.) .....	1111
Cyanide, Exempt from Duty in China. (T.R.) .....	74
Cyanide, Tests on Extraction of Gold by Infuse. (Gwyder) .....	523
Determination of Reducing Action of Organic Substances in Water upon Permanganate of. (Zegen) .....	427
Iodate, Preparation of Pure. (Grosjean) .....	419
Nitrate, Occurrence of Perchlorate in. (Haussmann) .....	917
Nitrate, The Analysis of. (Hedrich) .....	979
Phosphate as Manure. (Meyer) .....	401
Salts, Influence of, on Nitrication. (Dumont and Chetelle) .....	672
Sulphate, German Trade in. (T.R.) .....	1111
Tanks for Electrical Decomposition of Chloride of, in Solution. (P) Richardson .....	616
Potato Pulp, Drying of, for Cattle Food, in Germany. (Delbrück) .....	965
Starch, Manufacture of, by Continual Flow System. (Bosdonnan) .....	759
Pottery. (Class XIII.) 39, 153, 251, 395, 518, 637, 735, 811, 888, 918, 1069, 1197	
Apparatus for Manufacture of. (P) Slack .....	1062
Decorating. (P) Shelley .....	949
Industries of the United States, Chemical Considerations on the. (Langenbeck) .....	153
Manufacture of. (P) Moran .....	1197
Manufacture of. (P) Worcester Royal Porcelain Co. and others .....	1197
Ovens for. (P) Hanquinn .....	1018
Ware, Means for Ornamenting. (P) Daulton and Rix .....	395
Powder, Improved Baking. (P) Brothers .....	752
Smokeless. (P) Brothers .....	1088
The Manufacture of Smokeless. (Guttmann (illus.) .....	575
See also Explosives.	
Powderhall Refuse Destructor, Visit to the .....	765
Powders, Manufacture of Coated Metallic. (P) Imray. From Sachs .....	958
Powers of the Local Sections, Discussion on .....	693
Precipitates formed during the Heating of Beet Juice. (Hertzfeld) .....	719
Obtained in Sugar Juice Evaporating Apparatus. (Shorey) .....	759
Presentation to Dr. J. T. Dunn .....	1154
To Mr. J. Carter Bell .....	697
Presidential Address, The. (Stanford) .....	614
Press, Hydraulic Oil. (P) Greenwood, Batley and Lambert .....	495
Presses, Filter. (P) Critchlow .....	929
Filter. (P) Seitz .....	714
For Oil, &c. (P) Johnson. From Gonnelle .....	1048
Plates for. (P) Leechman .....	377
Pressure in Gas Cylinders, Utilisation of, in Employing the Compressed Gases. (P) Manchester Oxygen Co. and Jackson .....	715, 1180
Prices of General Commodities in England, Course of Average. (Sauerbeck) (illus.) .....	763
Printing and Bleaching, Improvements in .....	386
Processes of the Japanese. (Tetaz) .....	1194
Prize for Substitute for Gutta-Serena .....	73
Prizes for Chemical Research. (T.R.) .....	1091
Offered by City and Guilds of London Institute .....	1212
Offered by the Soc. Industrielle de Mulhouse .....	73
Problem in Electro-Chemical Economy, A .....	817
Proceedings of Chambers of Commerce. (T.R.) .....	1233
Of the Thirtieth Annual Meeting .....	691
Producer-Gas. See under Gas.	
Products of Combustion of Gas by Alzand and by Auer Burners. (Graham) .....	1215
Propionyl Compounds of Parahydroxyphenylurethanes, Production of. (P) von Marburg .....	9
Proteids of Cotton-Seed. (Osborne and Voorhees) .....	828
Of the Kidney-Bean. (Osborne) .....	828
Protopine .....	826
Pseudo-Speise, A Japanese: Its Composition and Relations. (Gowland (illus.) .....	693
Pulp. See also Paper.	
Bleaching Paper. (P) Abma .....	824
Dyeing Paper in. (Falkel) .....	880
Machinery for Straining. (P) Kunkel .....	713

	PAGE		PAGE
So. Food, Preparation of Malted, (P. Wats.)	270	Report, Testing Milks with, (Lezé and Hulsont)	1009
Stone, Manufacture of a Substitute for, (P. Salmon and Whiting)	735	Report (Eighteenth Annual) of H.M. Inspectors of Explosives for 1893, (T.R.)	837
New Form of Mercury Vapour, (Kahlbaum, illus.)	769	Of Commission on Nitrates in Egypt, (T.R.)	1236
Pumps for Pumps, 2, Compressing Gases or Vapours, (P. Murray, illus.)	377	Of Committee of Inquiry on Lucifer Match Works, (T.R.)	75
For Raising Acids, (P. Long and Burkitt, illus.)	380	Of Committee on Action of Light upon Dyed Colours, (Hummel)	803
Soil, Indigo Cultivation in the, (T.R.)	232	Of Committee on Chemical Works, (T.R.)	76
Compression and Treatment of Solids for Use as, (P. Walters)	407	Of Committee on China and Earthenware Manufacture (T.R.)	75
Production of Basic Compounds from, (P. Newton, From The Farb. vorm. Bayer and Co.)	510	Of Council	691
Pyrazole Reactions, (Von Rottenburg)	705	Of French Committee on Natural Mineral Waters, (T.R.)	850
Pyridine, Determination of Nitrogen in, (Kaiser)	425	Of Polish Committee on Diffusion Process	748
Pyros, Analysis of, (Blaser)	1065	Of Select Committee on Petroleum, (T.R.)	901
Separation of Blende from, (Blake)	521	Of the Comptroller-General of Patents for the Year 1893, (T.R.)	674
Treatment of Cuprous Iron, at P. (Kaiser)	472	On Alkali Works, Thirtieth Annual	729
Pyrocholine, Production of, (P. Baum)	658	On Bonnet's Dyeing Process, (Kopp, Noelling, and Grandmoulin)	726
Pyrocholine, Action of Sulphuric Acid on, (Günther)	60	On Castor Oils, (Deering and Redwood)	959
Pyrometer, A, Action of, (P. Lantache, illus.)	693	On the India-Rubber Industry in 1892, (T.R.)	81
Pyrometer, Pneumatic, (M. G. G. illus.)	809	Reports, Diplomatic and Consular, 71, 290, 430, 556, 769, 992, 1234	
Pyrometers, Improvements in, (P. Loh and Steinbart, illus.)	175	On Modified Starches Prepared by Siemens and Halske	261
Pyroxylin, Compounds of Blow, Oils with, (P. Fairfax, From Field)	164	Resalzin, On, (Petit and Fevre)	902
Compounds, Solutions and Solvents, (P. Fazel)	262	Research Committee of the Imperial Institute, (T.R.)	293
Pyroxylin, Spontaneous Combustion of, (P. Fazel)	773	Prizes for Chemical, (T.R.)	993
		Reactive of Wood Pulp Manufacture, Treating the Black-Ash, (P. Langvick)	170
		Residues, See also under Refuses and Wastes.	
		Resin, Gardenia, (Heckel and Schlagdenhauffen)	47
		Resins, (Class XIII.), 46, 162, 282, 406, 532, 648, 749, 821, 893, 962, 1071, 1209	
		And Fats, New Method of Analysing, (McIlhenny)	668
		Explosives from Nitrate of Ammonia and, (P. Abel, From König)	543
		On Xanthorrhoea, (Bamberger)	47
		Resist and Discharge Styles with Aniline Black, (Dreyfus)	485
		And Discharges on Silk, (Koechlin)	727
		Employment of Ammoniacal Salts as, (Bontemps)	1193
		On Steam-printed Indigo, Azo-Colour, (Bloch and Schwartz)	1193
		Or Discharges for Use in Printing Fabrics with Aniline Black, (P. Whitehead)	390
		With Tannin Mordants, (Cassanovas)	882
		Resorcinol Green, Formation of, on Cotton, Wool, and Silk, (P. Korny)	727
		Retort and Apparatus for Obtaining Gas from Coal-Tar, (P. Smith)	626
		For Treating Town Refuse, (P. Hanson)	899
		For Use in Kjeldahl's Process, (P. Edwards)	760
		Process for Avoiding Choking of Mouth of, in Manufacture of "Thomas" Steel, (P. Raze)	158
		Retorts for Lining, (P. Bowing)	873
		For Distilling Shale, &c., (P. Gray)	1054
		Retting of Fibres, (P. Pennington and Allison)	725
		Revenue and Expenditure for 1893, Statement of, (T.R.)	374
		Cessions on Export of Tinctures and Essences, (T.R.)	772
		Rewards for Discoveries Offered by the Franklin Institute	73
		Rhamnose, Fermentation of, by Lactic Acid Ferment, (Tate)	268
		Influence of Acid Molybdate of Soda and Ammonia on Rotatory Power of, (Gernez)	1226
		Rhamnosides, Dyeing with, (Schunck and Marchlewski)	633
		Rhen, Extraction and Preparation of the Fibres of, (P. Fellows, Crozier, and Ferguson)	632
		Rhodinol, A New Source of, (Monnet and Barbier)	658
		Characteristics of, (Barbier)	171
		Of the Essence of Pelargonium, (Barbier and Boureault)	1085
		Rice Beer, Production of, (Calmette)	268
		Treatment of, to obtain Material for Brewing, (P. Lannes)	268
		River Tay, Visit to the	705
		Rock Matrix of Platinum, Constitution of the, (Mennier)	639
		Roscher-Eichmann Process, The, (Knoertzen)	757
		Roofing, Manufacture of Flax or other Vegetable Fibre, (P. McTeer)	737
		Rose, Bulgarian Essence of, (Markownikoff and Reformatsky)	272, 758
		Oil Otto of, (Bertram and Gildemeister)	834
		Rosin, the Iodine Number of, (McIlhenny)	668
		Rosin and Roslyn Glen, Visit to	700
		Rotation, Influence of Solvents on, (Schuster)	72
		Rowan Berries, Oil of, and its Isomerism with Sorbie Acid, (Diedrich)	559
		Rubber, Apparatus for Separating Foreign Substances from Waste, (P. Anderson, jun.)	871
		See also under India-Rubber.	
		Rubia Sikkimensis, The Colouring Principles of, (Perkin and Hummel)	244
		Sikkimensis, Tinctorial Properties of, (Hummel and Perkin)	348
		Rule for Calculating Japanese ad valorem Duties, (T.R.)	991

## Q

Quarantine, Sanitary, (Shi and Reifsoyden)	277
Quarantine, The Inertness of, II. The Interaction of Chlorine and Iodine, (Veley)	250
Quarantine, The Inertness of, (Veley)	903
Quarantine, The Inertness of, (Veley)	1112
Quarantine, The Inertness of, (Veley)	1083
Quarantine, The Inertness of, (Veley)	171
Quarantine, The Inertness of, (Veley)	425
Quarantine, The Inertness of, (Veley)	798
Quarantine, The Inertness of, (Veley)	148

## R

Radiation, The Measurement of, by the Radiometer	428
Radiation, The Measurement of, by the Radiometer	653
Radiation, The Measurement of, by the Radiometer	945
Radiation, The Measurement of, by the Radiometer	57
Radiation, The Measurement of, by the Radiometer	284
Radiation, The Measurement of, by the Radiometer	257
Radiation, The Measurement of, by the Radiometer	1222
Radiation, The Measurement of, by the Radiometer	110
Radiation, The Measurement of, by the Radiometer	177
Radiation, The Measurement of, by the Radiometer	41
Radiation, The Measurement of, by the Radiometer	970
Radiation, The Measurement of, by the Radiometer	701
Radiation, The Measurement of, by the Radiometer	158
Radiation, The Measurement of, by the Radiometer	638
Radiation, The Measurement of, by the Radiometer	787
Radiation, The Measurement of, by the Radiometer	269
Radiation, The Measurement of, by the Radiometer	271
Radiation, The Measurement of, by the Radiometer	832
Radiation, The Measurement of, by the Radiometer	829
Radiation, The Measurement of, by the Radiometer	412
Radiation, The Measurement of, by the Radiometer	1233
Radiation, The Measurement of, by the Radiometer	138
Radiation, The Measurement of, by the Radiometer	427
Radiation, The Measurement of, by the Radiometer	1089
Radiation, The Measurement of, by the Radiometer	72

Rules for the Preparation of Standard Sperm Candles.....	65
Special, for Chemical Works, under the Factory and Work- shops Acts. (T.R.).....	77
Russian and Germany, New Commercial Treaty between. (T.R.).....	280, 551
Customs Decisions in. (T.R.).....	909, 980, 1232
Granting Patents in. (T.R.).....	81
Mineral Industries of South. (T.R.).....	819
Petroleum Industry and Output in 1883. (T.R.) Thues... ..	1235
Production of Alcohol in. (T.R.).....	200
Sugar Production of. (T.R.).....	201
The Platinum Deposits of. (T.R.).....	395
<b>S</b>	
Saccharimeter Degrees on Ventzke Scale, Ratio of, to Angular Degrees for Sodium Light. (Rindbach).....	1222
Saccharin, Investigation of. (Hefelmann).....	835
Manufacture of Anhydro-ortho-sulphamino Benzoic Acid or its Salts from Commercial. (P) Ling.....	836
Saccharine, Estimation of, in Presence of Salicylic Acid. (Hairs).....	124
Juices and Solutions, Purifying and Decolorising. (P) Lach and Benies.....	824
Saccharometers, Improved. (P) Kostine.....	810
Saccharose, Dissemination of, in the Seeds of Plants. (Schulze and Frankfurt).....	533
Estimation of.....	1101
Production of, during Germination of Barley. (Lindet) ..	265
Saffron and its Adulterants. (Vinassa).....	71
Safranines and Indulines, Relation between the. Fischer and Hepp).....	30
"Sal Acetol," Quantitative Estimation of Acetone and Salicylic Acid in. (Eckemroth and Kock).....	288
Acetol, Quantitative Estimation of Acetone and Salicylic Acid in. (Helling and Passmore).....	288
Salicylate of Bismuth, Basic. (Schubardt).....	57
Salicylates, Mercurial. (Lajoux and Grandval).....	58
New Method for Estimating. (Barthe).....	1105
Salicylic Acid and Acetone in "Sal Acetol," Quantitative Esti- mation of. (Eckemroth and Kock).....	288
Acid and Acetone in "Sal Acetol," Quantitative Estimation of. (Helling and Passmore).....	288
Acid, Azo-Colouring Matters, Influence of Constitution on Fastness of. (Bony).....	381
Acid Ester of Acetol, Manufacture of. (P) Fritsch.....	274
Acid, Estimation of Saccharine in Presence of. (Hairs)....	424
Acid, German Trade in. (T.R.).....	1111
Acid in Food, Detection of. (McElroy).....	670
Acid, New Method for Estimating. (Barthe).....	1105
Salines in Mexico, Discovery of. (T.R.).....	910
Salipyrine, Electrical Phenomenon observed in the Preparation of. (Schrinen).....	428
Salols, Manufacture of. (P) Byk.....	274
Production of. (P) Kolbe.....	275
Salt, Action of, on the Putrefying Bacteria of Hides. (Hach- lein).....	49
Decomposed in the Leblanc and Ammonia Soda Process....	729
Discovery in Turkestan. (T.R.).....	819
Evaporators. (P) Craney.....	1186
In the Isle of Man. (T.R.).....	673
Treatment of, and Formation of Blocks by Fusion. (P) Haddan. From La Cie du Sel Agglomere (illus.).....	395
Saltenke, German Trade in. (T.R.).....	1111
Saltpetre, Bengal. (T.R.).....	676
German Trade in. (T.R.).....	1111
Salts. (Class VII.).....	34, 151, 249, 321, 513, 631, 729, 808, 884, 947, 1057, 1195
Action of, in Rendering Clay Flocculent. (Sachse and Becker).....	409
Action of, on Acids. (Wood).....	515
And Compounds thereof, Electrical Separation of Bases of, from Saline Solutions. (P) Vautin.....	256
And Sulpho-Derivatives of Certain Colouring Matters. Manufacture of. (P) Petersen-Müller.....	725
Apparatus for Electrolysis of. (P) Hargreaves and Bird.....	958
Apparatus for Electrolytic Decomposition of Metallic. (P) Kellner.....	401
Effect of Metallic, in Development of Colouring Matters on the Fibre. (Carbetti and Peco).....	946
Electrolysis of Various. (P) Hargreaves and Bird.....	886
Employment of Ammoniacal, as Resist. (Bontemps).....	1195
Extraction of Pure Acids from Alkaline. (P) Paet and Bonnet.....	37
Fusibility of Mixtures of Isomorphous. (Le Châtelier) ..	634
Manufacture of Barium.....	322
Of Alkalies, Minimum Electro-Motive Force Required for the Electrolysis of Dissolved. (Nourisson).....	742
Of Ammonia, Apparatus for Obtaining. (P) Lake. From Sternberg.....	887
Of $\beta$ -Naphthylsulfamine, Preparation of. (P) Tobia.....	800
Of Fatty Acids and Soaps, Behaviour of, in Presence of Water. (Krafft and Stern).....	1207
Of Iron and Zinc, Separation and Precipitation of, from Solutions. (P) Bonlt. From Warzee.....	1193

## Salts—cont.

Of Soda and Potash, Separation of, from Solution of Magnesium Chloride. (P) Bell.....	37
Of Zinc, Art of Preparing Solutions containing. (P) Choate.....	670
On the Poisonous Action of Lead. (Bodson).....	270
Preparation of Basic Organic. (Kassner).....	270
Preparation of Ferricyanide. (P) Warnek.....	270
Preparation of Ferrons. (P) Warnek.....	270
Process and Apparatus for Electrolytically Dissolving Soluble. (P) Haddan and Co. From Blackmore.....	257
Process and Apparatus for Treating Electrolytically. (P) Stormer.....	1067
Production of Basic Lead. (P) Lunge and Maxwell (illus.) ..	731
Solubility of Some Inorganic, in Organic Liquids. (Van Lascezyński).....	1195
The Electrolysis of Fused. (Vautin) (illus.).....	118
Volatility of Alkaline. (Cameron).....	1074
Volumetric Estimation of the Bases in certain Metallic (English).....	245
Sampling by Gravitation. (Shiner and Reinsnyder).....	277
Notes on. (Sorley).....	955
Of Materials, The. (Clarkson) (illus.).....	211
Sandstones, The Weathering Properties of. (Bilton).....	80
Sanguinarine.....	80
Sanitary Chemistry. (Class XVIII.).....	56, 160, 271, 452, 537, 656, 753, 829, 898, 950, 1083, 1215
Sarcostemma australe.....	897
Saturation of Sugar Juice, Continuous. (Reboux).....	719
Saturators and Jets for Producing the Oxy-hydrogen Light. (P) Suiter.....	874
Sauer's Method for Estimation of Sulphur. (Marchlewski) ..	283
Scab Wash for Sheep. (P) Robertson.....	833
Scale in Boilers, Prevention of. (Walter).....	622
Scandinavia as a Source of Iron Ore Supply. (T.R.).....	675
Schlossing's Process for Estimating Nitric Acid, New Method of Performing. (De Koninck) (illus.).....	769
Semi-Fermentation in Japanese Sugar Factories. (Gurlinks) ..	823
Seal Oil, The Analytical Constants of. (Chapman and Rolfe) ..	813
Sections, Discussion on the Powers of the Local.....	693
Seed Industry of India. (T.R.).....	1118
Seeds, Dissemination of Saccharose in. (Schulze and Frankfurt).....	533
Seggars for Earthenware Kilns. (P) Copeland.....	888
Separator, Improved Oil, for Use with Feed-Water Apparatus. (P) McDonnell.....	22
Separators, Centrifugal. (P) Linden.....	871
Centrifugal. (P) Ström (illus.).....	1181
Centrifugal, for Cream and Butter. (P) Walden.....	56
Centrifugal Liquid. (P) Lundström and Barrel (illus.).....	24
Sesame Oil in Olive Oil, Detection of. (Musset).....	423
Sewage and Foul Waters, Compositions for Treatment of. (P) Burghardt.....	872
And Waste Waters, Treatment of. (P) Jarman.....	892
Compound for Purifying. (P) Whalley.....	696
Disinfection of by Sulphuric Acid. (Kanehl).....	537
Electrical Purification of, on the Heron System. (Vitoux).....	753
Filtering. (P) Rushford and Edwards.....	271
Iron Compounds for Treating. (P) Lovinstein.....	1216
Manufacture of Manure from. (P) Currier.....	964
Matter, Treatment of. (P) Nadein, Ewdokimoff, and De Bachmakoff.....	56
Precipitating the Solid Matter contained in. (P) Mosley.....	960
Precipitation and Purification of and Means therefor. (P) Caudy.....	1215
Production of Compounds for Use in Treatment of. (P) Sharp and Petrie.....	1211
Purification and Filtration of. (P) Sharples.....	656
Purification of. (P) Archibult and Deely.....	830
Purification of. (P) Meldrum.....	113
Purification of, and Apparatus therefor. (P) Ashney and Parry.....	831
Purifying and Disinfecting. (P) Hope.....	831
Receptacles or Channels for, and Disinfecting Same. (P) Stoffort.....	950
Sludge, Treatment of. (P) Tanner.....	832
Sludge, Utilisation of. (P) Twyman.....	1084
Tanks for Precipitation of. (P) Ives (illus.).....	832
Treating the Effluent of Purified. (P) Webster.....	169
Treatment and Utilization of. (P) Jones.....	56
Treatment, Comparative Results of some Modern Systems of. (Naylor).....	714
Treatment, Discussion on W. Naylor's Paper on.....	608
Treatment of. (P) Tanner.....	832
Treatment of, by Ferric Compounds. (P) Tanner.....	768
Utilising Precipitated, for Manufacture of Cement. (P) Bruch.....	1188
Shale Oil, Bounties on Manufacture of, in France. (T.R.).....	183
Sheep, Scab Wash for. (P) Robertson.....	833
Shellac Industry of Bengal, The. (T.R.).....	774
Industry of India, The. (T.R.).....	1226
Shoes, Dressing for. (P) James.....	717
Siam, The Gum Bezoar of. (Ludy).....	821
Sialosinotannol. (Ludy).....	81
Sicily, Manufacture of Curic and Tartaric Acids in. (T.R.) ..	676

	PAGE		PAGE
Soda, Decomposition of Liquids by Contact with Powdered.....	428	Soda—cont.	
Esterification of Camphor.....	1094	And Monocalcic Phosphate, Manufacture of. (P) Hurter	394
Silicates, <i>See</i> In An Application of. (Weissenheimer).....	727	And Potash, Densities of Solutions of. (Pickering).....	511
Silicates, Manufacture of Alkaline. (P) Reich.....	1029	And Sulphate of Potash, Duty on, in Finland. (T.R.).....	290
Preparation of Anhydrous Crystalline. (Franken).....	518	Apparatus for Calculating Bicarbonate of, into Carbonate.	
Silicon Carbide, "Carborundum." (Muhlhauser).....	402	(P) La Soc. Marcheville Daguin et Cie.....	518
Carbide, Production of. (Muhlhauser).....	35	Apparatus for Making Caustic. (P) Petrie.....	188
Variation in Percentage of, in Samples of Pig-Iron from		Baryta Process, The. (Vivien).....	165
Same Tap. (Rubrics).....	889	Crystals, Manufacture of. (P) Browne and Guthrie.....	334
Silk, (Class V.).....	32, 146, 218, 384, 512, 632, 725, 802, 880, 914	German Trade in Chromate of. (T.R.).....	1110
Action of Nitric and Nitrous Acids on. Vignon and		Influence of Acid Molybdate of, on Rotatory Power of	
Lasky.....	32	Rhamnose. (Görnez).....	1220
Discharges and Resist on. (Kochlin).....	727	Manufacture of Caustic. (P) Garroway.....	735
Formate of Resorcinol of Green on. (Pokorny).....	727	Manufacture of Permanganate of. (P) Brock and Hurter	394
Influence of "Weighting" on Specific Gravity of. (Still-		Method and Apparatus for Manufacturing Caustic. (P)	
mann).....	907	Craney (illus.).....	636
Method and Apparatus for Producing Artificial. (P)		Nitrate of, in Egypt. (Mackenzie).....	611
Lehner.....	1192	Obtainment of Carbonate of. (P) Penakoff.....	957
Rendering Sensitive to Light. (P) Junk.....	173	Production of Caustic and Chlorine by Electrolysis of	
Sample. (Silbermann).....	1066	Salt. (P) Drake.....	744
Treatment of Tussah. (Silbermann) (illus.).....	880	Production of Pure Caustic from Sodium. (Rosenfeld).....	249
Wild. (Silbermann).....	384	Statistics of Nitrate of, 1880-1893. (T.R.).....	81
<i>See also under</i> Textiles.		Treatment of Bicarbonate of. (P) Bell.....	725
Silks, On Vegetable. (Arnand).....	147	Utilising Acid Sulphates of. (Chatfield).....	735
Silver and Bismuth, Separation of. (Jannasch and Lesinsky)		Sodium and Lead, Electrolytic Production of an Alloy of. (P)	
And Gold, Extraction of, from Solutions. (P) Raleigh.....	402	Vautin.....	744
And Lead, Separation of. (Jannasch).....	60	And Potassium, Utilisation of Chlorides of. (P) Parker	
And Lead, Separation of. (Jannasch and Lesinsky).....	67	and Robinson.....	250
Bismuth Alloys, On the Cupellation of. (Smith).....	765	Biborate, Manufacture of. (P) Ascough.....	1197
Copper Alloys, The Liquidation of. (Matthey).....	889	Boro-Sulphate. (Adam).....	834
Electrolytic Separation of from Gold. (Schmiedel).....	743	Chloride, Apparatus for Electrolysis of. (P) Drake.....	887
Extraction of, from Ores. (P) MacArthur, Ellis, and The		Chloride, Utilisation of Products of Electrolysis of. (P)	
Class T.G. Gold Extracting Co.....	526	Haddon, From La Comp. Electro-Chim. de St.	
Extraction of, from Ores, &c. (P) Montgomerie.....	956	Beron.....	886
Extraction of, from Ores or Matte. (P) Mierisch.....	1067	Ethylate, Action of, on Magenta. (Prud'homme and	
Haloid Emulsions for Photography. (P) Prestwich.....	417	Rabaut).....	382
Method and Apparatus for Extracting, from Ores. (P)		Hydroxide, Process and Apparatus for Manufacture of.	
Ting.....	527	(P) Kellner.....	152
Ore of Moly. (T.R.).....	432	Nitrate Deposits of Columbia, The New. (T.R.).....	1001
Precipitating, from Cyanide Solutions. (P) Bell and		Peroxide, Action of Ammonia Gas on. (Michel and	
Carter.....	816	Grandmouzin).....	37
Recovery of, from Lead. (P) Lyte (illus.).....	614	Peroxide, Action of, on Acids in Alcoholic Solution.	
Treating Zinc-Lead-Sulphide Ores containing. (P) Fell.		(Tafel).....	885
From The Ermen's Zinc Co.....	402	Peroxide, Analytical Use of. (Poleck).....	939
Volumetric Determination of. (Demers).....	664	Peroxide, Behaviour of, towards Alcohol. (Tafel).....	1195
Siphon Level Regulator. (Bidel) (illus.).....	661	Peroxide, Decomposition of, by Aluminium. (Rossel and	
Sizes, (Class XIV.).....	49, 164, 408, 552, 619, 746, 821, 894, 961	Frank).....	516
Skins or Hides, Tanning. (P) Zahn.....	822	Peroxide, Explosion caused by. (Dupré).....	118
Treatment of. (P) Lake, From Strauss.....	51	Polysulphide for Bleaching and Cleansing Purposes.	
<i>See also under</i> Hides and Leather.		(Bankelman).....	945
Slag Cement Experiments. (Mahon).....	756	Production of Pure Caustic Soda and Hydrogen from.	
Detection of Adulterants in Basic. (Böttcher).....	978	(Rosenfeld).....	249
Plant for Granulating. (Terhune).....	522	Salt of Perchlomic Acid. (Hausermann).....	289
The Principal Smelting Reactions of Blast Furnace.		Silicofluoride, Preservation of Wines by Means of. (Surre)	
(Ebers).....	308	Sulphate, Production of. (P) Enquist.....	948
The Utilisation of Blast-Furnace. (Ebers).....	643	Sulphate, Action of Hydrogen and Sulphurous Acid on	
Slags, Valuation of Ground Phosphate. (Wraupelmeyer).....	422	Oleic and Erucic Acids. (Saytzeff).....	1208
Slip, Apparatus for Drying. (P) Peake.....	39	Tanks for Electrical Decomposition of Chloride of, in	
Slide, Spontaneous Ignition of Dried Petroleum. (Kissling)		Solution. (P) Richardson.....	646
Smelting, Electric and Furnaces therefor. (P) Newerth.....	160	Thiosulphate for Iodometry. (Meineke).....	420
Electrical. (Mason).....	711	Soils, Determination of Probable "Available" Mineral Plant	
Smoke, Colouring Tanned Hides by means of. (P) Hermann		Food in. (Dyer).....	288
Heating Power of. (Tatlock).....	931	Soldier for Aluminium and other Metals. (P) Nicolini.....	645
Smoking Concert.....	708	For Use with Aluminium and Alloys. (P) Roman.....	956
Snap Manufacture. (Class XIII.).....	15, 162, 257, 404, 529, 617, 744, 819,	Soldering, Blow Lamps for. (P) Lake, From Hjorth and Co.	
And Washing Compounds. (P) Field.....	891, 959, 1068, 1207	Soldiers for Brass, Hard. (Schwirkus).....	1200
An Improved. (P) Taylor.....	1208	Solubility Tests of Dark Mineral Lubricating Oils. (Holde).....	668
A Superfatted Bimide of Mercury. (P) Hingworth.....	531	Solutions for Waterproofing Cloth. (P) Hendrichs.....	1192
Cakes and Bars. (P) Kummer.....	618	A Dropping Flask for Standard. (Vanderpoel) (illus.).....	602
Compositions, Manufacture of. (P) Baerlein.....	962	Apparatus for Electrolytic Decomposition of Salt. (P)	
For Removing Hair from the Skin. (P) Mülhner.....	106	Kellner.....	162
Improvement in Dye. (P) Holt.....	746	Apparatus for Evaporating Saccharine. (P) Morrell and	
In Lubricants New Reaction for Detection of. (Schwitzer		Fromertz.....	377
and Lungw.).....	1178	Carrying Salts of Zinc, Preparing. (P) Cheate.....	395
Manufacture of. (P) Peacock and Gills.....	230	Decolorising or Bleaching. (P) Abel, From König.....	824
Method of Coating. (P) Hildbrand.....	962	Electrical Separation of Bases of Salts and Compounds	
New Composition of. (P) Hamilton.....	106	thereof from Saline. (P) Vanlin.....	256
Powder, Manufacture of. (P) Scott.....	1070	Electrolysis of Saline. (P) Lienard.....	819
Process and Apparatus for Making. (P) Palmer.....	16	Evaporating Saline. (P) Bell.....	734
Solutions, Disinfecting Power of. (Jolly).....	754	Hydrostatic Apparatus for Treating. (P) Spence.....	713
Tablets, Manufacture of, and Apparatus therefor. (P)		Oil Phenols, &c., Obtaining Aqueous. (P) Helmers.....	906
Tinkley.....	405	Of Sweet Camphides in Oils, Fats, &c. (P) Sommer.....	1071
Soaps, German Trade in. (T.R.).....	1112	Purifying and Decolorising Saccharine. (P) Lach and	
Manufacture of. (P) Klumseh.....	1208	Benies.....	824
Most Useful for Securing Woolen Goods. (Lewkowitzsch)		Standard. (Buchanan).....	1093
258		Solvents, Apparatus for Driving off Heavy Vapours of. (P)	
Société d'Encouragement pour l'Industrie Nationale, Prize for		The Parent Syndicate Oil Extractor Co. and Dombain.....	240
Substitute for Gutta-Serena offered by.....	73	Influence of, on Rotation. (Schuster).....	72
Industrielle de Mulhouse, Prizes offered by the.....	73	Sophora Secundiflora.....	897
Sod Oil, Treating and Purifying. (P) Turney.....	16	South African Nitrate Fields. (T.R.).....	993
Soda, Action of Light on Metastable state of, and its Applica-		African Sugar Industry. (T.R.).....	675
tions. (Schön).....	388	Carolina Cotton-Oil Industry in. (T.R.).....	992
An Application of Silicate of. (Geisenheimer).....	727	Saxhelt's Arcometric Method of Determining Fat in Milk.	
And Chlorine, Apparatus for Electrolytically Producing.		(Tunpe).....	1104
(P) Lake, From the Union Chemical Co.....	257	Spain, Iron Mining in Northern. (T.R.).....	769
		Leather and Glove-making Industry in.....	821
		The Olive Oil Industry in. (T.R.).....	910

	PAGE
Specific Gravity Bottle, Note on an Improved. (Lomis) (illus.)	322
Gravity of Calcium Chloride Solution. (Pickering)	733
Spiese, A Japanese Pseudo, and its Composition and Relations. (Gowland) (illus.)	643
Sperm Candles, Rules for the Preparation of Standard	65
Oil and Mineral Oil, Analysis of Mixtures of. (Lobry de Bruyn)	126
Spermaceti, Determining the Melting Point of	66
Spoke Oil (Lavandula Spici). (Boucharlat)	60
Oil, Presence of Camphene in. (Boucharlat)	658
Spirit Duties, The New. (T.R.)	430
Sources of Error in Roscoe's Method of Estimating Fusel Oil in. (Glaserapp)	682
Spirits. (Class XVII.)	52, 167, 263, 410, 533, 653, 750, 821, 895, 966, 1077, 1211
Aeration of. (P) Fenn and Abraham	846
Alcoholic Strength of Imported. (T.R.)	130
Apparatus for Distilling. (P) Morris and Edney (illus.)	501
Distillation of, and Manufacture of Yeast. (P) Lederer	827
Manufacture of, and Apparatus therefor. (P) Murphy	751
Method and Apparatus for Maturing. (P) Leaker	655
Of Wine, Detection of the Higher Alcohols in. (Bardy)	287
Spots in Paper	538
Spray-Tanning. (Reinsch)	651
Squibs, Miners'. (P) Powell	700
Stability of a 1 per mille Solution of Corrosive Sublimite exposed to Air. (Tanret)	657
Stains on Woolen Cloth caused by Ammonio-Cupric Oxide. (Walter)	946
Standards for the Analysis of Iron and Steel, International	127
International, for the Analysis of Iron and Steel. (T.R.)	185
St. Andrews, River Tay and Perth, Visit to	705
Stannous Chloride Method for Titration of Iron, Modification of. (Mahon)	282
Starch. (Class XVI.)	52, 165, 263, 410, 533, 652, 718, 822, 965, 1076, 1210
Bleaching Raw. (P) Siemens Bros. From Siemens and Halske	34
Degradation of, by the Action of Diastase. (Lindner and Duhl)	53
Estimation of. (Munsche)	985
In Pressed Yeast, Determination of. (Filsinger)	908
Manufacture of Potato, by Continuous Flow System. (Bondonneau)	750
Manufacture of Soluble. (P) Lake, From Ancolec	265
Transformation, Some Products of. (Heipe)	267
Starches, Reports on Modified, prepared by Siemens and Halske	264
Statement of Revenue and Expenditure for 1893	571
Steam, Amount of, required in Sugar Manufacture. (Clasen)	824
Apparatus for Superheating. (P) Musgrave and Dixon	1018
Filter for. (P) Moeller	711
From Cane Juice, Separating and Utilising. (P) Chapman	1077
Jets, Wear of Metal Parts by Action of. (Walter)	134
Superheaters. (P) Grouvelle and Arqueboug (illus.)	930
Stearin, Cotton, and Maize Oil. (Hart)	257
Pitch. (Donath and Strasser)	350
Steel, Analysis of. (Bamber)	665
And Iron, International Standards for Analysis of	185, 327
And Iron, Manufacture of. (P) Sattmann and Homatsch	401
And Iron, On the Oxidation and Corrosion of. (Thomsen)	118
And its Alloys, The Electric Fusion of. (P) Thwaite	528
Avoiding Choking of Mouth of Retort in Manufacture of "Thomas." (P) Raze	158
Basic, Production in 1893. (T.R.)	292
Baths for Toughening, Strengthening, and Hardening. (P) Bates	645
Casting Solid Ingots of. (P) Frick	402
Castings, The Walrand-Legenis Process applied to. (Snelus)	642
Coating, with Brass and other Alloys. (P) Fenby and Moore	400
Conditions in which Carbon Exists in. (Arnold and Read)	710
Containing Silicon, Estimation of Phosphorus in. (Spuller and Kahan)	175
Conversion or Purification of. (P) Boulton, From Kone-man, Singer, and Hatch	42
Determination of Carbon in. (Hempelt (illus.)	421
Determination of Carbon in. (Koehl) (illus.)	979
Determination of Nickel in. (Westesson)	666
Determination of Nickel in Nickel. (Campbell)	666
Determination of Phosphorus in. (Doollite and Eavenson)	666
Determination of Phosphorus in. (Dudley and Pease)	665
Determination of Phosphorus in. (Handy)	666
Elimination of Sulphur from Molten. (P) Phillips	42
Estimation of Chromium and Manganese in. (Schneider)	546
Estimation of Chromium in Chromium. (Spuller and Kalman)	281
Founding. (P) McRoberts	645
Furnace, The Economic Position of the Open-Hearth. (Campbell) (T.R.)	82
Manganese. (Le Chatelet)	1198
Manufacture of. (P) Allen and Bay	815
Manufacture of. (P) Justice, From La Soc. Anon. des Hauts Fourneaux de Dufelange	42

## Steel—cont.

Manufacture of. (P) Thompson, From Bismarck	4
Thick. (P) Thompson	516
Manufacture of. (P) Thwaite	528
Manufacture of, in the Open-Hearth. (Benjamin)	731
Manufacture of Manganese. (P) Hatfield	731
Manufacture of Open-Hearth, in Sweden. (Odelstrom)	1067
Method and Apparatus for Hardening and Tempering. (P) Fairfax and Bendin	1067
Plates, Pickling. (P) Kirkman	645
Plates, Preparation of, for Plating. (P) Chapman	644
Process for Surfaceing. (P) Thompson, From Kone-man	32
Production of. (P) Cochran and Fawcett	711
Results of High Treatment on Manganese and Iron. (P) Raze	641
Surfaces, Effecting the Protection of. (P) Bart and Bower	11
The Structure of. (Osmond)	1065
Sterilisation, Apparatus for. (P) Hussener	1215
Of Alimentary Liquids. (Kuhm)	1133
Still or Retort for use in Kjeldahl's Process. (P) Edwards	760
Stills, The Locality of Laboratory. (T.R.)	78
Stone and Marble, Manufacture of Artificial. (P) Baumeit and Pock	630
Bricks, Tiles, &c, Artificial. (P) Gaskell and Robinson	154
Manufacture of Artificial. (P) Grate	255
Manufacture of Artificial. (P) McLean and Wilson	888
Stoneware, Composition of. (P) Sellers and Rogers	888
Producing Enamelled Letters and Designs on. (P) Hodan	811
Stop-Clack for Vacuum Desiccators. (Ernst) (illus.)	62
Storage Batteries. See under Batteries	
Straw Cellulose, Manufacture of. (Heveridge)	101, 213
Stress, Effect of Mechanical, on Electrical Properties of Metals. (Gray and Henderson)	957
Effect of, on Corrosion of Metals. (Andrews)	1269
Strontium and Calcium, Separation of. (Browning)	282
Strumite. (Niederlander)	292
Styrene, Behaviour of, towards Vital's Atropine Reaction. (Menyaz)	981
Styandra glauca	897
Sublimite, Influence of Alcohol, &c, on Bacterioid Action of Corrosive. (Lense)	963
Succinic Acids formed in Fermentations, Estimation of. (Bau)	287
Succinimides, Production of Iodo-derivatives of, and of Substituted. (P) Murray, From The Farb. vorm. Meister, Lucius and Brunn	800
Production of Para-methoxy- and Para-methoxy-phenyl. (P) Murray, From The Farb. vorm. Meister, Lucius and Brunn	800
Sugar, Starch, Gum, &c. (Class XVI.)	52, 165, 263, 410, 533, 652, 718, 822, 965, 1076, 1210
Amounts of Coal and Steam required in Manufacture of. (Clasen)	824
Assay, Muffle for. (Schweitzer and Lenzwitz) (illus.)	858
Beet Returns of Germany. (T.R.)	202
Beetroot Growing Experiments in 1893. (Schack Sommer)	213
Camphor as a Reagent for. (Seitz)	843
Contained in Massicot, Quantity of Crystalline. (Nylen)	1210
Content of Tanning Materials, &c. (Von Schroeder and others)	1101, 1227
Continuous Refining or Bleaching of, and Apparatus therefor. (P) Prancey	265
Decomposition of, during Evaporation. (Lexal)	965
Decomposition of, during Evaporation. (Strohmer)	1210
Decomposition of Invert, by Alkalis. (Prinsen-Georgius)	410
Estimation of. (Von Schroeder, Bartel, and Schmitz-Dumont)	1101
Factories, Recovery of Nitrates in	729
Factories, Seum Fermentation in Javmose. (Gurlings)	823
Factory, Loss of Heat in the Steam Engines of a. (Clasen)	718
Formation of Cane, during Germination of Barley and Ripening of the Apple. (Lindet)	750
Formation of, in the Liver. (Cavazzani)	845
Gravimetric Estimation of Cupric Reducing Power of Invert. (Bortrager)	1236
In Beetroots, Determination of Crystallisable. (Pellet)	669
In Beetroots, Estimation of. (Kroeker)	823
Industry, Improvements in the Beetroot. (Jarkovsky)	965
Industry of South Africa, The. (T.R.)	675
Industry of Sweden, Beet-. (T.R.)	556
Influence of Calcium Salts on Yield of, from Massicot. (Szyfer)	965
Influence of Cane, on Valuation of Malts and Worts. (Prior)	1080
Influence of Lead Acetate on Cupric Reducing Power of Invert. (Bortrager)	1230
Influence of Percentage of Fat in Beet Seed on Richness of Beets in. (Doerflinger)	410
Influence of Volume of Lead Acetate Precipitate on Titration of. (Bortrager)	1230
Inversion of, by Glycerol. (Bord)	1211
Juice, Continuous "First Saturation" of. (Francis) (illus.)	166
Juice, Continuous Saturation of. (Rebock)	749
Juice, Defecation of Diffusion, with Pulverised Slaked Lime (Mittelmann)	166
Juice, Defecation and Filtration of. (Mittelmann)	965
Juice, Filter for Diffusion. (Bouvier)	167
Juice, Filtration of Diffusion, after Heating	749
Juice, Precipitates obtained in Evaporating. (Shorey)	750

	PAGE		PAGE
Juice, Purification of, (Luedtke).....	749	Sulphuretted Hydrogen, Washers for Separating, from Gas, (P) Cripps (illus.).....	379
Juice, Purification of, (Triple Effect Apparatus) (Mittelschmidt).....	163	Hydrogen, <i>See also under Hydrogen.</i>	
Juice, Treatment of, (P. Leach, From Cambray).....	1211	Sulphuric Acid, Action of, on Metallic Copper, (Schuster).....	957
Juices and Syrops, Use of Asbestos for Filtration of, (M. Ziegler).....	244	Acid, Action of, on Pyrocatechol and Homopyrocatechol, (Consin).....	60
Liquefaction and Boiling of, (Lexal).....	823	Acid, Apparatus for Concentrating, (P) Levinstein.....	152
Liquefaction, Distillation and Saturation of, (Beaudet).....	965	Acid, Apparatus for Concentrating, (P) Bradbury.....	251
Loss, (M. Ziegler).....	965	Acid, Apparatus for Concentrating or Vaporising, (P) Boulton, From Penchen and Clarke.....	251
Liquefaction, Boiling, Estimation of, (Feleemann and Herber).....	165	Acid Chambers, Pipe Columns for, (Gilchrist) (illus.).....	1142
Manufacture, Control of, (Battut).....	750	Acid, Concentrating, in Lead Vessels, (P) Wolters.....	887
Manufacture of, (P) Lake, From Van der Weide and Lues.....	265	Acid, Disinfection of Sewage by, (Ivanoff).....	537
Manufacture of, and Apparatus therefor, (P) Johnson, From Pollicium illus.).....	1076	Acid, Employing Electrical Heat for Concentrating, (Haußermann and Niethammer).....	301
Manufacture of, Waste, and Apparatus therefor, (P) Hubner.....	824	Acid, German Trade in, (T.R.).....	1111
Moulds, Galvanised Iron, with Internal Coating, (Donath).....	823	Acid in Commercial HCl, (Rurap) (illus.).....	904
Production of Russia, (T.R.).....	291	Acid Industry, Improvements in the, (D. Boissieu) (illus.).....	1057
Rate of Filtration of Solutions of, (Briedel).....	749	Acid, Manufacture of, (P) D'Andria.....	163
Refining, (P) Lungen.....	533	Acid, Manufacture of, (P) Garway.....	755
Solutions, Purifying and Decolorising, (P) Steffen.....	965, 1211	Acid, Manufacture of, in America, (Lunge) (illus.).....	884
Solutions, Purifying and Decolorising, (Steffen and Bruckert).....	823	Acid, New Processes and Apparatus for Concentrating, (Gerber).....	56
Use of Sulphurous Acid in Manufacture of, (P) Abraham.....	165	Acid, Production of Ethers of, (P) Newton, From the Farb, vorm. P. Bayer and Co.....	60
Yield of, from Maccumb, (Chisen).....	1210	Acid, Recovering and Concentrating Waste, (P) Levinstein.....	152
Years and After-products, Valuation of, (Karcz).....	1210	Acid, Use of Electrical Heat for Concentrating, (Bucherer).....	249
And Syrops, Manufacture of, (P) Lake, From Tedesco.....	886	Acid, Volumetric Method for Estimating, (Windisch).....	905
Behaviour of Various, towards Pure Yeast Cultures, (Fischer and Thierfelder).....	1211	Acid Works of Johannesburg, (T.R.).....	431
Calculation of the Rendement of R.V., (Seiffert).....	965	Sulphurous Acid and Hydrogen Sodium Sulphite, Action of, on Oleic and Erucic Acids, (Saytzeff).....	1208
Compounds of Hydrogen Cyanide with the Anilides of the, (Von Miller, Floed and Struss).....	767	Acid, Employment of, in Making, (Kukla).....	968
Compounds of, with Polyhydric Phenols, (Fischer and Jennings).....	765	Acid in Wines, Alteration of the, (Chuard and Jaccard).....	969
Compounds of, with Iron, (Evers).....	415	Acid, Manufacture of, (P) D'Andria.....	163
Estimating, by their Cupric Reducing Powers, (Grunhut).....	845	Acid, Use of, in Diffusion, (Mittelmann).....	165
Estimating Cupric Reducing Power for, (Allen and Gando).....	1229	Acid, Use of, in Sugar Manufacture, (Abraham).....	165
Estimating, by their Cupric Reducing Powers, (Nihoah).....	845	Sumatra, Petroleum in.....	796
Gravimetric Estimation of Cupric Reducing, (Prager).....	1227	Sunday-Rest Act, Chemical Works and the, (T.R.).....	1234
Grey Colour of Raw Beetroots, (Drenckmann).....	1210	Sunflower Cake, Meal of, (Theodor).....	960
In Malt, Estimation of the Ready-formed, (Jais).....	53	Oil, Properties of, (Holde).....	892
Of Malt, The Ready-formed, (Ehrich).....	805	Sunlight, Decomposition of Tartaric and Citric Acids by, (Seckamp).....	810
The Multitration of, (Muller).....	748	Superheater for Vapours, (Bidel) (illus.).....	601
Scent, Extraction of, from Wood, &c., (P) Richard-Lazerie.....	962	Superheaters, Steam, (P) Grouvelle and Arquebourg, (illus.).....	930
Sulfate of Alumina, Basic, (Perguson).....	675	Superphosphates, Volumetric Estimation of Soluble Phosphoric Acid in, (Kalmann and Meissels).....	842
Of Ammonia produced in the United Kingdom.....	729	Volumetric Estimation of Soluble Phosphoric Acid in, (Kalmann and Meissels).....	905
<i>See also under Ammonia.</i>		Suranji, Finetorial Properties of, (Hummel and Perkin).....	318
Of Copper, Manufacture of, (P) Strawson and Freun.....	57	Surinam, Balata Production in, (T.R.).....	430
Of Morphine, Manufacture of, (Calvert).....	172	Production of Banana-meal in, (T.R.).....	430
Of Potash and Soda, Duty on, in Finland, (T.R.).....	290	Susini's Ether Motor, (Freitag) (illus.).....	135
Sulphates, The Metallurgy of Metallic, (P) Mills.....	711	Swansonia.....	897
Action of Aluminium on, (Rossel and Frank).....	515	Sweden, Beet Sugar Industry of, (T.R.).....	556
Sulfide of Nickel, Oxidation of, (de Clement).....	151	Manufacture of Open-Hearth Steel in, (Odelstjerna).....	1065
Sulphite Cellulose, Decoloration of Papers made from, (Muth).....	538	Switzerland, Customs Decisions in, (T.R.).....	282, 959
Process, Liquors, Obtaining Useful Products from, (P) Ekman.....	1085	Synthesis of Azo Colours on Animal Fibres, (Pokorny and Werner).....	388
Pulp, Treatment of, (P) Partington.....	1085	Syrups, Characteristics of Glucose, (Stone and Dickson).....	178
Sulpho Acids and Derivatives thereof, Production of, (P) Newton, From The Farb, vorm. Bayer and Co.....	512	Clarification and Preservation of, (P) Evans.....	827
Acids of Phenyl rosindolime, Manufacture of, (P) Johnson, From The Badische Anilin und Soda Fabrik.....	510	Use of Asbestos for Filtration of, (Mainzen).....	264
Sulphogamides, Manufacture of, (P) Growther and Ross.....	887		
Manufacture of, and Apparatus therefor, (P) Brock and others.....	1195		
Sulpho-derivatives of certain Colouring Matters, Manufacture of, (P) Petersen-Muller.....	725		
Sulphonic Acids and Compounds, Obtaining, free from Inorganic Salts, (P) Helmers.....	508		
Acids, New Method of Estimating Naphthol and Naphthylamine, (Waubel).....	518		
Sulphur, Burning, for Disinfecting Purposes, (P) Lake.....	657		
Candles and Luminators, (P) Kugzett.....	833		
Candles and their Holders, (P) Kugzett.....	657		
Candle, Production of, (Muller).....	35		
Compounds accompanying Anilin Bases, and their Colour Reactions, (Hantzsch and Freesen).....	1180		
Compounds in Ohio Petroleum, (Mabery and Smith).....	597		
Compounds in the Atmosphere, Attempts to Estimate, (Oates).....	1222		
Dioxide, Vessels for containing Lignelled, (P) Soy.....	271		
Elimination of, from Molten Iron or Steel, (P) Phillips.....	12		
Estimation of, in Sulphides, with Simultaneous Determination of Arsenic, (Jannasch) (illus.).....	965		
In Cast-Iron, (Kepp).....	524		
In Gas-purifying Material, Estimation of, (Meyer) (illus.).....	283		
Mining in Louisiana, (T.R.).....	965		
Petroleum, Preliminary Examination of Canadian, (Mabery).....	597		
Reducing Sulphuretted Ore into Oxides and, (P) Bemelmann.....	153		
Removal of, from Mineral Oils at Chicago, (Riche and Roume).....	719		
Studies of Methods for Estimation of, (Marchlewski).....	283		
Test for Free, (Gil).....	175		

## T

Table Oils in Germany, New, (T.R.)	80
Tallow, Estimation of the Titre of, (Wolfbauer)	908
Method for the Valuation of, (Wolfbauer)	181
Refined Fish, (Eitner)	894
Tanks for Electrolytical Decomposition of Solutions of Chloride of Sodium or Potassium, (P. Richardson)	646
For Precipitation of Sewage, (P. Ives (illus.)	832
For Storage of Compressed Air, Gas, &c., (P. Timmis)	788
For Subsidence of Sewage Sludge, (P. Lockwood)	970
Tannic Extracts, Clarifying or Decolorising, (P. Sinan)	164
Tannin, Action of, on Hides, (Passler)	964
Course of Absorption of, in the Layers, (Sadlon)	49
Does it Tan Hides? (Procter)	717
Extraction of, from Myrabolans, (Von Schroeder)	1073
In Hops, Estimation of, (Hayduck and Goldner)	965
Worlands, Resist, with, (Casanovas)	882
Orange, (Schuch)	1189
Tanning, Leather, Glue, and Size, (Class XIV.)	49, 104, 408, 532, 619, 746, 821, 894, 964, 1072, 1209
Canagire as a Substitute for Barks in, (Von Schroeder and Passler)	49
Improvements in, (P. Reinzerling)	651
Liquors, Origin of the Acid Fermentation in, (Haelelin)	650



	PAGE		PAGE
Tanning— <i>cont.</i>		Theory of Mordant Dyeing	
Materials and Extracts, Sugar Content of, Von Schroeder and others	1101, 1227	Thermometer for High Temperatures, a New	1227
Materials, Water-Content of Common, (Von Schroeder)	1072	For High Temperatures, Mercury, (Mahlke)	62
Materials, Analysis of, (Felsing)	426	For High Temperatures, Mercury, (Recklinghausen)	71
Materials, Extraction of, (Schroeder and Bartel)	649	Thermostatic Instruments, (P) Beckes, From Gregory	71
Materials Qualitative Determination of, (Procter)	187	The Thermostat, Improvements in, (P) Shells	167
Matter, On the Estimation of, (Procter) (illus.)	494	"Thomas' Metallurgical Process, Improvements in the, (P) Wild	161
Sprays, (Reimsch)	651	Thymol Acetate, Constitution of the Methyl, (C) and (J)	84
With Extracts, (Vilhou)	746	Tiles, Manufacture of, (P) Gaskell and R. (illus.)	151
Tannins, Preparation of Oak, (Trimble and Peaseck)	59	Manufacture of Glazed, (P) Martin	46
Tapestry, Obtaining a Substitute for, (P) Lake, From The Fratelli Mora	34	Timber, Preservation of, from the Attacks of Insects, (De Garmun)	43
Tar Products (Class III.)	27, 213, 350, 507, 627, 719, 791, 871, 938, 1053, 1182	Tin and Cadmium, Electrolytic Separation of, (Schmuck)	283
Distillation of Birch, from Pines, (Hirschmann)	423	And Copper, Electrolytic Separation of, (Schmuck)	280
From Fuel, Apparatus for Obtaining, (P) Mond (illus.)	938	Consumption of, in India, (T.R.)	1116
German Trade in, (T.R.)	1113	Plate, Utilization of Old Articles of, (P) Gentsch	40
Of Pinus Maritima, (Reinard)	1188	Recovery of, from Plates and Waste, (P) Krebs and Harper	641
Of the Bark of Populus Tremula, (Pharmakofsky)	794	Recovery of, from Scrap and Metal, (P) Hurd	122
Pines, (Reinard)	1188	Resist for Silk, (Koechlin)	727
Valves for, (P) Cripps	508	Separation and Estimation of, in Alloys, (Menein)	1224
Washers for Separating, from Coal-Gas, (P) Cripps (illus.)	379	Separation of Lead from, (Jannasch and Reimann)	56
Tars, Mixing for Use for Heating or Lighting Purposes, (P) Sahlstrom and Farr	130	Solutions or Platines, Volumetric Estimation of Lead in, (Bayr)	420
Tariff Changes and Customs Regulations	74, 182, 280, 430, 550, 672, 703, 816, 903, 989, 1232	Sulphide, White Form of, Soluble in Ammonium Carbonate, (Schmidt)	1261
Changes in Canada, (T.R.)	846	Temperature of Fusion of, (Bogdanoff)	1053
Changes in Guatemala, (T.R.)	989	Tinctures and Essences, Revenue Concessions on Export of, (T.R.)	772
Changes in Jamaica, (T.R.)	1232	General Order as to Export of, (T.R.)	183
Changes in Norway, (T.R.)	290	Recovery of Residual, from Mares, (Parker) (illus.)	973
Changes in Victoria, (T.R.)	430	Table of Results of Estimating, Gravimetrically and Volumetrically	984
Law, Proposed New, (T.R.)	78	Titre of Tallow, Estimation of the, (Wolffbauer)	908
Of the United States, (T.R.)	847, 901	Tobacco-Juice, Extraction of Nicotine from, (T.R.)	850
Tarragon, Essence of, and its Transference into Anethol, (Grimaux)	658	Pipes, Porous Material suitable for, (P) Smith	395
Tartar Emetic Resist for Silk, (Koechlin)	727	Treatment of, to Prevent the Injurious Action of Nicotine, (P) Lake, From Gerold and Wendl	275
Tartaric Acid, Decomposition of, by Sunlight, (Seckamp)	810	Tolyl-Antipyrine, Derivatives of, (Ebert)	79
Acid, German Trade in, (T.R.)	1111	Tomatoes, Detection of Eosin in Coloured, (McElroy and Bicklow)	284
Acid in Wines, Methods of Estimating Free, (Carpene)	908	Tortimolite Process, The	89
Acid, Manufacture of, in Sicily, (T.R.)	676	Toxers and Ducts, Motion of Gases in, (Jurisch)	513
Acid, Spontaneous Formation of Pyruvic Acid from, (Otto)	733, 733	Trachymene Australis	897
Acid, Supposed occurrence of Free, in Wine, (Carpene)	1080	Tracing Cloth, Production of Material for Use as, (P) Goodwin	803
Acid, The Tendering of Cotton Fibre by, (Scheurer)	118	Trade of Germany in 1891, The Foreign, (T.R.)	290
Materials, Italian Trade in, (T.R.)	677	Trade Report, 71, 182, 280, 430, 550, 672, 703, 846, 903, 989, 1108, 1232	
Materials, Production of, in Italy, (T.R.)	1234	Train Oil, Detection of Paraffin and "Lubricating Oil" in Distillate from, (P) Engler and Sauer	45
Tatlock's Process for Estimation of Potash, (Bodsi)	1005	Transvaal Mines, The Cyanide Process in the, (Feldmann)	951
Tea and Coffee, Preparing Extracts of, and Utilising By-Product, (P) Sonstadt	270	Treaty between Russia and Germany, Proposed New Commercial, (T.R.)	289
Tea-Seed Oil, (T.R.)	79	Trigonellin, Occurrence of, in Seeds of Pisum Sativum and Cannabis Sativa, (Schulze and Frankfurt)	737
Telluric Acid, Physiological Action of, (Bokorny)	289	Trihydroxy-naphthalene Monosulpho Acid, Production of a, (P) Newton, From The Farb, vorm. Bayer and Co.	1054
Temperature, Determination of High, by Fusion Tests, (Schneider)	702	Trinitroresorcinol Smokeless Drying Powder, Employment of, (P) Hauff	975
Influence of, on the Tensile Strength of Metals, (Rudeloff)	520	Triphenylmethane Colouring Matters, Behaviour of, to Nascent Bromine, (Vaubel)	876
Of Bread as it Comes from the Oven, (Balland)	55	Tube for Polarimetric Observations, (Hamp)	278
Of Fermenting Liquids, Apparatus for Regulating, (P) Collins	890	Tubes for Heating, Cooling and Evaporating Apparatus, (P) Thompson, From Dumas	716
Of Fusion of Tin, (Bogdanoff)	1006	For Heating, &c., (P) Dumas	690
Temperatures, Mercury Thermometer for High, (Mahlke)	62	For Heating Liquids, Metallic, (P) Goldthorp	929
Mercury Thermometer for High, (Recklinghausen)	61	Of Covered Glass, Manufacture of, (P) Browne, From Kastner	199
Tephrosia Purpurea and Rosen	857	Tungsten, Electrometallurgy of, (T.R.)	14
Ternary Alloys, On certain, Part VIII, (Alder Wright)	521	Turkistan, Salt Discovery in, (T.R.)	869
Terpene Series of Hydrocarbons, Discussion on Production of Oil-Gas from	501	Turkey, Importation of Patent Medicines into, (T.R.)	508
Series, Production of Gas from Pure Members of the, (Tocher) (illus.)	231	Red Dyeing, Improvements in, (Erhan and Specht)	159
Terpenes in Petroleum, (Zaloziecki)	1187	Red Dyeing, Improvements in, (Muller)	159
The Aerial Oxidation of, (Kinzett)	275	Red Oils, Constitution of, (Julliard)	823
Terra-Cotta Blocks, Moulds for, (P) Doulton and Carr	948	"Turkish Honey," The So-called, (Ejano)	672
Cottas, Manufacture of, (P) Schliekeyson	154	Turpentine, Buying, by Weight, (T.R.)	1115
"Tesu," The Colouring Matter of the Indian Dyestuff, (Hummel and Cavallo)	144	Determination of Oil of, in Alcohol, (Pohl)	427
Testers, Improvements in Milk, (P) Pedersen	56	Oil, German Trade in, (T.R.)	1112
Textile Fibres, Influence of Structure of, upon Absorption of Colouring Matters, (Georgievics)	807		
Fibres, Process for Retting, Cleansing, and Treating, (P) Grischwitz	1053		
Textiles, (Class V.)	32, 148, 248, 384, 312, 632, 725, 802, 880, 944, 1056, 1192		
Apparatus for Drying and Carbonizing, (P) Dasse and Fettweis	1192		
Apparatus for Dyeing and Washing, (P) Labhardt	391		
Apparatus for Soaping and otherwise Treating, (P) Jackson and Hunt	248		
Detergent Compound for Securing, (P) Brothers	260		
Dyeing, (P) Lake, From Michaelis and Co.	634		
Dyeing, in Cops or Spools, (P) Erhan and Specht	1194		
Dyeing, Sizing, Bleaching, &c., (P) Smith	513		
Method and Apparatus for Dyeing, (P) Lake, From Mattei	728		
Rendering, Waterproof and Flame-proof, (P) Rous and West	725		
Research on the Hygroscopic Properties of, (Schloessing, jun.) (illus.)	146		
The Clouding of, in Dyeing, (P) Boulton, From Renard	1194		
Treatment for Stiffening, (P) Goodwin	803		
See also under Fabrics and Fibres,			

Ultramarine, (Jordan)	493
United Kingdom Mineral Production for 1892, (T.R.)	554
States, Chemical Considerations on the Pottery Industries of the, (Langenbeck)	153
States, Chemical Industry of the, (T.R.)	557

	PAGE		PAGE
United States		Walrand-Legentis Process applied to Steel Castings, The. (Schelus).....	612
States, Chemical Manure in the. (T.R.).....	431	Washers for Extraction of Tar, Ammonia, Carbonic Acid, and Sulphuretted Hydrogen from Coal-Gas. (P) Cripps (illus.).....	379
States, Customs Decisions in the. (T.R.).....	74, 182, 569, 818, 100, 1232	Washing and Cleaning Compounds. (P) Field.....	962
States Exports of Paraffin Wax. (T.R.).....	293	Blue. (P) Ammann.....	1209
States Legislation Discharge of Refuse. (T.R.).....	1233	Machines, Rotary. (P) Crabtree.....	881
States Mineral Production of the. (T.R.).....	184	Waste from Gas-Liquor, Treatment of. (P) Crowther.....	635
States Mineral Production in 1894. (T.R.).....	432	Liquors from the Refining of Petroleum, Examination of. (Zaloziecki).....	628
States Patent Office Rules, Changes in. (T.R.).....	433	Manufacture of Cement from Alkali. (P) Riehy, Neill, and Farr.....	638
States, Proposed Changes in the Patent Law of the. (T.R.).....	184	Tin-Plate, Recovery of Tin from. (P) Kriens and Harper.....	614
States Reports on the Dyewool Industry. (T.R.).....	431	Wastes. See also under Refuse and Residues.	
States, Statistics of Manufacture of. (T.R.).....	1170	Water, Action of, on Bicalcium Phosphate. (Joly and Sorel).....	634
States Tariff, Contemplated Changes in. (T.R.).....	532	Apparatus for Distilling and Sterilising. (P) Nagel.....	623
States Tariff of the. (T.R.).....	847	Apparatus for Distilling Sea-. (P) Fouché.....	714
States Tariff, The New. (T.R.).....	991	Apparatus for Electrolysis of. (P) Siemens Bros. and Co. and Obach.....	529
Strontium, Manufacture of. (Niederlander).....	262	Apparatus for Filtering and Purifying. (P) Winkler.....	377
Tannin, Manufacture of. (Mühlbauer).....	35	Apparatus for Filtering and Sterilising. (P) Kowalski (illus.).....	1084
Tar, Fixation of, on Textile Fibres. (Odenheimer).....	386	Apparatus for Obtaining Distilled and Sterilised. (P) Nagel.....	169
Tar, Application of, as Mordants. (Odenheimer).....	386	Apparatus for Producing Artificial Chalybeate. (P) March.....	529
Ten's Brewery, Visit to Messrs. ....	701	Apparatus for Purifying. (P) Webster.....	169
		Apparatus for Purifying Feed-. (P) Wotherspoon and Davie.....	505
		Apparatus for Removing Impurities from. (P) Shedlock.....	23
		Apparatus for Softening. (P) Gittins.....	271
		Apparatus for Softening and Purifying. (P) Smith.....	538
		Apparatus for Sterilising. (P) Yazu.....	413
		Contained in Air-dried Bark-tanned Leather. (Von Schroeder).....	1073
		Content of Common Tanning Materials. (Von Schroeder).....	1072
		Continuous Apparatus for Sterilisation of. (P) Herscher.....	1216
		Decomposition of Glass by. (Foerster).....	1060
		Determination of Nitrates in Potable. (Gill).....	663, 664
		Determination of Reducing Action of Organic Substances of, upon Potassium Permanganate. (Zegn).....	427
		Device for Filtering. (P) Wittman.....	1083
		Drinking, obtained from Artesian Wells at Mantua. (Bonato).....	752
		Effect of, on Glass. (Foerster).....	518
		Effect of the Saline Constituents of, on the Character of Beer. (Readman).....	367
		Gas. See under Gas.	
		Influence of Ferruginous, in Malting. (Kukla).....	1080
		Method and Apparatus for Purifying. (P) Walker.....	169
		Of Rivers, Natural Diminution of the Dissolved Organic Matter in. (Young).....	318
		Ovens and Water Baths, Automatic Alarm for. (Webster) (illus.).....	205
		Parantrophol as an Acid Indicator in Testing Feed-. (Goldberg).....	419
		Purification of. (Vilken).....	271
		Purification of and Apparatus therefor. (P) Maiche.....	831
		Purifier, the Dervaux Feed-. (Simon Thomas).....	376
		Purifying, by Oxide of Tin. (P) Von der Lind and Hess.....	56
		Removal of Iron from Drinkings. (Schuster).....	1083
		Removal of Pathogenic Bacteria from Drinking-. (Filler).....	830
		Separating Impurities from Boilers. (P) Lyon.....	377
		Separating Oleaginous or Fatty Matters from Boiler-Feed or other. (I) Weir.....	504
		Softening and Purifying, and Apparatus therefor. (P) Doulton and Manger.....	832
		The Bacteriological Analysis of. Part 1. (Richardson).....	1157
		The Use of Lead Pipes for Conveying. (Lecoq).....	547
		Waterproof Cloth, Manufacture of India-rubber. (Weber).....	33
		Fabrics, Manufacture of. (P) Abbott and Wilks.....	248
		Fabrics, Manufacture of Coloured. (P) Birnbaum.....	728
		Fabrics, Producing Designs on. (P) Frankenburg and Weber.....	725
		Waterproofing Material, Use of "Albano" as a. (P) Renison.....	648
		Of Materials, The. (Foerster).....	944
		Waterproofing Solution. (P) Hendrichs.....	1192
		Waters, Colorimetric Estimation of Iron in Spring. (Gerhard).....	546
		Discrimination in Drinking. (Vedroli).....	897
		Estimation of Nitrates and Nitrites in. (Fraser).....	1095
		German Trade in Mineral. (T.R.).....	1111
		Hernite's Process for Purifying, by Electricity.....	898
		Means for Purifying Effluent Waste. (P) Pughley.....	957
		Normal Chlorine in Spring. (Haines).....	828
		Of Coal Mines, On the Composition of. (König).....	1220
		On the Suitability of, for Dietetic Purposes. (Tatlock and Thomson).....	497
		Purification and Filtration of Foul. (P) Sharples.....	656
		Purifying and Disinfecting Foul. (P) Hope.....	831
		Report of French Committee on Natural Mineral. (T.R.).....	850
		Treatment of Waste. (P) Jarmain.....	832
		Wax, Analysis of Smelling. (Mangold).....	1100
		Carnauba. (Villon).....	744
		West Indies, Lime Cultivation in the. (T.R.).....	849
		Whalebone, Artificial. (P) Tourrette.....	657
		Manufacture of Artificial, and Apparatus therefor. (P) Levy, Rustin, and Boyer.....	1074
		Production of Artificial. (P) Fehrenbach and Prud'hon.....	726
		Production of Substitute for. (P) Hartmann.....	1075

Walter China Mosaic, Production of. (P) Bale.....	519
Wall Paper. See under Papers.	
Walls, Glazing and Embellishing. (P) Martin.....	40



	PAGE
Wheat Germ, Occurrence of Radinose in. (Schulze and Frankfurt).....	653
Whey, Milk, and Skim Milk, Their Comparative Composition and Sp. Gr. (Cochran).....	269
White Lead, German Trade in. (T.R.).....	1115
-Lead, Manufacture of. (P) De Ferranti and Nodt.....	532
-Lead, Manufacture of. (P) Nodt and De Ferranti. 1071, 1072	
-Lead, Manufacture of. (P) Waller.....	163
-Lead, Manufacture of. (P) Walton.....	163, 163
-Lead, Production of. (P) Haddon. From La Comp. Electro-Chim. de St. Bérn.....	886
Whisky, Influence of Materials used and Processes of Manufacture on Flavour of. (Nettleton).....	654
Wicks, Method of Washing Standard Sperm Candle.....	66
Wine, Chemical Action of Abrastol on. (Scheurer-Kestner).....	654
Compositions for Preserving. (P) Pickles.....	1083
Detection of "Abrastol" in. (Sangle-Ferrière).....	177
Detection of Boric Acid in the Ash of. (Künzsch).....	304
Detection of the Higher Alcohols in Spirits of. (Bardy).....	287
Diseases of. (Knyser).....	750
Employment of Selected Yeasts in Production of. (Fabre).....	1213
Method and Apparatus for Collecting Carbonic Acid during Fermentation of. (P) Michel and Frank.....	655
On the "Cassage" of. (Bonfard).....	654
Supposed occurrence of Free Tartaric Acid in. (Carpene).....	1080
Wines. (Class XVII.) 52, 167, 265, 410, 533, 653, 730, 824, 895, 966, 1077, 1211.....	826
Aeration of. (P) Fenn and Abraham.....	969
Alteration of the Sulphurous Acid in. (Chuard and Jaccard).....	1029
Detection of Abrastol in. (Bründl).....	548
Detection of Abrastol in. (Simbaldi).....	906
Detection of Coal-Tar Dyes in. (Sosterni and Carpenteri).....	286
Determination of Malic Acid in. (Mieke).....	750
Formation of Mannite in. (Molbot).....	52
Influence of Acidity in Worts on Composition of Low. (Lindet).....	998
Methods of Estimating Free Tartaric Acid in. (Carpene).....	531
Preservation of, by means of $\beta$ -Naphthol Sulphonic Acid. (Simbaldi).....	333
Preservation of, by means of Sodium Silicofluoride. (Surre).....	1230
Removal of Lead from, after Clarification. (Borntrager).....	526
Wire, Alloys for Manufacture of. (P) Macdonald.....	929
Gauge, The Standard.....	545
Wires in Glass, The Sealing of. (P) Lake. From Tibbits.....	352
Wong-koudou, Tinctorial Properties of. (Hummel and Perkin).....	170
Wood, Brown Paper from.....	1188
-Oil, New Constituents of. (Looff).....	949
Preservation of. (P) Groche.....	154
Preservation of. (P) Hely.....	628
Process for Preserving, and obtaining Products of Distillation. (P) Grumbacher.....	674
-Pulp Industry in Norway, The. (T.R.).....	170
-Pulp, Treating the Black Ash Residue of. (P) Langville.....	117
-Pulps, On the Estimation of Moisture in. (Carter Bell).....	380
The Carbonisation of. (Sartiz).....	285
-Vinegar, Rectified. (Brandes).....	285
-Vinegar, Tests of Purity of Rectified. (Buchner).....	1093
Vulcanising and Drying. (P) Howard.....	886
Woods of the Coniferae, Behaviour of, to Solutions of the Chlorides of Copper. (Jensch).....	802
Wool. (Class V.)..... 32, 146, 248, 384, 512, 652, 725, 802, 880, 944, 1056, 1192.....	803
Apparatus for Scouring and Washing. (P) McNaught.....	802
Apparatus for Washing. (P) Dawson.....	945
Carbonisation of. (P) Schull.....	388
Dyeing Light Shades on. (Schmabel).....	884
Dyeing, with Chromium Lakes. (P) Hoffman.....	633
Dyeing, with Fast Colours. (P) Abel. From The Farb. vorm. Meister, Lucius and Brünning.....	962
Extraction of Suint from. (P) Richard-Lagerie.....	1068
-Fat, the Examination of. (Herbig).....	1100
-Fat, Valuation of. (Von Cochenhausen).....	162
-Fats, Separation of Cholesterin and Fatty Acids from. (P) Krause and Mayer.....	727
Formation of Resorcinol-Green on. (Pokorny).....	529
-Grease, Crude, and Lanolin.....	260
-Greases, Treatment of, for Separation of their Constituents. (P) Hutclinson.....	34
Method and Apparatus for Washing. (P) Kelly.....	222, 356
On the Mordanting of, with Chromium. Parts III. and IV. (Liechti and Hummel).....	880
Process and Apparatus for Scouring. (P) Heys. From Malard.....	884
Process for Dyeing. (P) Imray. From The Farb. vorm. Meister, Lucius und Brünning.....	728
Scouring, Bleaching, and Disinfecting. (P) Hughes.....	633, 944
Scouring, Dyeing, and Cleansing, and Apparatus therefor. (P) Turay.....	1057
The Chlorinating of. (Bulard).....	258
Woollen Goods, Soaps most useful for Scouring. (Lewkowsch).....	37
Wormseed, Occurrence of Betaine and Choline in. (Jahns).....	267
Wort and Hops, Evolution of Carbon Dioxide and Hydrogen Sulphide in Boiling. (Elion).....	655
Apparatus for Aerating or Raising. (P) Stone.....	967
"Gluten Particles" occurring in. (Will).....	

## Wort—cont.

Influence of Hops on. (Aubry).....	411, 1077
Quantitative Separation of Amorphous Nitrogenous Organic Compounds contained in. (S. Jerning).....	1169
Quantity of Steam and Boiler Capacity Required in Washing and Boiling.....	1089
Relation of Hops to. (Aubry).....	895
Saccharification and Treatment of, and Apparatus therefor. (P) De Geyler (illus.).....	536
The Oxidation of Beer. (Pédro).....	1079, 1079
Worts, Influence of Vidity in, on Composition of Low Wines. (Lindet).....	5
Influence of Cane Sugar on Valuation of. (P).....	1080
The Vinumtoris contained in. (Wahl and Hantke).....	899
Writing and Drawing Materials, German Trade in. (T.R.).....	1114

## X

Xanthine Bodies, On the. (Balke).....	284
Xanthorrhiza Resins, On. (Bamberger).....	47
Xylenol, Volumetric Estimation of. (Keppler).....	1099

## Y

Yarn, Apparatus for Dyeing and Washing. (P) Rhodes and Perkins.....	390
Apparatus for Gassing. (P) Clouch.....	880
Apparatus for Sizing. (P) Maaz.....	886
Dyeing Machines. (P) Haslam.....	391, 391
Machines for Dyeing and Sizing. (P) Collins.....	399
Yarns, Apparatus for Imparting Silky Appearance to. (P) Jacob.....	944
Bleaching or Cleansing Vegetable. (P) Riley and The Thornelohank Co. ....	728
Dyeing Certain Colours on Cotton. (P) Hay and Park.....	391
Dyeing, Mordanting, and Treating. (P) Redfitt.....	513
Dyeing Turkey-Red and Rose Colour in. (P) Thompson. From Haeffely.....	513
Dyeing Dyed Cotton. (P) Thompson. From Salzuann.....	947
Machine for Loosening and Dyeing. (P) Blass.....	947
Process for Dyeing Vegetable. (P) Thompson. From Holken and Co. ....	391
See also Textiles.	
Yeast, Action of Certain Antiseptics on. (Mann).....	1160
Action of Oxygen on. (Van Laer).....	1078
Capacity of, to Live in Media containing Hydrofluoric Acid. (Sarel).....	653
Cells, Phenomena accompanying Dialysis of Beer. (Onimus).....	1215
Cells, Specific Character of Fermentative Functions of. (Brown).....	824
Chemical Properties of the Alcoholic Extract of Beer. (De Rey-Pailhade).....	751
Composition and Analysis of. (Guichard).....	844
Culture, Hansen's System of Pure, in Top Fermentation. (Jørgensen).....	826
Cultures, Behaviour of some Sugars towards Pure. (Fischer and Thierfelder).....	1211
Determination of Starch in Pressed. (Filsinger).....	908
"Gluten Particles" occurring in. (Will).....	967
Gum. (Salkowski).....	1213
Influence of Certain Chemical Conditions on Activity of Brewers'. (Effront).....	267
Influence of Compounds of Fluorine on Beer. (Effront).....	1079
Influence of Fluorine Compounds on Beer.....	968
Manufacture of. (P) Polsky.....	55, 268
Manufacture of. (P) Squire.....	1082
Manufacture of, and Distillation of Spirits. (P) Lederer.....	827
Method of Utilising and Means for Treating. (P) Greening.....	1082
Preservation of, for Transport. (P) Ieron.....	168
Pure Cultivation, and Use of Antiseptics in Distilling. (Chuss).....	595
Studies on. (Elion).....	266
The Carbohydrates of. (Salkowski).....	111
Treatment of. (P) Cave.....	1214
Yeasts, Contribution to the Study of. Hantke-Hille and Perrey.....	654
Employment of Selected, in Wine Production. (Fabre).....	1213
Ylang-Ylang, Essence of. (Reychler).....	758, 1083
Yonker and Co.'s Brewery, Visit to Messrs. ....	709

## Z

	PAGE
Zelutna, Manufacture and Refining of Carbon Disulphide at, (Farbaky, Illinois) .....	808
Zinc and Lead Pigments or Compounds, Manufacture of. (P) Chaudier .....	532
Acid of, Separation of. (Jannasch and Lomsky) .....	67
Acid Magnesium, Action of, on Metallic Solutions. (Vilhiers and Forz) .....	68
Cadmium, &c., Furnaces for Distilling. (P) Golby, From Francelet .....	402
Dust, Method and Apparatus for Analysis of. (Meyer) Illinois .....	841
Dust, Valuation of. (Bach) .....	765
Electrodeposition of. (P) Cowper-Coles and Walker .....	1067
Formicamide as a Fixing Agent. (Oswald) .....	1193

## Zinc—cont.

	PAGE
Lead Process, The Bartlett. (Hawker) .....	41
Muffles, Formation of Cast-Iron in. (Jensch) .....	398
Ores and Products, The Testing of. (Meyer) .....	1225
Oxide, Manufacture of. (P) Gray .....	401
Pigments, Preparation of Oxide of. (P) Laurie .....	262
Plates, Treatment of Lithographic. (P) Forrest and Bucholz .....	527
Preparing Solutions carrying Salts of. (P) Choate .....	335
Process and Apparatus for Extraction of. (P) Lynen .....	402
Produced by Electrolysis, Treatment of. (P) Wright and Ramond .....	1067
Producing Metallic. (P) Choate .....	741
Production of, in Germany. (T.R.) .....	74
Recovery of, from Waste Galvanised Metal. (P) Hayward .....	401
Resist for Silk. (Koschlin) .....	727
Smelting at the Bertha Works, Virginia. (Moxham) .....	41
Smelting in the Blast Furnace, Hempel's Experiments on. White, German Trade in. (T.R.) .....	814
	1115

## INDEX OF NEW BOOKS.

	PAGE		PAGE
Analyses of Twelve Thousand Prescriptions. W. Martindale . . . . .	768	Laboratory Guide, The. A. H. Church . . . . .	769
Alchemical Essence and the Chemical Element, The. M. M. Pattison Muir . . . . .	289	Law and Theory in Chemistry. D. Carnegie . . . . .	549
Chemiker-Kalendar, 1895. Dr. R. Biedermann . . . . .	1107	Micro-Organisms in Water. Percy Frankland and Mrs. Percy Frankland . . . . .	671
Chemisch-Technisches Repertorium. Dr. E. Jacobsen 182, 845, 1232		Mineral Resources of the United States, Calendar Year 1893. D. T. Day . . . . .	846
Descriptive Catalogue of Essential Oils and Organic Chemical Preparations. F. B. Power . . . . .	989	Nature's Hygiene. C. T. Kingzett . . . . .	768
Dictionary of the Active Principles of Plants: Alkaloids; Bitter Principles; Glucosides: Their Sources, Nature, and Chemical Characteristics. C. E. Sohn . . . . .	72	Odorographia: A Natural History of the Raw Materials and Drugs used in the Perfume Industry. J. Ch. Sawyer . . . . .	429
Die Chemische Industrie auf der Columbianischen Weltausstellung zu Chicago und in den Vereinigten Staaten von Nord America im Jahre 1893. Dr. Bosse, erstattet von Dr. O. N. Witt . . . . .	768	Practical Paper-Making. G. Clapperton . . . . .	549
Die Fabrikation von Schwefelsaurer Thonerde. Dr. K. W. Jurisch . . . . .	1108	Principles and Practice of Agricultural Analysis. Harvey W. Wiley . . . . .	289
Die Maschinellen Hilfsmittel der Chemischen Technik. A. Parnicke . . . . .	1108	Rasa-Ranga-Rahasya. M. G. Deshmukh and T. K. Gajjar . . . . .	988
Die Selbstentzündung von Schiffsladungen, Baumwolle, und anderen Faserstoffen, Steinkohlen, Heuhaufen, Tabak, &c. Dr. L. Hapke . . . . .	289	Rise and Development of Organic Chemistry, The. C. Scharlemmer. Edited by A. Smithells . . . . .	1106
Einführung in das Studium der Qualitativen Chemischen Analyse. C. Friedheim . . . . .	1107	Select Methods in Chemical Analysis (Chiefly Inorganic). W. Crookes . . . . .	845
Engineering Chemistry. H. J. Phillips . . . . .	549	Systematic Survey of the Organic Colouring Matters. G. Schultz and P. Julius . . . . .	671
Gas Engineer's Laboratory Handbook, The. J. Hornby . . . . .	429	Tabellarische Uebersicht über die Künstlichen Organischen Farbstoffe, &c. Dr. Adolf Lehner . . . . .	73, 429, 768, 1231
Grundriss der Pharmakognosie. F. A. Flückiger . . . . .	429	Tannins, The. H. Trimble . . . . .	559
Handbook of Gold Milling. A. H. Louis . . . . .	429	Technisch-Chemisches Jahrbuch, 1892-1893. Dr. R. Biedermann . . . . .	845
Handbuch der Anorganischen Chemie. Dr. O. Dammer . . . . .	1231	Teinture et Impression. M. Prud'homme . . . . .	429
Handwörterbuch der Pharmacie. A. Brestowski . . . . . 182, 768, 989		Treatise on Chemistry. A. Sir H. E. Rose and C. Scharlemmer. Vol. I. The Non-Metallic Elements. New Edition. Revised by Sir H. E. Rose, H. G. Colman, and A. Harden . . . . .	1107
Jahrbuch der Chemie. R. Meyer . . . . .	846	Universal Exposition at Paris, 1889. Report on Chemical and Pharmaceutical Products. S. B. Newbury . . . . .	289
Jahresbericht über die Leistungen der Chemischen Technologie, mit besonderer Berücksichtigung der Gewerbestatistik für das Jahr 1893. Dr. F. Fischer . . . . .	671	Water Analysis. J. A. Wanklyn and E. T. Chapman. Ninth Edition. J. A. Wanklyn . . . . .	768
Jahres-Rundschau über die Chemische Industrie und deren wirtschaftliche Verhältnisse für das Jahr 1893. Ein übersichtlich geordneter Bericht über die Fortschritte der Chemischen Gross- und Klein-Industrie, &c. Dr. A. Bender . . . . .	671	Watts' Dictionary of Chemistry. Revised and Re-written by M. M. Pattison Muir and H. Forster Morley . . . . .	989

# LIST OF ERRATA FOR 1894.

Page.	Column.	Line.	Description.
52	2	41	For "Tannet" read "Tannet."
62	1	28	From top: for "phosphorous" read "phosphorus."
165	2	16	From bottom: for "Edemann" read "Telemann."
166	1	27	From top: for "Mittelmann" read "Mittelmann."
171	1	37	From top: for "pyrozollol" read "pyrozollol."
255	1	24	From top: for "slaxwood" read "slagwood."
428	1	53	From top: for "techn" read "techn."
428	1	22	From bottom: for "chromoscope" read "chronoscope."
428	1	26	From bottom: for "chromoscope" read "chronoscope."
652	1	2	From top: insert "Gis."
760	..	..	The third Abstract from top should be under "XXIII., Apparatus, &c."
780	2	31	From bottom: for "connection" read "convection."
780	2	13	From bottom: for "when" read "where."
818	2	17	From bottom: for "Bewitt" read "Hewett."
820	2	17	From bottom: for "Gentzsch" read "Gentzsch."
823	1	9	From top: for "60" read "69° R."
824	1	20	From top: for "degration" read "degradation."
824	1	24	From top: for "Prinsen Gurlugs" read "Prinsen Geerlugs."
842	2	10	From top: for "Trommedorffs" read "Trommsdorffs."
846	1	23	From top: for "1894" read "1895."
891	2	24	From bottom: for "as" read "whilst."
945	2	9	From top: for "Georgievics" read "Georgievics."
965	2	17	From top: for "Mittelman" read "Mittelmann."
1026	..	..	Morzan's Tabular Scheme for Iron Ore Analysis, col. 1, under sub-heading <i>Reagents</i> : for "HCl" read "HF."

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 Bruce, Geo., 11, New Gravel Lane, Shadwell, E.  
 Bruce, Jas., The Irish Distillery, Connswater, Belfast, Ireland.  
 Bruckmann, G. T., 192, 18th Street, Brooklyn, N.Y., U.S.A.  
 Brunner, H., Holly Mount, Tarbock Road, Huyton, near Liverpool.  
 Brunner, J. F. L., Winnington Old Hall, Northwich, Cheshire.  
 Brunner, J. T., M.P., Druid's Cross, Wavertree, Liverpool.  
 Brunner, Dr. P., 29, Fenney Street, Higher Broughton, Manchester.



- Brunton, J. Dixon, Wire Mill, Musselburgh, N.B.  
 Bryce, John Annan, Messrs. Wallace Bros., 8, Austin Friars, London, E.C.  
 Bryce-Smith, N. J., Oakfield, Barrow, Whalley, near Blackburn.  
 Buchanan, D. G., Mount Vernon House, Glasgow.  
 Buchanan, Jas., jun., Caledonia Foundry, Brasenose Road, Liverpool.  
 Buchanan, Joshua, 63, West Cumberland Street, Glasgow.  
 Buckley, Edwin, Beaver Park, Didsbury, Manchester.  
 Budden, E. R., 11, Furnival Street, Holborn, E.C.  
 Bull, Johannes C., Erith, Kent.  
 Bullock, J. L., 3, Hanover Street, Hanover Square, London, W.  
 Bumby, H., c/o Millom and Askam Hematite Iron Co. Ltd., Askam-in-Furness, Lancashire.  
 Bunker, H. E., 1, Deanley Terrace, St. Mary's Road, Newton Heath, Manchester.  
 Bunting, W. Lightfoot, c/o F. W. Ashton & Co., Hyde, near Manchester.  
 Burbridge, Jas., India-rubber Mills, Tottenham, N.  
 Burdekin, G., jun., Sutton Lodge Works, St. Helens.  
 Bürger, Dr. J., 1, Birch Avenue, Talbot Road, Old Trafford, Manchester.  
 Burgess, Geo., Golding Davis Works, Widnes.  
 Burgess, Herb. E., 144, Warwick Street, Eccleston Square, London, S.W.  
 Burgess, Wm. T., 1, Ringley Cottages, Reigate, Surrey.  
 Burchardt, Dr. C. A., 35, Fountain Street, Manchester; and Owens College, Manchester.  
 Burnard, R., Plymouth Chemical Works, Plymouth.  
 Burnett, Henry K., North Brook Vitriol Works, Bradford, Yorks.  
 Burnham, J. C., 24, St. Stephen's Road, Lewisham, S.E.  
 Burn-Murdoch, J. V., Elm-tree House, Pembroke, South Wales.  
 Burrell, B. A., 5, Mount Preston, Leeds.  
 Burrough, Horace, jun., 1130 Lafayette Avenue, Baltimore, Md., U.S.A.  
 Burroughs, S. M., Snow Hill Buildings, Holborn Viaduct, E.C.  
 Burrows, Edw., Belle Vue Road, Low Fell, Gateshead-on-Tyne.  
 Burton, F., 2, Green Street, Bethnal Green, E.  
 Burton, Wm., Clifton Junction, near Manchester.  
 Bury, J. H., Church Chemical Works, near Accrington.  
 Bush, R. A., 20, Artillery Lane, London, E.C.  
 Bush, Baron W. de, 20, Artillery Lane, London, E.C.  
 Butler, Paul, Lowell, Mass., U.S.A.  
 Butler, Samuel, Compton, Wolverhampton.  
 Butler, W. W., The Cedars, Duchess Road, Edgbaston, Birmingham.  
 Butterfield, J. C., 13, Victoria Street, Westminster, S.W.  
 Butterfield, W. J. A., Beckton, E.  
 Butt, E. N., 77, Hamilton Terrace, Maida Vale, W.  
 Buttfeld, Horace V., 16, Thistlewaite Road, Clapton, N.E.  
 Byard, A. G., c/o Burt, Boulton, and Heywood, Apartado 8, Bilbao, Spain.  
 Byron, T. H., Laboratory, Wigan Coal and Iron Co., Wigan.  
 Bythway, M., 43, Lloyd Street, Albert Street, Manchester.
- C**
- Cabot, Godfrey L., 82, Water Street, Boston, Mass., U.S.A.  
 Cadett, Jas., Ashted, Surrey.  
 Caines, Chas. M., 7, Rochester Terrace, Camden Road, London, N.W.  
 Caines, G. S. A., 7, Rochester Terrace, Camden Road, London, N.W.  
 Caldecott, Wm. A., Eureka City, Barberton, S.A.R.  
 Calderwood, J., Gowanlea, Spence Park, Wandsworth, S.W.; and Price's Patent Candle Co., Battersea.  
 Caldwell, Alfred S., Greenfield Lodge, Lasswade, Midlothian, N.B.  
 Caldwell, Wm., Murray Street, Paisley, N.B.  
 Caley, A. J., Chapel Field, Norwich.  
 Catkin, Wm. S., Johnsonburg Quay P.O., Pa., U.S.A.  
 Cameron, Jas., c/o Nobel's Explosives Co., Ltd., Polmont Station, N.B.  
 Cameron, Peter, Bath Bridge Colour Works, Bristol.  
 Cameron, R., Wellpark House, Bathgate, N.B.  
 Cammack, J., 8, Salisbury Street, St. Helens.  
 Campbell, Andrew, c/o Burnah Oil Co., Ltd., Rangoon, Burmah.  
 Campbell, Archibald, 42, Whittaker Road, Upton Park, E.  
 Campbell, Colin M., c/o Long & Robertson, Habana, Mackay, Queensland.  
 Campbell, John, 118, Warren Street, New York City, U.S.A.  
 Campbell, Jno. D., Wynton Lodge, Chatsworth Road, West Norwood, S.E.  
 Candlish, J. J., Bottle Works, Seaham Harbour, co. Durham.  
 Candy, F. Pullen, 56, Nightingale Lane, Balham, S.W.  
 Cannon, J. C., 70, Ham Park Road, Forest Gate, E.  
 Cannon, M., Beaufoy's Chemical Works, Lavender Hill, London, S.W.  
 Canziani, Enrico, 3, Palace Green, Kensington, W.  
 Carden, Albert J., Lea Valley Distillery, Warton Road, Stratford, E.  
 Carey, Arthur, Browside, Gateacre, near Liverpool.  
 Carey, E., Browside, Gateacre, near Liverpool.  
 Cargay, W. E., c/o Sclopis and Co., 31, Via Aosta, Torino, Italy.  
 Carlile, T., 23, West Nile Street, Glasgow.  
 Carmichael, H., 12, Post Office Square, Boston, Mass., U.S.A.  
 Carmody, Prof. Patrick, Government Laboratory, Port of Spain, Trinidad.  
 Caro, Dr. H., Mannheim, Germany.  
 Carpenter, C. C., Gas Works, Bridge Foot, Vauxhall, S.E.  
 Carpenter, H. S., Beckingham House, Weighton Road, Annerley, S.E.  
 Carpenter, R. F., Bent Terrace, Prestwich, near Manchester.  
 Carruthers, J. G., Burnbrae House, Milngavie, N.B.  
 Carswell, Thos. R., Day House, Day Street, Newcastle-on-Tyne.  
 Cartelghe, M., 180, New Bond Street, London, W.  
 Carter, W. C., 5, Abercorn Street, Lisburn Road, Belfast, Ireland.  
 Carulla, F. J. R., 84, Argyll Terrace, Derby.  
 Carulla, F. M., Apartado 689, Buenos Ayres, Argentina; and (Journals) 84, Argyll Terrace, Derby.  
 Castner, Hamilton Y., c/o The Aluminium Co., Ltd., Oldbury, Birmingham.  
 Cawley, G., 29, Great George Street, Westminster, S.W.  
 Cawley, J., 278, Passaic Street, Newark, N.J., U.S.A.  
 Chadwick, L. N., Ivy Lawn, Ponders End, Middlesex.  
 Chadwick, Walter M., 24, West 3rd Street, Bayonne, N.J., U.S.A.  
 Chaloner, G., 30, Weston Park, Crouch End, N.  
 Chance, A. M., Lawnside, Edgbaston, Birmingham.  
 Chance, J. F., 51, Prince's Gate, London, S.W.  
 Chandler, Dr. C. F., School of Mines, Columbia College, New York.  
 Chaney, Harry, 29, Chalcot Crescent, Regent's Park, N.W.  
 Chaplin, Edw. M., 60, Westgate, Wakefield, Yorks.  
 Chapman, Alf. C., 23, Euston Buildings, Gower Street Station, London, N.W.  
 Chapman, S., 84, Eccleston Square, London, S.W.; and 36, Mark Lane, E.C.  
 Charlier, Andrew, 107, Breadallbane Terrace, Hill Street, Glasgow.  
 Chase, R. L., 155, East Main Street, North Adams, Mass., U.S.A.  
 Chatard, Dr. T. M., 1410 G. Street, Washington, D.C., U.S.A.  
 Chattaway, Wm., 23, Leadenhall Street, E.C.  
 Cheney, J. P., c/o Cheney Bros., South Manchester, Conn., U.S.A.  
 Cheyne, A. M., c/o Messrs. Burgoyne, 16, Coleman St., E.C.  
 Cholerton, A. F., Lyndum House, Lincoln Street, Leicester.  
 Chope, J. A., The Laboratory, Somerset House, London, W.C.  
 Chorley, Jno. C., 85, Haverstock Hill, N.W.  
 Christie, J., Levenfield, Alexandria, N.B.  
 Christy, Thos., Malvern House, Sydenham, S.E.; and 25, Lime Street, London, E.C.

- Chrysal, W. J., Shawfield Works, Rutherglen, near Glasgow.  
 Church, Prof. A. H., F.R.S., Shelsley, Kew, Surrey.  
 Church, Elihu D., jun., c/o Church and Co., 36, Ash Street, Brooklyn, N.Y., U.S.A.  
 Clananahan, H. C., 88, King Street, Manchester.  
 Clapp, Ralph R., c/o Standard Ammonia Co., Ltd., Iceland Wharf, Old Ford, E.  
 Clapperton, J., jun., 25, Queen Square, Regent Park, Glasgow.  
 Clark, Durand G., 14, St. John's Wood Park, N.W.  
 Clark, Franklin S., 327, Madeira Avenue, New York City, U.S.A.; and (Journals) Southern Chemical Works, Wilmington, N.C., U.S.A.  
 Clark, Dr. J., 138, Bath Street, Glasgow.  
 Clark, John, 80, Great Brook Street, Birmingham.  
 Clark, R. Ingham, 3, Albert Hall Mansions, Kensington Gore, W.  
 Clarke, Arthur, St. Ann's Hill Factory, Nottingham.  
 Clarke, C. Goddard, Ingleside, Elm Grove, Peckham, S.E.  
 Claret, A. C., 6, Coleman Street, London, E.C.; and (Journals to) 9, Dalcham Gardens, Hampstead, N.W.  
 Claudet, F. G., 6, Coleman Street, E.C.; and (Journals) 3, Manor Rise Terrace, Dulwich, S.E.  
 Claus, C., c/o Francis Arding, 5, Jeffrey Square, London, E.C.  
 Claus, Carl P., jun., Wannarwydd House, Gowerton, South Wales.  
 Claus, Wm. H., 24, Egerton Road, Fallowfield, Manchester.  
 Claypole, Dr. E. W., Buchtel College, Akron, Ohio, U.S.A.  
 Clayton, E. G., Chemical Laboratory, 43 & 44, Holborn Viaduct, London, E.C.  
 Clayton, J. W., Bentfield, Alma Road, Aigburth, Liverpool.  
 Clayton, Robt. H., 762, Roehdale Road, Manchester.  
 Clemes, J. H., Cheriton, Chelston, Torquay.  
 Clemmshaw, E., Alkali Works, Oldbury, near Birmingham.  
 Clemons, G. H., Cudbear Street, Hunslet Road, Leeds.  
 Clerk, Dugald, Driffield Villa, Sutton Coldfield, near Birmingham.  
 Cliff, J., Nisbet Hall, Fulneck, Leeds.  
 Cliff, Stephen, Wortley, near Leeds.  
 Clift, J., Tar Works, Knottingley, Yorks.  
 Clifton, C. D., Royal Oak Brewery, Stockport.  
 Cloud, T. C., Wallaroo Smelting Works, Wallaroo, South Australia.  
 Clowes, Dr. F., University College, Nottingham.  
 Clowes, G. A., Needham Market, Suffolk.  
 Clutton, J. H., Goring Villas, Burry Port, R.S.O., Carmarthenshire.  
 Coats, Jno. T., 91, Broughton Street, Edinburgh.  
 Cobb, Jno. W., Farnley Iron Co., near Leeds.  
 Cochrane, Chas., Green Royle, Pedmore, near Stourbridge.  
 Cockrell, E. Gordon, 17, Grosvenor Chambers, Manchester.  
 Coffey, Eneas H., Hatherley Distillery, Pretoria, S.A.R.  
 Coghill, P. de G., Borax Works, Old Swan, Liverpool.  
 Cogwell, W. B., Syracuse, New York, U.S.A.  
 Cohen, Dr. J., Yorkshire College, Leeds.  
 Colby, Albert L., 131, South Center Street, Bethlehem, Pa., U.S.A.  
 Colby, W. H., Carreg-wen, Aberystwith.  
 Colefax, Dr. Arthur, 26, Ashgrove, Bradford, Yorks.  
 Coleman, Jas. B., Burley House, Carlton, Nottingham.  
 Coleman, W. H., 9, Lloyd Road, East Ham, Essex.  
 Collin, Theodore F., P.O. Box 393, Pittsburgh, Pa., U.S.A.  
 Collens, E., Vinegar Works, Stourport, Worcestershire.  
 Collett, J. M., Guy's Cliff, Wotton, Gloucester.  
 Collin, Dr. C. A., Ferguslie Threadworks, Paisley, N.B.  
 Collins, H. S., c/o General Apothecaries' Co., 49, Berners Street, W.  
 Collins, J. H., 14—15, Broad Street Avenue, London, E.C.  
 Collins, W., Hepworth, 14 & 15, Bradford Buildings, Mawdley Street, Bolton-le-Moors.  
 Collins, C. S. A., c/o Mrs. Stevens, 56, Elm Row, Leith Walk, Edinburgh.  
 Colman, Dr. H. G., 29, Noel Road, Edgbaston, Birmingham.  
 Colquhoun, D., Maulesbank, Carnoustie, N.B.  
 Colquhoun, Lewis, Ardeer Ironworks, Steveston, Ayrshire, N.B.  
 Colson, A., Gas Office, Millstone Lane, Leicester.  
 Colwell, Walter E., c/o B. P. Clapp Ammonia Co., Cincinnati, Ohio, U.S.A.  
 Comer, H., P.O. Box 31, Jersey City, N.Y., U.S.A.  
 Connor, C. C., M.P., Notting Hill House, Belfast, Ireland.  
 Conrad, E. C., Portland Road, Gravesend.  
 Conradson, Pontus H., Great Northern Railroad, St. Paul, Minn., U.S.A.  
 Conroy, Jas. T., Woodstock, New Brighton, Cheshire.  
 Constable, W. H., Hale Bank, Widnes, Lancashire.  
 Coode, J. Charles, 19, Freeland Road, Ealing, W.  
 Cook, E. Rider, East London Soap Works, Bow, London, E.  
 Cook, H. J., East London Soap Works, Bow, London, E.  
 Cook, Jas. Williams, 30, Bury Street, St. Mary Axe, E.C.  
 Cook, Jno. J., Atlas Foundry, St. Helens, Lancashire.  
 Cooke, Arthur W., c/o Messrs. Brotherton & Co., Calder Vale, Wakefield.  
 Cookson, Jas. H., Stauley, near Wakefield.  
 Cookson, N. T., Newcastle-on-Tyne.  
 Coomber, Thos., 40, Clarendon Road, Redland, Bristol.  
 Cooper, A., North-Eastern Steel Co., Middlesbrough-on-Tees; and (Journals) Erdely, Middlesbrough.  
 Cooper, H. P., 2, New North Road, Hoxton, E.  
 Cooper, Harry J., Drinagh, Wexford, Ireland.  
 Cooper, Walter J., c/o South Wales Cement Co., Penarth, Cardiff.  
 Corbett, Jno., M.P., Impney, Droitwich, Worcestershire.  
 Corbould, Wm. H., Ediaara Consols Silver Mine, Beltana, South Australia.  
 Coreoran, Bryan, 31, Mark Lane, London, E.C.  
 Cordner-James, J. H., 14, Sherborne Lane, King William Street, London, E.C.  
 Cornett, Jas. P., Ford Paper Works, Hylton, near Sunderland.  
 Cornish, Vaughan, 1, St. James' Terrace, Winchester.  
 Corputaau, J., 6, Cliff Villas, Toller Lane, Bradford, Yorks.  
 Corrie, David, c/o Nobel's Explosives Co., Ltd., Polmont Station, N.B.  
 Coste, F. H. Perry, 7, Fowkes Buildings, Great Tower Street, London, E.C.  
 Cottam, J. C., 39, Lombard Street, London, E.C.  
 Cotterill, Thos., The Poplars, West Bromwich.  
 Cotton, W. F., Hollywood Roebuck, Co. Dublin.  
 Couche, Chas. W., c/o United Alkali Co., Ltd., Weston Works, near Runcorn.  
 Cowan, W. J., 77, Trinity Road, Wood Green, N.  
 Cowley, A. J., 13, Fenchurch Avenue, London, E.C.  
 Cowper, C. E., 6, Great George Street, Westminster, S.W.  
 Cowper-Coles, Sherard Osborn, 16, Adam Street, Manchester Square, W.  
 Coxe, Eekley B., Drifton, Luzerne Co., Pa., U.S.A.  
 Crabh, W., Border Counties Chemical and Manure Works, Silloth, Cumberland.  
 Craig, G., Lugar Iron Works, Camnock, N.B.  
 Crake, Wm., Dresden Terrace, Bawtry Road, Attercliffe, Sheffield.  
 Craven, Chas. E., Hawthorne Cottage, White Cote Hill, Bramley, near Leeds.  
 Craven, Jno., jun., Smedley Lodge, Cheetham, Manchester.  
 Craw, John, 15, Cadogan Street, Glasgow.  
 Crawford, D., Langdale's Manure Works, Newcastle-on-Tyne.  
 Crawford, D., Kersal Vale Works, Higher Broughton, Manchester.  
 Crawley, Arthur H., c/o Elmore's Patent Copper Depositing Co., Ltd., Pontefract Road, Hunslet, Leeds.  
 Crawshaw, E., 25, Tollington Park, London, N.  
 Crenshaw, S. D., Crenshaw Building, Richmond, Va., U.S.A.  
 Cresswell, C. G., Ermyngarth, Ashted, Surrey; and 9, Bridge Street, Westminster, S.W.  
 Cresswell, C. N., 1, Hare Court, Temple, E.C.  
 Crichton, Donald G. (Journals), Nundle, via Tamworth, New South Wales; (subs.) Logan Bank, Cupar Fife, N.B.  
 Criper, Wm. R., Cossipore Chemical Works, Cossipore, Calcutta, India.  
 Crippin, Wm., The Hollies, Eccles, Manchester.  
 Critchley, C. A., Victoria Works, St. Helens, Lancashire.  
 Croft, Arthur, 10, Abbey Street, Old Lenton, Nottingham.  
 Croft, Joseph, 26, Beeston Road, Dunkirk, Old Lenton, Nottingham.

- Crompton, Percy R., Dearden's House, Bury, Lancashire.  
 Cronquist, A. Werner, Royal Wharf, Skeppsholmen, Stockholm, Sweden.  
 Crookes, W., F.R.S., 7, Kensington Park Gardens, Notting Hill, London, W.  
 Crosfield, A. L., Casino Ingles, Minas de Rio Tinto, Huelva, Spain.  
 Crosslaw, Jno. F., 11, Rose Street, Fountain Road, Hull.  
 Cross, C. F., 4, New Court, Lincoln's Inn, London, W.C.  
 Crossley, Dr. Arthur W., The Owens College, Manchester.  
 Crossley, L., 28, Parkfield Road, Sefton Park, Liverpool.  
 Crossman, Tom, Albion Brewery, Coldhurst Street, Oldham.  
 Crow, Dr. J. K., Hillingdon Varnish Works, West Drayton, Middlesex.  
 Crowder, W., 271, Evering Road, Upper Clifton, London, N.  
 Crowther, Edw., Woodland Dyeworks, Huddersley, Leeds.  
 Crowther, Horace W., 21, Beeches Road, West Bromwich.  
 Crowther, W. M., Field House, Gomersal, near Leeds.  
 Crumie, W. D., 146, Washington Street, East Orange, N.J., U.S.A.  
 Cullen, Wm., Ardler Terrace, Stevenston, Ayrshire, N.B.  
 Cuning, James, jun., Chemical Works, Yarraville, Melbourne, Australia.  
 Cumming, T. A., Craiglea, Causewayhead, near Stirling, N.B.  
 Cunliffe, E. T., The Parsonage, Handforth, near Manchester.  
 Cunliffe, Jos., Kern Mill, Whittle-le-Woods, near Chorley, Lancashire.  
 Cunningham, Edw., Milton, Mass., U.S.A.  
 Cunningham, H. D., 24, Rood Lane, London, E.C.  
 Curphey, W. S., 15, Bute Mansions, Hillhead, Glasgow.  
 Curry, W. A., Giltbrook Chemical Works, Awsworth, Notts.  
 Cuthbert, R. M., 27a, Ashley Place, London, S.W.  
 Cuthbertson, Sir J. N., 29, Bath Street, Glasgow.  
 Cuthbertson, William, Caroline Park, Edinburgh.
- D**
- Dacie, J. C., Soap Works, Putney, London, S.W.  
 Dagger, J. H. J., 5, Lorne Street, Fairfield, Liverpool.  
 Dale, Jas., 182, Lordship Road, Stoke Newington, N.  
 Dale, R. S., 1, Chester Terrace, Chester Road, Manchester.  
 Danglede, Ernest, Vevay, Indiana, U.S.A.  
 Daniell, Louis C. (Journals), Royal Standard Brewery, Tamworth, New South Wales; and (subs.) c/o W. T. Allen & Co., 132, Queen Victoria Street, London, E.C.  
 Darby, J. H., Pen-y-Garth, near Wrexham.  
 Darling, W. H., 126, Oxford Street, Manchester.  
 Davenport, Dr. B. F., 161, Tremont Street, Boston, Mass., U.S.A.  
 Davidson, J. E., 40, Percy Gardens, Tynemouth.  
 Davidou, R., c/o Fiji Sugar Co., Tamanna, Navua River, Fiji.  
 Davidson, Richard, 15, Lilybank Road, Dundee.  
 Davidson, R. H., c/o Messrs. Golding, Davis, & Co., Marsh Alkali Works, Widnes.  
 Davies, G. W., 8, Spring Hill, Stockport.  
 Davies, Meurig L., Mayfield, Norman Road, Runcorn.  
 Davies, W. E., Beaumaris, R.S.O., North Wales.  
 Davis, A. R., Dunowen, Knutsford, Cheshire.  
 Davis, Chas., 3, Leitrim Terrace, High Street, East Ham, E.  
 Davis, Fred., 26-28, Newington Causeway, London, S.E.  
 Davis, G. E., Kersal House, Higher Broughton, Manchester.  
 Davis, Herbert J., 255, West 71st Street, New York, U.S.A.  
 Davis, T. S., 199, South Lambeth Road, London, S.E.  
 Davis, W. Walley, jun., c/o Crozer Iron Co., Roanoke, Va., U.S.A.  
 Dawson, B., York House, Malvern Link, Worcestershire.  
 Dawson, C. A., Holly Bank, Frodsham, Cheshire.  
 Dawson, Jno., Silver Street Colour Works, Mold Green, Huddersfield.  
 Dawson, W. Haywood, British Alizarin Co., Limited, Silvertown, Victoria Dock, E.
- Deacon, H. W., Appleton House, Widnes.  
 Deakin, E., Belmont Bleach and Dyeworks, near Bolton.  
 Deakin, H., Ryecroft Dyeworks, Belmont, near Bolton.  
 Dearden, Thos., 12, Heywood Street, Bury, Lancashire.  
 Deaville, B., Hyson Green Works, Nottingham.  
 De Clerck, Maurice, Courtrai, Belgium.  
 Deering, W. H., Chemical Department, Royal Arsenal, Woolwich, S.E.; and (Journals) Endleigh, Glenhee Road, Westcombe Park, S.E.  
 Delahaye, Philibert, 65, Rue de Provence, Paris.  
 Dempsey, Geo. C., 165, Market Street, Lowell, Mass., U.S.A.  
 Demuth, Dr. L., Wharfedale, Church Road, Edgbaston, Birmingham.  
 Denison, Joseph R., 1, Park View Terrace, Manningham, Bradford.  
 Dent, W. Y., 5, Caithness Road, Brook Green, W.  
 Derrick, Wm. H., c/o The Pahang Corporation Ltd., Kuantan, via Singapore, S.S.  
 De Velling, F. W., Upper Board School, Heckmondwike, Yorks.  
 Devey, A. C., c/o La Société Hermite, 4, Rue Drouot, Paris.  
 Dewar, Prof. J., F.R.S., Royal Institution, Albemarle Street, W. (for Journals); and 1, Seroupe Terrace, Cambridge.  
 Dewey, Fred. P., 621, F. Street Northwest, Washington, D.C., U.S.A.  
 De Wilde, Prof. P., 339, Avenue Louise, Brussels, Belgium.  
 Dey, Proo Lal, 4, Beadon Street, Calcutta.  
 Diddin, W. J., London County Council, 40, Craven Street, London, S.W.; and (Journals) Mayfield, Grange Road, Sutton, Surrey.  
 Dick, A., 110, Cannon Street, London, E.C.  
 Dickerson, E. N., 15, Wall Street, New York, U.S.A.  
 Dickinson, A. J., Neptune Tar and Chemical Works, Deptford, London, S.E.  
 Dickson, Jno., 54, Brown Street, Broomielaw, Glasgow.  
 Dittich, Julius C., Ozone Manufacturing Co., 47, Liberty Street, New York, U.S.A.  
 Divers, Dr. E., F.R.S., Hongo, Tokyo, Japan.  
 Dixon, Prof. Harold B., F.R.S., Owens College, Manchester.  
 Dixon, Jos., Spring Grove, near Sheffield.  
 Dixon, M. T., 5, Brandling Park, Newcastle-on-Tyne.  
 Dixon, Wm., 102, Spring Street, Bury, Lancashire.  
 Dixon, William, 3, Belle Vue Park, Sunderland.  
 Dixon, W. Hepworth, Fairfield Works, Bow, E.  
 Dobb, Thos., Audrey Cottage, Union Road, Sharrow, Sheffield.  
 Dobbie, Dr. J. J., University College of North Wales, Bangor.  
 Dobbie, Dr. L., Chemical Laboratory, University, Edinburgh.  
 Dodd, A. J., River View, Belvedere, Kent.  
 Dodd, Archelaus, 135, Coleman Street, Whitmorecans, Wolverhampton.  
 Dodd, W. R., Dunsmore Road, Stamford Hill, W.  
 Doherty, Daniel, Inland Revenue, Hailsham, Sussex.  
 Doidge, H., 6, Beech Avenue, Sherwood Rise, Nottingham.  
 Domeier, A., 13, St. Mary-at-Hill, London, E.C.  
 Donald, George, Arnold Printworks, North Adams, Mass., U.S.A.  
 Donald, Jas., 5, Queen's Terrace, Glasgow.  
 Donald, Samuel, Corporation Gasworks, Dundee.  
 Donald, W., 29, Eglinton Street, Saltcoats, N.B.  
 Donald, W. J. A., 40, Lilybank Gardens, Glasgow.  
 Doolittle, Orrin S., Philadelphia and Reading Railroad, Cor. 7th and Franklin Streets, Reading, Pa., U.S.A.  
 Doran, Roht. E., 1, Goldsmith Terrace, Bray, Co. Wicklow.  
 Dore, Jas., Copper Works, High Street, Bromley-by-Bow, E.  
 Dott, D. B., c/o Duncan, Flockhardt, & Co., 104, South Canongate, Edinburgh.  
 Dougall, A., Gasworks, Sealecoates, Hull.  
 Dougall, Archibald, Gasworks, Kidderminster.  
 Douglas, William, Diamond Plantation, Demerara.  
 Doulton, Sir Henry, Lambeth Pottery, London, S.E.  
 Dowling, Edmund, 83, Cable Street, London, E.  
 Down, F. J., 28, Victoria Road, Old Charlton, S.E.  
 Down, T., Tharsis Copper Works, Cardiff.  
 Dowson, J. Emerson, 3, Great Queen Street, Westminster, S.W.

Drake, Chas. A., Three Mills Distillery, Bromley-by-Bow, E.  
 Dreyer, W. P., Silk Crape Works, Ponders End, N.  
 Drew, D., Lower House Printworks, near Barnley.  
 Dreyfus, Dr. C., Clayton Aniline Co., Limited, Clayton, Manchester.  
 Dreyfus, S., Clayton Aniline Co., Ltd., Clayton, Manchester.  
 Driffield, V. C., Appleton, Widnes.  
 Brown, Thos. M., Mass. Inst. of Technology, Boston, Mass., U.S.A.  
 Drummond, Hon. G. A., Montreal, Canada.  
 Duckworth, William, 93, Corporation Street, Manchester; and (Journals) Shawe Hall, Flixton, near Manchester.  
 Dudley, Dr. C. B., 1219, 12th Avenue, Altoona, Pa., U.S.A.  
 Dudley, Prof. W. L., Vanderbilt University, Nashville, Tenn., U.S.A.  
 Duffield, Dr. S. P., Dearborn, Wayne Co., Mich., U.S.A.  
 Duggan, T. R., Sunnyside, Vanbrugh Hill, Blackheath, S.E.  
 Dukes, T. William, c/o Dukes Bros., Caledon and Primrose Streets, Cape Town, South Africa.  
 Duncan, And., Daw-holm Gasworks, Maryhill, Glasgow.  
 Duncan, Arthur W., 99, Heaton Moor Road, near Stockport.  
 Duncan, J., 9, Mining Lane, London, E.C.; and (Journals) Maison Ganich, Biarritz, France.  
 Dunlop, Robt., Stanring Oil Works, Airdrie, N.B.  
 Dunn, Fred., 306, Flinders Lane, Melbourne, Victoria.  
 Dunn, J., 53, Brown Street, Manchester.  
 Dunn, John, Morgan Academy, Dundee.  
 Dunn, Dr. J. T., The School, Gateshead-on-Tyne.  
 Dunn, P., 53, Brown Street, Manchester.  
 Dunn, S.  
 Dunn, W. H., jun., 1, Belle Vue Terrace, Gateshead-on-Tyne.  
 Dunwoody, R. G., 369, Piedmont Avenue, Atlanta, Georgia, U.S.A.  
 Dupee, H. D., Walpole, Mass., U.S.A.  
 Dupré, Dr. A., F.R.S., Westminster Hospital Medical School, Caxton Street, London, S.W.  
 Durham, H., City of London School, Thames Embankment, E.C.  
 Dutton, W. H., 88, Wickham Road, Brockley, S.E.  
 Duxbury, Thos., Gas Works, Darwen, Lancashire.  
 Dvorkovitch, Dr. P., 6, Willow Bridge Road, Canonbury, N.  
 Dyer, Dr. B., 17, Great Tower Street, London, E.C.  
 Dyson, C. E., Flint, North Wales.  
 Dyson, Dr. G., Temple House, Cheetham Hill, Manchester.  
 Dyson, H., Pooley Hall Colliery, Polesworth, near Tamworth.  
 Dyson, Septimus, 92, Beausley Road, Frizinghall, Bradford, Yorks.

## E

Earnshaw, Edwin, 24, Mark Lane, London, E.C.  
 Earp, W. R., Halton Road, Runcorn, Cheshire.  
 Eastick, C. E., Clyde Wharf Refinery, Victoria Docks, E.  
 Eastick, J. J., c/o The Australasia Sugar Refining Co., Ltd., Melbourne, Victoria (Journals); and c/o J. Council & Co., Dunster House, Mining Lane, E.C. (subscription).  
 Eastlake, A. W., 17, Temperley Road, Balham, S.W.  
 Eastwick, Jos. H., Mellon Street, above Margaretta, East Liberty, Pittsburg, Pa., U.S.A.  
 Eastwood, Chas., Linaere Gas Works, Bootle, near Liverpool.  
 Eastwood, Edward, Tunnel Soap Works, Wapping, E.  
 Eddy, Harrison P., Sewage Purification Works, Worcester, Mass., U.S.A.  
 Ede, Henry E., (Journals) c/o W. C. Tripler, Coquimbo, Chile; and (snbs.) c/o Rev. Moore Ede, The Rectory, Gateshead-on-Tyne.  
 Edge, Anthony, Readville, Mass., U.S.A.  
 Edgell, G. E., 3, Queen's Terrace, Gateshead-on-Tyne.  
 Edmunds, Dr. Lewis H., 1, Garden Court, Temple, E.C.  
 Edwards, Henry W., Murray Mines, Sudbury, Ont., Canada.  
 Edwards, Vincent, 1, Coverdale, Montague Road, Richmond-on-Thames.

Ekman, C. D., Paper Mills, Northfleet, Kent.  
 Elborough, T., 59, Mark Lane, London, E.C.  
 Elliot, John, Free Library, Wolverhampton.  
 Elliott, Dr. A. H., Consolidated Gas Co., 4, Irving Place, New York, U.S.A.  
 Ellis, Alex., Skelton-in-Cleveland, Yorks.  
 Ellis, C. J., Cassel Gold Extracting Co., 13, West Scotland Street, Kinning Park, Glasgow.  
 Ellis, E. Victor, 21, Castle Street, Edinburgh.  
 Ellis, H., 112, Regent Street, Leicester.  
 Ellis, Prof. W. Hodgson, School of Practical Science, Toronto, Canada.  
 Ellison, Henry, Platt Lane, Cleckheaton, Yorks.  
 Elmore, A. S., Oulton Green, Oulton, Yorks.  
 Elwen, Geo., 47, Faulkner Street, Manchester.  
 Elworthy, H. S., 4, Hill Road, Bandra, Bombay, India.  
 Emmens, S. H., P.O. Box 2094, San Francisco, Cal., U.S.A.  
 Erdmann, Dr. H., 25—27, William Street, New York City, U.S.A.  
 England, R. J., Broomhill Lodge, Woodford Green, E.  
 Entwisle, Edw., Woodlands, Ashton-upon-Mersey, Cheshire.  
 Ermen, F., jun., Nassau Mills, Patricroft, Manchester.  
 Ernst, Adolf, Oherlangenbielan, Schlesien, Germany.  
 Erskine, J. K. (Subscriptions), 6, Lascotts Road, Wood Green, N.; and (Journals) c/o Randfontein Estates Gold Mining Co., Krugersdorp, S.A.R.  
 Esilman, A., 25, Roe Lane, Southport, Lancashire.  
 Esteourt, C., 20, Albert Square, Manchester.  
 Evans, Enoch, 181, Herbert Road, Small Heath, Birmingham.  
 Evans, Sir John, K.C.B., F.R.S., Nash Mills, Hemel Hempstead, Herts.  
 Evans, R. E., Swan's Nest Hotel, Stratford-on-Avon.  
 Evershed, F., Atlas Works, Hackney Wick, London, E.  
 Evershed, Henry G., Soap Works, Station Street, Brighton.  
 Evershed, Wallis, c/o Whitwell & Co., Ltd., Kendal, Westmoreland.  
 Exley, Arthur, Meanwood Grove, Meanwood, near Leeds.

## F

Fahlberg, Dr. C., Saccharin fabrik, Salbke-Westerhüsen a Elbe, Germany.  
 Fairley, T., 16, East Parade, Leeds.  
 Fairlie, H. C., 2, University Gardens, Glasgow.  
 Fallon, J. H. M., c/o Peruvian Corporation, Ltd., Lima, Peru.  
 Fallowfield, T., Clayton-le-Moors, near Accrington.  
 Fanta, F., 85, Gresham Street, E.C.  
 Farrant, N., c/o J. Nicholson and Sons, Chemical Works, Hunslet, Leeds.  
 Farries, T., 16, Coleman Street, London, E.C.  
 Farmer, Thos. H., 35, Oliver Street, Boston, Mass., U.S.A.  
 Farrington, T., 4, Waterloo Place, Cork, Ireland.  
 Fasnacht, A. E., 296, Ashton New Road, Clayton, Manchester.  
 Faulkner, E., The Laboratory, Bath Row, Birmingham.  
 Fawcett, Jas. H., Broken Hill Assay Office, 72, Mark Lane, E.C.  
 Fawsitt, C. A., Atlas Chemical Works, East Nelson Street, Glasgow.  
 Feld, Walther, Chemische Fabrik, Hönningen a/Rhein, Germany.  
 Fellowes, F. W., 61—62, Chancery Lane, London, W.C.  
 Felton, Thos., Granville Villa, Cleveland Road, South Woodford, E.  
 Fenwick, Jas., 23, Ronald Street, Glasgow.  
 Ferguson, Prof. J., The University, Glasgow.  
 Fergusson, H., Prince Regent's Wharf, Victoria Docks, E.  
 Ferguson, Wm. B., 3, Plowden Buildings, Temple, E.C.  
 Ferrie, And., Roslin House, Ellesmere Park, Eccles, Manchester.  
 Fiebing, John H., 644, 28th Street, Milwaukee, Wis., U.S.A.  
 Field, C. H., The Nottingham Brewery, Nottingham.  
 Field, E. W., The Brewery, Nottingham.  
 Field, S. E., Lewin's Mead Brewery, Bristol.

Field, S. S., 1, Bell Rock Villas, Mycenæ Road, Westcombe Park, S.E.  
 Field, Walter D., Wyoming, N.J., U.S.A.  
 Field, Wm. Eddington, 65, Sutherland Road, Armadale, Melbourne, Victoria.  
 Fielding, A., George Street, Salford, Manchester.  
 Fikentscher, Dr. F., 50, Lansdowne Road, Crumpsall, Manchester.  
 Filecock, P., Cumberland House, Cumberland Street, Macclesfield.  
 Findlay, T. J., c/o Messrs. Chapman and Messel, Silver-town, London, E.  
 Finlay, Kirkman, c/o Milne & Co., 123, Bishopgate Street, London, E.C.  
 Fischesser, Alf., Lutterbach, bei Mülhausen, Alsace.  
 Fisher, W. W., 5, St. Margaret's Road, Oxford.  
 Fitzbrown, G., Ditton Copper Works, Widnes.  
 Flanagan, Chas. A., Hegewisch, Ill., U.S.A.  
 Fleck, Hermann, American House, Reading, Pa., U.S.A.  
 Fleming, J. Arnold, Woodland Pottery, Tunstall, Staffs.  
 Fleming, R. G., 5, Cheapside, Tudhoe Grange, Spenny-moor, co. Durham.  
 Fletcher, A. E., 13, Christchurch Road, Crouch End, N.  
 Fletcher, E. Lyon, Grappenhall House, Grappenhall, near Warrington.  
 Fletcher, E. Morley, 82, George Street, Portman Square, London, W.  
 Fletcher, F. W., Beauchamp Lodge, Enfield.  
 Fletcher, G., 3, East Ascent, St. Leonards-on-Sea.  
 Fletcher, R. Jaques, The Hawthorns, Bromley, Kent.  
 Flintoff, R. J., Haxby, Crumpsall Lane, Manchester.  
 Flower, Major Lamoreck (Lee Conservancy Board), 12, Finsbury Circus, E.C.  
 Foden, Alfred, 52, Everton Valley, Everton, Liverpool.  
 Fogg, Jas., Waterloo Estate, Carapichaima, Trinidad, West Indies.  
 Foord, Geo., Royal Mint, Melbourne, Victoria, Australia.  
 Forbes, J., Chemical Works, Old Ford, London, E.  
 Ford, J. B., jun., The J. B. Ford Chemical Co., Wyandotte, Mich., U.S.A.  
 Ford, Jno. S., Abbey Brewery, Edinburgh.  
 Formoy, J. Arthur, 12, Railway Approach, London Bridge, S.E.  
 Forrester, Albert, c/o Arbuthnot & Co., Madras, India.  
 Forrester, A. M., Port Dundas Chemical Works, 20, Canal Bank, Glasgow.  
 Forrester, J., Leyton Villa, Church End, Finchley, N.  
 Forster, Ralph C., c/o Messrs. Bessler, Waechter, & Co., 18 and 19, Fenchurch Street, E.C.  
 Fort, Jas., 16, Adelphi Bank Chambers, South John Street, Liverpool.  
 Forth, Henry, Regent Street, New Basford, Nottingham.  
 Foster, R. Le Neve, The Pirs, North Road, Droydsden, Manchester.  
 Foster, Wm., Esholt House, Chapeltown, Leeds.  
 Foulis, Wm., 2, Montgomerie Quadrant, Kelvinside, Glasgow.  
 Fowler, Gilbert J., Dalton Hall, Victoria Park, Manchester.  
 Fox, J. Wesley, 115, Lower Thames Street, London, E.C.  
 Fox, T., jun., Court, Wellington, Somerset.  
 France, G. T., Friar's Goose Works, Gateshead-on-Tyne.  
 France, Joseph, 43, Church Street, Rotherham, Yorks.  
 France, H. C. D., 8, Vearage Road, Edgbaston, Birmingham.  
 Francis, E., Ivy Bank, Park Valley, Nottingham.  
 Francis, E. G., c/o Manbré Saccharine Co., Hammersmith, W.  
 Francis, G. B., 38, Southwark Street, London, S.E.  
 Francis, Wm., jun., City Laboratory, Cork Hill, Dublin.  
 Francis, W. H., 38, Southwark Street, London, S.E.  
 Frankel, L. K., 1315, Marshall Street, Philadelphia, Pa., U.S.A.  
 Frankenburg, Isidor, Greengate Rubber Works, Salford, Manchester.  
 Frankland, Dr. E., F.R.S., The Yews, Reigate, Surrey.  
 Frankland, H., Streonshalh House, The Crescent, Linthorpe, Middlesbrough.  
 Frankland, Dr. P. F., F.R.S., University College, Dundee.  
 Fraser, Leslie McG., 98, Commercial Road East, London, E.

Fraser, W. J., 98, Commercial Road East, London, E.  
 Free, R., The Elms, Mistley, Essex.  
 Freear, H. M., Hedgefield, Harpenden, Herts.  
 Freeman, A., 111, Needham Road, Edge Hill, Liverpool.  
 Freestone, J. W., 9, Clark's Terrace, New Ferry, Cheshire.  
 Frew, Wm., 2, King James' Place, Perth, N.B., and (Journals), Theresienstrasse, 21, München, Bavaria.  
 Fries, Dr. Harold H., 92, Reade Street, New York, U.S.A.  
 Friswell, R. J., 115, Darenth Road, Stamford Hill, London, N.  
 Froehling, Dr. H., 17, South 12th Street, Richmond, Virginia, U.S.  
 Frost, Dr. Howard V., Arlington, Mass., U.S.A.  
 Frost, Joe, Mold Green, Huddersfield.  
 Frusher, Thos., White Abbey Dyeworks, near Bradford, Yorks.  
 Fryer, Dr. A. C., Cornwallis Lodge, Clifton, Bristol.  
 Fuerst, Jos. F., 17, Philpot Lane, London, E.C.  
 Fukahori, Yoshiki, 6, Sanjikkembori, Sanehome, Tokyo, Japan.  
 Fuller, Chas. J. P., L. and Y. Railway Works, Horwich, near Bolton.  
 Fuller, W. M. (Journals), Lawn Side, Stow Park Circus, Newport, Mon.; and (subscription) c/o Morris & Griffin, Lim., Maidee, Newport, Mon.  
 Fultun, H. B., 33, St. Dunstan's Road, West Kensington, S.W.  
 Fyfe, Jno., 7, West George Street, Glasgow.

## G

Gabbett, E. R., Prince Regent's Wharf, Victoria Docks, London, E.  
 Gadsden, Capt. H. A.  
 Gair, Wm., 20, Cardigan Terrace, Heaton, Newcastle-on-Tyne.  
 Gajjar, Professor T. K., Temple of Art, Baroda, India.  
 Galbraith, Wm., West Park, Saltergate, Chesterfield.  
 Gallsworthy, Frank, 5-7, Meadow Lane, Leeds.  
 Gall, Henry, L'Usine de Produits Chimiques de Villers par Hermes, Oise, France.  
 Galt, Hugh Allen, c/o J. B. Ford Chemical Co., Wyandotte, Mich., U.S.A.  
 Gamble, Col. D., Windlehurst, St. Helens.  
 Gamble, D., jun., Millbrook, Eccleston, Prescot, Lancashire.  
 Gamble, J. C., Cowley Hill, St. Helens.  
 Gamble, Jas. N., The Laboratory, Procter and Gamble Co., Ivorydale, Ohio, U.S.A.  
 Gamble, W., Haresfinch, St. Helens.  
 Gans, Adolf, Farbenfabrik von L. Cassella & Co., Frankfurt o/Main, Germany.  
 Gargon, Prof. Jules, 13, Boulevard de Latour Maubourg, Paris.  
 Gardner, Walter M., Yorkshire College, Leeds.  
 Garibaldi, Joachim A., 21, Church Place, Gibraltar.  
 Garner, D. C., c/o Hicks & Co., Queen Street, Cardiff.  
 Garrett, F. C., The Polytechnic, Woolwich, S.E.  
 Garriek, Dr. A. R., Hyn-ton, near Liverpool.  
 Garton, R. (Bill, Garton & Co.), Southampton Wharf, Battersea, S.W.  
 Garton, Rd. S., 17, Winson Green Road, Birmingham.  
 Gascoyne, Dr. W. J., 36, South Holliday Street, Baltimore, Md., U.S.A.  
 Gaskell, H., jun., Clayton Lodge, Aigburth, near Liverpool.  
 Gaskell, Holbrook, Woolton Wood, Liverpool.  
 Gaskell, J., Woodlands Road, Cheetham Hill, Manchester.  
 Gatheral, G., 174, Soho Hill, Handsworth, Birmingham.  
 Geisler, Dr. Jos. F., New York Mercantile Exchange Building, 6, Harrison Street, New York, U.S.A.  
 Gendall, W. H. S., Gas Works, Elton, Bury.  
 Gerland, Dr. B. W., 4, Denmark Place, Accrington.  
 Gibb, Thos., The Court, Hal-wood, Liverpool.  
 Gibbins, H. B., Holly Lawn, Beechen Cliff, Bath.  
 Gibbs, D. Cecil, 16, Finsbury Circus, London, E.C.  
 Gibbs, R. D., 140, Manor Street, Clapham, S.W.  
 Gibbs, W. P., 57, Clive Road, Canton, Cardiff.  
 Gibbs, W. T., Laboratory, Ottawa, Canada.

- Gibson, Albert M., 11, Queen Victoria Street, London, E.C.  
 Gibson, Dr. J., 15, Hartington Gardens, Edinburgh.  
 Gibson, J. M., c/o Buckley Brick and Tile Co., Buckley, and Chester.  
 Gilbard, Francis, The Laboratory, 17, Great Tower Street, E.C.  
 Gilbert, Sir Jos. H., F.R.S., Harpenden, near St. Albans.  
 Gilchrist, P. C., F.R.S., Palace Chambers, 9, Bridge Street, Westminster, London, S.W.; Journals to Frogual Bank, Finchley New Road, Hampstead, N.W.  
 Gilchrist, Peter S., Brown's Wharf, Charleston, S.C., U.S.A.  
 Giles, W. B., 1, The Avenue, Church Road, Leyton, E.  
 Gill, Dr. Aug. H., Massachusetts Institute of Technology, Boston, Mass., U.S.A.  
 Gillman, Gustave, Ferro-carril de Murcia á Granada, Aguilas, Prov. de Murcia, Spain.  
 Gilmonr, J. D., 142, Aitkenhead Road, South Side, Glasgow.  
 Gillingham, Edw. A., Stamford House, Northumberland Park, Tottenham, N.  
 Girdwood, Dr. G. P., 82, University Street, Montreal, Canada.  
 Gladstone, Dr. J. H., F.R.S., 17, Pembroke Square, London, W.  
 Glaeser, F. A., Carpenter's Road, Stratford, E.  
 Glaser, Chas., 412, East Lombard Street, Baltimore, Md., U.S.A.  
 Glatz, Jos., Riverside Chemical Works, 485—493, Kent Avenue, Brooklyn, N.Y., U.S.A.  
 Glen, J., jun., Glengowan Printworks, Caldererux, N.B.  
 Glendinning, H., Mount House, The Hill, Sandbach, Cheshire.  
 Glendinning, N., Merton Bank, St. Helens.  
 Glendinning, Tom A.  
 Gloag, Robt. F., Grove Hill, Middlesbrough.  
 Glover, G. T., The Phospho Guano Co., Limited, Seacombe, Cheshire.  
 Glover, John, 20, Holly Avenue, Newcastle-on-Tyne.  
 Glover, T., Messrs. Mort, Liddell & Co., Widnes.  
 Glover, W., Rio Tinto Mines, Huelva, Spain.  
 Goldschmidt, Dr. S. A., 43—51, Sedgwick Street, Brooklyn, N.Y., U.S.A.  
 Goodall, Geo., 182, Mansfield Road, Nottingham.  
 Goodall, Reginald, Linden House, Highgate Road, N.W.  
 Goodall, Thos., Hendon Grange, Sanderland.  
 Goodall, Walter, Alma House, Pudsey, near Leeds.  
 Goodwin, C. C., Throstle Nest, Old Trafford, Manchester.  
 Goppelsroeder, Dr. F., Mulhausen, Elsass, Germany.  
 Gordon, J. G., The Mannesmann Tube Co., Ltd., 110, Cannon Street, London, E.C.  
 Gore, Dr. G., F.R.S., 67, Broad Street, Birmingham.  
 Gervin, Jno. C., English Crown Spelter Works, Port Tennant, Swansea.  
 Gessage, F. H., Widnes.  
 Gombing, Wm. Joshua, 25, Eden Quay, Dublin.  
 Gow, R. J., Ditton Iron Works, Widnes.  
 Gowlan, W., 19, Beaumont Crescent, West Kensington, W.  
 Goyder, G. A., Ootalinka, Hawker's Road, Medindie, near Adelaide, South Australia.  
 Grabfield, Dr. J. P., 1915, Indiana Avenue, Chicago, Ill., U.S.A.  
 Gracey, R., Faircombe, The Barnfield, Exeter.  
 Graesser, R., Cefn, near Ruabon, North Wales; and Argoed Hall, Llangollen, North Wales.  
 Graham, Dr. C., 23, Euston Buildings, Gower Street Station, London, N.W.  
 Graham, C. C., c/o Blundell, Spence & Co., Beverley Road, Hull.  
 Graham, J. A., The Rookery Farm, Dovercourt, Essex.  
 Grandage, H., Calder Dye Works, Brighouse, near Leeds.  
 Gratama, Dr. W. D., Polytechnic School, Delft, Holland.  
 Gray, G. Watson, 14, Argy'e Road, Garston, near Liverpool.  
 Gray, Jno., Pentland Oil Works, Loanhead, N.B.  
 Gray, W., Oil Refinery, Hull.  
 Graves, I. A. R., The Old Rectory, Grappenhall, Cheshire.  
 Green, Alfred H., Oaklands, Lowton, Newton-le-Willows, Lancashire.  
 Green, A. G., Atlas Works, Hackney Wick, London, E.; and (Journals) 54, Thistlewaite Rd., Lower Clapton, E.  
 Green, F. C., Chihuahua, Mexico.  
 Green, H., Hayle Mill, Maidstone.  
 Green, Jno. Edw., 42, Gilesgate, Durham.  
 Green, L., Lower Tovil, Maidstone.  
 Green, R., Soho Mill, Woodburn, near Beaconsfield.  
 Green, Samuel, 28 and 29, St. Swithin's Lane, London, E.C.  
 Greenaway, A. J., Frogmal, Hampstead, N.W.  
 Greenhalgh, J. Herbert, Shepherd's, Tottington Mill, near Bury.  
 Greenhough, D. W., 5, Rood Lane, London, E.C.  
 Greenway, T. J., 8th Avenue, East Adelaide, South Australia.  
 Greenwood, H., Holland Bank House, Church, near Acreington.  
 Gregory, Wm., Steam Brewery, Dartford, Kent.  
 Gregory, Wm. J., 1, St. John's Terrace, Weymouth, Dorset.  
 Greville, H. L., Diersheim, Churehfields, Woodford, Essex.  
 Griffin, John R., 22, Garriek Street, Covent Garden, W.C.  
 Griffin, Martin L., Mechanicville, Saratoga Co., N.Y., U.S.A.  
 Griffith, D. Agnew, 41, Hamilton Square, Birkenhead.  
 Griffith, R. W. S., Eyeworth Lodge, Lyndhurst, Hants.  
 Grime, J., Rosebank Cottage, Busby, near Glasgow.  
 Grimsshaw, H., Thornton View, Clayton, Manchester.  
 Grimwood, R., 41, Lady Margaret Road, London, N.W.  
 Grindley, J., Upper North Street, Poplar, London, E.  
 Gripper, Harold, Stores Department, M. S. and L. Railway, Gorton, Manchester.  
 Gronow, W. T., Port Pirie Smelting Works, Port Pirie, South Australia.  
 Grossmann, Dr. J., Hendham Vale Chemical Works, Manchester.  
 Groves, C. E., F.R.S., 352, Kennington Road, London, S.E.  
 Gunn, W. L., Broad Plain Soap Works, Bristol.  
 Gurney, J. Clare, Fabrica Roma, La Union, Prov. de Murcia, Spain.  
 Guthrie, John A., 34, Lancaster Park, Richmond, Surrey.  
 Guthrie, John, 50, Prospect Terrace, Hunslet Moor, Leeds.  
 Guttman, Oscar, 110, Adelaide Road, N.W.  
 Guyatt, T., Ceara Gas Co., Limited, 9, Queen Street Place, Cannon Street, London, E.C.

## H

- Habirshaw, W. M., 159, Front Street, New York City, U.S.A.  
 Hacking, W. H., The Grange, Clayton-le-Moors, near Acreington.  
 Haddow, A., 1, Easter Road, Edinburgh.  
 Hadfield, R. A., Newhall Road, Attercliffe, Sheffield.  
 Hadfield, Thos., Garrison Bleachworks, Birch Vale, near Stockport.  
 Hadkinson, F., Pamphila Oil and Soap Works, Mitylene, Mediterranean.  
 Hadkinson, R., Smyrna, Asia Minor.  
 Hadley, H. E., The School of Science, Kidderminster.  
 Haga, Tamenasa, Chemical Department, Science College, Imperial University, Tokyo, Japan.  
 Haig, Robert, Mechanical Retorts Co., Limited, Murray Street, Paisley, N.B.  
 Haig-Brown, R., jun., 21, Ladyborn Road, Fallowfield, Manchester.  
 Haigh, Ben, 21, Cavendish Road, Leeds.  
 Hailes, A. J. de, 2, Fisher Street, Red Lion Square, W.C.  
 Haines, Reuben, 201, South 5th Street, Philadelphia, Pa., U.S.A.; and (Journals) Haines Street, near Chew Street, Germantown, Philadelphia, Pa., U.S.A.  
 Hake, C. N., Department of Trade and Customs, Melbourne, Victoria.  
 Hale, Edw. P., c/o Wakefield & Co., Gatebeck, Kendal.  
 Hall, Allan T., Inglebank, Newland, Hull.  
 Hall, Archibald D., 34, Bishopsgate Street, London, E.C.  
 Hall, Edgar, (Journals) Albert Street, Brisbane, Queensland; and (subs.) c/o Geo. Bishop, 113, Powis Street, Woolwich, S.E.  
 Hall, J. Albert, 108, Lloyd Street, Greenheys, Manchester (subs.); and (Journals) Victoria Chemical Co., Victoria, British Columbia.

- Hall, Jas. W., Bombay, Baroda, and Central India Railway, Parell, Bombay, India.
- Hall, R. E., Box 12, Johannesburg, South African Republic.
- Hall, S., East London Soap Works, Bow, London, E.
- Haller, Geo., 86, Lendenhall Street, London, E.C.
- Haller, H. Left, St. Faith's House, St. Faith's Lane, Norwich.
- Hamaguchi, K., Hiro Mura, Arito Gori, Wakayama Ken, Japan.
- Hamilton, David, 224, Ingram Street, Glasgow.
- Hamilton, David R., 19, Graham Street, Bridgeton, Glasgow.
- Hamilton, Jas. C., c/o H. Swinglehurst, Hineaster House, Milnthorpe, Westmoreland.
- Hamilton, Oswald, c/o The British White Lead Co., Northfleet, Kent.
- Hamilton, Robert, c/o Furnace Gases Co., Ltd., Shotts, N.B.
- Hamilton, Robt., Leeds Steelworks, Ltd., Leeds.
- Hamlen, G. J., c/o Reptano Chemical Co., Chester, Pa., U.S.A.
- Hammersley, Wm., Tar Works, Beekton, E.
- Hammersley, W. A. L., Bridge House, Leek, Staffordshire.
- Hammill, M. J., 9, Windle Street, St. Helens.
- Hammond, Geo. W., Hotel Hamilton, Boston, Mass., U.S.A.
- Hammond, J., Gas Works, Eastbourne, Sussex.
- Hammond, R. Percy, Sunny Side, Reddish Vale, near Stockport.
- Hampson, Jas., 42, Woodland Street, Cheetham Hill, Manchester.
- Hand, T. W., Public Library, Oldham.
- Handy, Jas. O., c/o Hunt and Clapp, Pittsburg, Pa., U.S.A.
- Hanks, Abbot A., 718, Montgomery Street, San Francisco, Cal., U.S.
- Haurez, Prosper, 190, Chaussée de Charleroi, Brussels.
- Hanson, A. M., Abbey Printworks, Whalley, Blackburn.
- Hanson, John, Highfield Villa, Belle Vue, Wakefield.
- Hardie, William, The Gas Offices, Newcastle-on-Tyne.
- Hardman, Josiah, Milton Chemical Works, Stoke-on-Trent.
- Hargreaves, J., Widnes.
- Hargreaves, Mark, 108, Fylde Road, Preston.
- Harkness, W., The Laboratory, Somerset House, London, W.C.
- Harland, R. H., Plough Court, 37, Lombard Street, London, E.C.
- Harlock, E. B., Queen's Road, Urmston, near Manchester.
- Harmon, L. E., c/o E. Elsworth and Co., 71, Park Place, New York, U.S.A.
- Harned, Frank P., 1332, Washington Avenue, Philadelphia, Pa., U.S.A.
- Harrington, W. B., Ardsullagh, Old Blackrock Road, Cork.
- Harris, Arthur, Marsh Gate Works, Stratford, E.
- Harris, Booth, jun., Hillside, Loughton, Essex.
- Harris, D., Caroline Park, Edinburgh.
- Harris, Sydney J., School of Science, Rossington Street, Leeds.
- Harris, Sydney W., 15, Lansdowne Terrace, Walters Road, Swansea.
- Harris, T., The Union Acid Co., Runcorn.
- Harrison, A., Thames Sugar Refinery, Silvertown, London, E.
- Harrison, C., 67, Surrey Street, Sheffield.
- Harrison, Dr. Franklin T., London, Ontario, Canada.
- Harrison, G. D., Netham Chemical Works, Bristol.
- Harrison, G. H., Hagley, near Stourbridge.
- Harrison, G. King, Hagley, near Stourbridge.
- Harrison, J., Madore, Ballintemple, Cork.
- Harrison, Jno., 35th and Gray's Ferry Road, Philadelphia, Pa., U.S.A.
- Harrison, Prof. John B., Government Laboratory, Georgetown, British Guiana.
- Harrison, M. C. C., Church Green, Witney, Oxon.
- Hart, Bertram H., The Elms, Old Charlton, S.E.
- Hart, Dr. E., Lafayette College, Easton, Pa., U.S.A.
- Hart, H. W., Love Clough, Rawtenstall, Lancashire.
- Hart, P., c/o Tennants & Co., Clayton, Manchester.
- Hartford, Jas., 3, Cedar Street, New York, U.S.A.
- Hartley, Arthur, Cannon Brewery, Brighton.
- Hartley, Edw., 62, Wall Street, New York City, U.S.A.
- Hartley, Joseph, Dalton Chemical Works, Brook Street, West Gorton, Manchester.
- Hartley, R. Kent, Springwood House, Chadderton, near Oldham.
- Hartley, Prof. W. N., F.R.S., Royal College of Science, Dublin.
- Hartog, Philip J., Owens College, Manchester.
- Hartridge, Jas. Hills, Holmwood, London.
- Harvey, E. Field, Omrac, St. John's, Newfoundland.
- Harvey, Ernest W., Hilsrig, Alderbrook Road, Balham, S.W.
- Harvey, H. C., Raglan House, Brooklands, near Manchester.
- Harvey, Sidney, South-Eastern Laboratory, Canterbury.
- Harvey, T. H., Cattedown, Plymouth.
- Harzer, C. A., Billiter Buildings, Billiter Street, London, E.C.
- Hasenclaver, R., Chemische Fabrik-Rhenania, Aachen, Prussia.
- Hastings, Hugh, 10, New Tree Road, Kidderminster.
- Hathaway, Nath., New Bedford, Mass., U.S.A.
- Hatsehek, M., 51, Fulham Park Gardens, Fulham, S.W.
- Hatton, Wm. P., c/o W. R. Hatton & Sons, Wormwood Scrubs, W.
- Hauff, Julius, Feuerbach, Stuttgart, Germany.
- Haussknecht, Dr. Willy, 123, Invalidenstrasse, Berlin, N.
- Hawkins, H., Eyeworth Lodge, Lyndhurst, Hants.
- Hawkins, J. Dawson, University Club, Denver, Col., U.S.A.
- Hawliczek, Josef, Claremont Park, Blackpool.
- Hayes, Jno., 11, Burlington Chambers, New Street, Birmingham.
- Heal, Carlton, Leather Department, Yorkshire College, Leeds.
- Heap, Chas., Caldershaw, near Rochdale.
- Heap, L., Stacksteads, near Manchester.
- Heape, Chas., 19, George Street, Manchester.
- Heath, G. L., Calmet and Hecla Smelting Co., South Lake, Linden, Mich., U.S.A.
- Heath, R. C., Myton Grange, near Warwick.
- Heaton, John, 744, Rochdale Road, Manchester.
- Hecht, Jos., 3435, Walnut Street, Philadelphia, Pa., U.S.A.
- Heckmann, C., 9, Gölitzerufer, Berlin, S.O., Germany.
- Hedley, Armorer, Mayfield, Gosforth, Newcastle-on-Tyne.
- Hedley, John, jun., 11, Crooked Lane, E.C.; and (Journals) 21, Russell Chambers, Bloomsbury, W.C.
- Heerlein, Robert, Pennsylvania Salt Manufacturing Co., Natrona, Pa., U.S.A.
- Heford, Geo., jun., 40, Chapel Lane, Huddersley, Leeds.
- Hehner, O., 11, Billiter Square, London, E.C.
- Heisch, Godfrey P., 325, Clapham Road, London, S.W.
- Hellier, E. A., 111, Westbourne Avenue, Hull.
- Hellon, Dr. R., 47, New Lowther Street, Whitehaven.
- Helm, H. J., Simonstone, Hammelton Road, Bromley, Kent.
- Hemingway, H., 60, Mark Lane, London, E.C.
- Hempleman, F. S., Wennington House, Wennington, Romford, Essex.
- Henderson, Prof. G. G., The Technical College, George Street, Glasgow.
- Henderson, W. F., Moorfield, Claremont Gardens, Newcastle-on-Tyne.
- Hendrichs, Frank H., Imperial Wharf, 46, Bankside, London, S.E.
- Hendrick, Jas., Young Laboratory, 60, John Street, Glasgow.
- Hennin, Alphonse, Monongahela Furnaces, McKeesport, Pa., U.S.A.
- Henshaw, Jno., Brook Street Soap Works, Manchester.
- Herf, O., Herf & Frerichs Chemical Co., St. Louis, Mo., U.S.A.
- Herman, W. D., Eccleston Park, Prescott, Lancashire.
- Heron, J., 74, North Side, Clapham Common, S.W.
- Herriot, Wm. Scott, De Willem, Demerara.
- Herrmann, R. W. (Herrmann, Keller & Co.), 102, Fenchurch Street, London, E.C.
- Hersam, Ernest A., State University, College of Mines, Berkeley, Cal., U.S.A.
- Herschel, Prof. A. S., F.R.S., Observatory House, Slough, Bucks.
- Heslop, Jos., 110, Rye Hill, Newcastle-on-Tyne.



- Neslop, Oliver, 110, Rye Hill, Newcastle-on-Tyne.  
 Hess, Dr. Adolph, Oil Works, Leeds.  
 Hetherington, Dr. Albert E., 65, Sandown Lane, Wavertree, Liverpool.  
 Hewitt, Dr. D. B., Oakleigh, Northwich, Cheshire.  
 Hewlett, John C., 40-42, Charlotte Street, Great Eastern Street, London, E.C.  
 Hey, Harry, Queen's Mills, Savile Town, Dewsbury.  
 Heyden, Dr. F. von, Chemische Fabrik, Radebeul, bei Dresden, Germany.  
 Heys, W. E., 70, Market Street, Manchester.  
 Heys, Z. J., Stonehouse, Barrhead, N.B.  
 Heys, Z. G., Springhill Villa, Barrhead, near Glasgow.  
 Heywood, J. G., 127, Sutherland Avenue, Maida Vale, London, W.  
 Heywood, J. H., 23, Holland Street, Rochdale.  
 Heywood, J. S., 7, Caledonian Road, King's Cross, London, N.  
 Hibbert, W., 14, Goldhurst Terrace, South Hampstead, N.W.  
 Hick, Alf. W., 12, Adwood Lane East, Stockport.  
 Hicks, Jas. A., c/o B. Redwood, 4, Bishopsgate Street Within, London, E.C.  
 Higgin, W. H., Hopefield, Bolton-le-Moors.  
 Higgins, C. L., c/o J. Muspratt & Co., Widnes; and 29, Falkner Square, Liverpool.  
 Hill, J. K., Fullarton Cottage, Irvine, N.B.  
 Hill, Sydney, 11, Salisbury Street, The Park, Hull.  
 Hills, C. H., Anglesea Copper Works, Low Walker, Newcastle-on-Tyne.  
 Hills, H., Chemical Works, Deptford, London, S.E.  
 Hills, M. H., Tower Varnish Works, Long Acre, Nechells, Birmingham.  
 Hills, W., 225, Oxford Street, London, W.  
 Hilton, Edgar G., 37, Kourland-sky Street, St. Petersburg, Russia.  
 Hindle, J. H., 8, Colham Street, Accrington.  
 Hinds, James, 127, Gosford Street, Coventry.  
 Hinman, Bertrand C., c/o Ironclad Manufacturing Co., 186, Cook Street, Brooklyn, E.D., U.S.A.  
 Hinchelwood, Thos., Glasgow Oil and Paint Works, Glenpark Street, Glasgow.  
 Hodges, Harry B., c/o B. and O. R. R., Mount Clare, Baltimore, Md., U.S.A.  
 Hodges, J. F., Tudor Park, Hollywood, Co. Down, Ireland.  
 Hodgkin, J., 12, Dynevor Road, Richmond, Surrey.  
 Hodgkinson, J. H., Messrs. E. Potter & Co., Dinting Vale, Glossop.  
 Hodgkinson, Dr. W. R., 8, Park Villas, Blackheath, S.E. (Journals); and Royal Military Academy, Woolwich, S.E.  
 Hodgson, C., High House, Eppleby, Darlington.  
 Hodgson, Wm., 8, Victoria Buildings, St. Mary's Gate, Manchester.  
 Hogben, W., 7, Brighton Terrace, Joppa, Midlothian, N.B.  
 Hogg, Quintin, 23, Rood Lane, London, E.C.  
 Hogg, T. W., c/o John Spencer & Sons, Newburn Steelworks, Newcastle-on-Tyne.  
 Holden, G. H., Langley Place, Victoria Park, Manchester.  
 Holgate, S. V., 29, Long Row, Nottingham.  
 Holgate, T. E., 146, Blackburn Road, Darwen, Lancashire.  
 Holgate, T., 12, Hyde Park Road, Halifax.  
 Holland, Jos., Brewery House, West Gorton, Manchester.  
 Holland, Philip, c/o Jas. Greenway, 33, London Street, Southport.  
 Holland, Philip H., 958, Sherbrooke Street, Montreal, Canada.  
 Holliday, R. (Read, Holliday & Sons), Huddersfield.  
 Holloman, Fred R., 51, Russell Road, Custom House, E.  
 Holloway, G. T., 57 and 58, Chancery Lane, W.C.  
 Holloway, Wm., Newlands, Middlesbrough.  
 Holmes, Eliwood, Wellburn, Jesmond, Newcastle-on-Tyne.  
 Holmes, F. G., 76, Pepys Road, New Cross, S.E.  
 Holmes, J., 96, Holland Road, Brixton, London, S.W.  
 Holt, J. W., North Road, Clayton, near Manchester.  
 Holton, E. C., Sherwin-Williams Co., 100, Canal Street, Cleveland, Ohio, U.S.A.  
 Holzappel, Max., Quayside, Newcastle-on-Tyne.  
 Homfray, D., 42, South Street, Greenwich, S.E.  
 Hooker, Benj., Pear Tree Court, Farringdon Road, London, E.C.  
 Hooper, E. G., The Laboratory, Somerset House, London, W.C.  
 Hooper, Ernest F., c/o Messrs. Burt, Boulton, & Haywood, Chemical Works, Silvertown, E.  
 Hooton, Edm., Fairfield, Hamilton Drive, Nottingham.  
 Hope, Jas., The Nickel Co., Kirkintilloch, N.B.  
 Hopkin, W. K., Fernbrae, Brondesbury Park, N.W.  
 Hopkins, Erasmus, 256, Benefit Street, Providence, R.I., U.S.A.  
 Hopkins, Gerald V., Estab<sup>o</sup> Bella Vista, Fondicion y Beneficiadora, Antofagasta, Chili.  
 Hopkins, T. J., Ivy Cottage, Staverton, near Trowbridge.  
 Hopkinson, John, Marion Street, Lister Hills, Bradford, Yorks.  
 Horn, Wm., Roxburgh Street Refinery, Greenock, N.B.  
 Horn, W. Freeman, Wandle Colour Works, South Street, Wandsworth, S.W.  
 Horner, Jarvis W., 27, Brookfield Road, Sheffield.  
 Horrocks, S., 41, Parkfield Road, Sefton Park, Liverpool.  
 Horrocks, W. A., 8, Aldred Street, Crescent, Salford.  
 Horton, William, 38, Belvidere Road, Prince's Park, Liverpool.  
 Hosie, G. H., Nobel's Villas, Stevenston, Ayrshire, N.B.  
 Hoskins, A. Percy, 25, Cromwell Road, Belfast.  
 Houston, John, 14, Charlotte Street, Manchester.  
 Houston, Robt. S., Brisbane House, Bellahouston, Glasgow.  
 Hovenden, Fred., Glenlea, Thurlow Park Road, West Dulwich, S.E.  
 Howard, A. G., Holmbury, Woodford, Essex.  
 Howard, D., Devon House, Buckhurst Hill, Essex.  
 Howard, D. L., City Mills, Stratford, London, E.  
 Howard, W. D., City Mills, Stratford, London, E.  
 Howarth, R. S., The Standard Perfumery Co., 12, Ardwick Green, Manchester.  
 Howarth, Franklin W., c/o Lloyd Wise, 46, Lincoln's Inn Fields, W.C.  
 Hughes, J., 79, Mark Lane, London, E.C.  
 Hughes, T., 31, London Square, Cardiff.  
 Hulme, J., Newton Chemical Works, Hyde, Manchester.  
 Humfrey, Chas., Winnington Park, Northwich.  
 Hummel, Prof. J. J., 152, Woodsley Road, Leeds.  
 Humphrys, N. H., Gasworks, Salisbury, Wilts.  
 Hunt, Bertram, P.O. Box 1606, Denver, Col., U.S.A.  
 Hunt, C., Gasworks, Windsor Street, Birmingham.  
 Hunt, E., Wood Green, Wednesbury, Staffordshire.  
 Hunt, F. F., 77, Pine Street, New York City, U.S.A.  
 Hunt, F. J., Bow Bridge Soap Works, Stratford, E.  
 Hunt, J. S., Appleton, Widnes.  
 Hunt, W., Hampton House, Wednesbury, Staffordshire.  
 Hunter, Prof. Matthew, Rangoon College, Rangoon, Burmah.  
 Hunter, Oliver, 9, Church Bank, Bolton.  
 Hunter, Sidney H., 202, Bow Road, E.  
 Hunter, T. G., 625, Drexel Building, 54th Street, Philadelphia, Pa., U.S.A.  
 Huntington, Prof. A. K., King's College, Strand, London, W.C.  
 Huntton, Henry, Greystone, Carlton, Ferryhill, co. Durham.  
 Hunzinger, A., Dinting Vale Printworks, Dinting, Derbyshire.  
 Hurst, G. H., 22, Blackfriars Street, Salford, Manchester.  
 Hurter, Dr. F., Widnes; and (Journals) Holly Lodge, Cressington Park, Liverpool.  
 Huskisson, P. L., 77, Swinton Street, London, W.C.  
 Huxon, C. W., 5, York Buildings, Dale Street, Liverpool.  
 Hutchinson, C. C., Engineering Works, Carpenter's Road, Stratford, E.  
 Hutchinson, Chas. H., Falcon Works, Sackville Street, Barnsley, Yorkshire.  
 Hutchinson, T. J., Aden House, Manchester Road, Bury.  
 Huxley, J. H., 15, Kenwood Park Road, Sharrow, Sheffield.  
 Hyde, C. F., 71, Higher Ardwick, Manchester.



Idris, T. H. W., Pratt Street, Camden Town, N.W.  
 Inuray, Harold, The Grange, Underhill, New Barnet.  
 Ingle, Harry, Pool, near Leeds; and (Journals) Jägerstrasse  
 7 IV. München, Bavaria.  
 Ingle, Herbert, Yorkshire College, Leeds.  
 Inglis, R. A., Culrain, Bothwell, N.B.  
 Innes, Murray, 76, Donohoe Building, San Francisco, Cal.,  
 U.S.A.  
 Iriyè, Koremasa, China and Japan Trading Co., Kobe,  
 Japan.  
 Irvine, R., Royston, Granton, Edinburgh.  
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 Copper Co., 30, George Square, Glasgow.  
 Irving, J. M., 17A, Dickinson Street, Cooper Street, Man-  
 chester.  
 Irwin, W., 3, Wilton Polygon, Cheetham Hill, Manchester.  
 Isaac, J. F. W.  
 Isaac, T. W. Player, 11, Cavendish Place, Brighton.  
 Isler, Otto, 23, Cooper Street, Manchester.  
 Ivatts, Harold E., c/o Lawes and Co., Chemical Works,  
 Barking Creek, Essex.

## J

Jackman, E. J., Northern Outfall Works, Beekton, E.  
 Jackson, Edgar, 106, Queen Victoria Street, London, E.C.  
 Jackson, Edward, Ashleigh, Beaufort Road, Edgbaston,  
 Birmingham.  
 Jackson, F., Spring Bank, Crumpsall Lane, Manchester.  
 Jackson, Frederick, 10, Half Moon Street, Manchester.  
 Jackson, G. B., 6, Booth Street, Piccadilly, Manchester.  
 Jackson, Jas., Dominion Cotton Mills Co., Magog, Quebec,  
 Canada.  
 Jackson, John, 98, Dobbie's Loan, Glasgow.  
 Jackson, R. V., c/o Scotch and Irish Oxygen Co., Pelmadie,  
 Glasgow.  
 Jackson, Robt., 18, Harrington Street, Dublin.  
 Jackson, Saml., 7, Mulgrave Terrace, Dalton, Huddersfield.  
 Jackson, T., Clayton, near Manchester.  
 Jackson, V., Park Hill, 10, Chorley Old Road, Bolton.  
 Jackson, Walter, 24, Sydenham Avenue, Sefton Park,  
 Liverpool.  
 Jackson, Rt. Hon. W. L., M.P., F.R.S., 27, Cadogan  
 Square, S.W.; and (Journals) Allerton Hall, Chapel  
 Allerton, near Leeds.  
 Jackson, W. P., Saxilby, near Lincoln.  
 Jago, Wm., Science Schools, Brighton; Journals to 32,  
 Clarendon Villas, Brighton.  
 James, Alf., Cassel Gold Extracting Co., 13, West Scotland  
 Street, Kinning Park, Glasgow.  
 James, Christopher, 34, York Road, Edgbaston, Birning-  
 ham.  
 James, E. T., British Alizarin Co., Ltd., Silvertown, Vic-  
 toria Docks, E.  
 James, J. Hernaman, Assay Office, Swansea.  
 James, Dr. J. Wm., Aylmer House, Weston-super-Mare;  
 and (Journals) 29, Redcliff Street, Bristol.  
 James, Lawrence S., 32, Hawley Street, Boston, Mass.,  
 U.S.A.  
 Jantzen, Paul, 132, Fenchurch Street, London, E.C.  
 Japp, Dr. F. R., F.R.S., The University, Aberdeen.  
 Jarman, George, 9, York Place, Huddersfield.  
 Jarman, Geo. S.  
 Jarmay, G., Hartford Lodge, Hartford, Cheshire.  
 Jarves, Deming, Michigan Carbon Works, Detroit, Mich.,  
 U.S.A.  
 Jayne, Dr. H. W., Chem. Lab., Bermuda Street, Frankford,  
 Philadelphia, U.S.A.  
 Jekyll, J., Castle Moat House, Lincoln.  
 Jenkin, W. A., Casino Ingles, Minas de Rio Tinto,  
 Provincia de Huelva, Spain.

Jenkins, Thos., Laboratorio, Minas de Rio Tinto, Spain.  
 Jenkins, Thos. H., 5, Bellott Street, Cheetham, Manchester.  
 Jenner, E., 209, Markhouse Road, Walthamstow, Essex.  
 Jemison, F. H., c/o A. Illingworth, Moscow, Russia.  
 Johnson, A. E., 10, Victoria Street, Wolverhampton; and  
 1, Eagle Villas, Penn Fields, Wolverhampton (for  
 Journals).  
 Johnson, Ben H., Caroline Cottage, West Grove Road,  
 Woodford, Essex.  
 Johnson, Chas. H., jr., Laboratory, Corn Exchange Works,  
 Leeds.  
 Johnson, Edmund E., 258, Brunswick Street, Choulton-on-  
 Medlock, Manchester.  
 Johnson, Frank, Tharsis Mines, Huelva, Spain.  
 Johnson, J. E., 40, Edmiston Road, Stratford, London, E.  
 Johnson, J. Grove, 23, Cross Street, Finsbury, London,  
 E.C.  
 Johnson, J. Henry, Mountains, Tunbridge, Kent.  
 Johnson, S. H., Warren Hill House, Loughton, Essex.  
 Johnson, T. A., Field House, Wington Park, Northwich,  
 Cheshire.  
 Johnston, Thos., Nobel's Explosives Co., Ltd., 149, West  
 George Street, Glasgow.  
 Johnston, Wm. A., The S.S. White Dental Manufacturing  
 Co., Princess Bay, New York, U.S.A.  
 Johnston, Wm. G., Chemical Works, Coatbridge Street,  
 Port Dundas, Glasgow.  
 Johnston, W. G., Warwicks' and Richardsons' Brewery,  
 Newark-on-Trent.  
 Johnstone, Jas., Shawfield Works, Rutherglen, Glasgow.  
 Johnstone, L., 1, Leadenhall Street, E.C.  
 Johnstone, Dr. W., Edlingham House, Arundel Street,  
 Strand, W.C.  
 Jones, A. Frederick, c/o Walters Bros., Pisagua, Chili.  
 Jones, David S., Wilson Terrace, Coatbridge, N.B.  
 Jones, Prof. D. E., County Council Offices, Stafford.  
 Jones, E. W. T., 10, Victoria Street, Wolverhampton.  
 Jones, H. Chapman, Royal College of Science, South  
 Kensington, London, S.W.  
 Jones, Herbert, c/o F. J. Pütz and Co., 21, Mining Lane,  
 London, E.C.  
 Jones, John Arthur, Gijón, Spain.  
 Jones, T. Simpson, 39, Lime Street, London, E.C.  
 Jones, T. Tolley, 486, Collins Street, Melbourne, Victoria.  
 Jones, Wm., 61, Chadwick Road, Peckham, S.E.  
 Jones, W. Norris, Runcorn Soap and Alkali Co., Weston,  
 near Runcorn.  
 Joseland, Walter H., Mitchell's Wood Cottage, near  
 Chesterton, Staffordshire.  
 Joslin, Omar, c/o Messrs. Swift & Co., Union Stock Yards,  
 Chicago, Ill., U.S.A.  
 Joüet, Cavalier H., Roselle, Union Co., N.J., U.S.A.  
 Journaud, Louis, Ste. Colombe-lez-Vienne, Rhône, France.  
 Jowett, W., Lower Hall, Mellor, near Stockport.  
 Joy, Douglas G., Welton Hill, Brough, East Yorkshire.  
 Joyson, F., Doncaster Road, Barnsley, Yorks.  
 Julien, Alfred, Campagne des Lions, Chemin de Morgion,  
 Mazargues, Marseilles, France.  
 Justice, P. M., 14, Southampton Buildings, Chancery Lane,  
 London, W.C.

## K

Kalle, Dr. Wm., Biebrich-am-Rhein, Germany.  
 Kater, R. McCulloch, Nobel's Explosives Co., Stevenston,  
 Ayrshire, N.B.  
 Kathreiner, Franz, Worms a/Rhein, Germany.  
 Kaufmann, Dr. Herbert M., 1325, Franklin Street, Phila-  
 delphia, Pa., U.S.A.  
 Kawakita, Michitada, Imperial College of Engineering,  
 Tokyo, Japan.  
 Kay, H. A., 71, Maida Vale, London, W.  
 Kay, Dr. Percy, Lilymount, Manningham, Bradford; and  
 (Journals) 196, Heerenstraat, Leiden, Holland.  
 Kay, W. E., Gowanbank, Busby, near Glasgow.  
 Kearns, H. W., Baxenden, near Acerington.

- Keen, Austin, Technical School, Huddersfield.  
 Keiser, E. H., Bryn Mawr College, Bryn Mawr, Pa., U.S.A.  
 Kellie, Jas. M., Straiton Oil Works, Loanhead, near Edinburgh.  
 Kellner, Dr. Wm., 13, Victoria Road, Old Charlton, S.E.  
 Kempson, John F., Pye Bridge Chemical Works, near Alfreton, Derbyshire.  
 Kenrick, Prof. Edgar B., St. John's College, Winnipeg, Manitoba, Canada.  
 Kent, Wm. J., P.O. Box 294, Johannesburg, South African Republic.  
 Kenyon, Thos., The Shrubbery, Hilton Park, Prestwich, near Manchester.  
 Ker, Alan D., Millburn Chemical Works, Garngad Hill, Glasgow.  
 Kerr, Saml. T., c/o Alex. Kerr Bros. & Co., Philadelphia, Pa., U.S.A.  
 Kershaw, J., Grease, Varnish, and Cement Works, Hollinwood, near Oldham.  
 Kestner, Paul, 40, Boulevard Vanban, Lille, France.  
 Keys, W. H., Hall End Chemical Works, West Bromwich.  
 Kibble, W. Oakes, Tavern Street, Stowmarket, Suffolk.  
 Kinch, E., Royal Agricultural College, Cirencester.  
 King, A. J., Ingersley Vale Bleachworks, Bollington, Macclesfield.  
 King, C. M., 21, Godliman Street, London, E.C.  
 King, C. M., Campsie Alum Works, Lennoxton, N.B.  
 King, Sir James, Bart., 115, Wellington Street, Glasgow.  
 King, Jas. E., The Know Mill Printing Co., Ltd., Entwistle, near Bolton.  
 King, J., Falconer, Russell Place, Edinburgh, N.B.  
 King, J. T., Clayton Square, Liverpool.  
 King, Robt., 115, Wellington Street, Glasgow.  
 King, Walter, 1, Florence Villas, Hadleigh Road, Southend.  
 Kingston, J. C., Devonshire House, Hunter Road, Westcombe Park, S.E.  
 Kingsford, T. P., Oswego, New York, U.S.A.  
 Kingzett, C. T., Elmstead Knoll, Chislehurst, Kent.  
 Kinnicutt, Prof. L. P., 77, Elm Street, Worcester, Mass., U.S.A.  
 Kipping, Dr. F. Stanley, Central Institution, Exhibition Road, London, S.W.; and (Journals) 7, Milborne Grove, South Kensington, S.W.  
 Kirk, Edw. C., Lock Box 1615, Philadelphia, Pa., U.S.A.  
 Kirkham, Thos., 22, Leinster Gardens, Runcorn, Cheshire.  
 Kirkman, R., Landore Alkali Works, Swansea.  
 Kirkpatrick, A. J., 179, West George Street, Glasgow.  
 Kitamura, Y. (Journals), c/o R. Fujihama, Yokoyamacho Sancho, Tokyo, Japan.  
 Kitchen, Theo. (Journals), 28, Flinders Lane North, Melbourne, Australia; and (subs.) Messrs. J. Connell & Co., Dunster House, E.C.  
 Kitson, Sir James, Bart., M.P., Gledhow Hall, Leeds.  
 Kitto, B., 26, Lancaster Road, Finsbury Park, London, N.  
 Kleemann, Dr. S., 25, Greenfield Road, Stonecroft, Liverpool.  
 Klipstein, A., 122, Pearl Street, New York, U.S.A. (P.O. Box 2433).  
 Klonowski, W. T. von, P.O. Box 64, Krugersdorf, S.A.R.  
 Knaggs, Alfred B., Springfield Mill, Morley, near Leeds.  
 Knecht, Dr. E., 239, Moss Lane East, Manchester.  
 Knight, A. H., 2, Gerald Road, Oxtou, Cheshire.  
 Knight, Henry, 33, Faraday Street, Breck Road, Liverpool.  
 Knight, J. B., Silvertown Soapworks, Silvertown, London, E.  
 Knight, J. J., Gorton Brook Chemical Works, West Gorton, near Manchester.  
 Knights, J. West, County Laboratory, 1, Sidney Street, Cambridge.  
 Knipler, F., c/o R. Harper and Co., 352, Flinders Lane, Melbourne, Victoria.  
 Knoertzer, Henri, "Le Nickel," 13, Rue Lafayette, Paris.  
 Knowles, Joshua, Stormer Hill, Tottington, near Bury.  
 Knox, E. W., Colonial Sugar Refining Co., Sydney, N.S.W.; and c/o F. Parbury & Co., 7, East India Avenue, Leadenhall Street, London, E.C.  
 Koechlin, Horace, 19, Avenue du Mont Riboudet, Rouen, France; and (subs.) c/o Koechlin, Baumgartner and Co., 79½, Watling Street, E.C.  
 Kohn, Dr. Chas. A., University College, Brownlow Street, Liverpool.  
 Kolb, J., Soc. Anon. des Manuf. de Produits Chimiques, Lille, France.  
 Koningh, L. de, 325, Kennington Road, S.E.  
 Kraftmeier, E., 55, Charing Cross, London, S.W.  
 Kranske, Dr. Albert H., 132, Woodland Avenue, Cleveland, Ohio, U.S.A.  
 Krause, Dr. G., "Chemiker-Zeitung," Cöthen, Germany.  
 Kranske, O. H., Box L, Jersey City, New Jersey, U.S.A.  
 Krohn, F. W. T., 3, Milton Villas, Harrow.  
 Kuhl, W. H., 73, Jägerstrasse, Berlin, Germany.  
 Kunheim, Dr. Hugo, 32, Doratheenstrasse, Berlin.  
 Kupferberg, Dr. H., 303, Collyhurst Road, Manchester.  
 Kynaston, J. W., 3, Oak Terrace, Beech Street, Liverpool.
- L**
- Lacey, E. C., 126A, Bermondsey Street, London, S.E.  
 Lacey, T. S., Gas Light and Coke Company, Lupus Street, Pimlico, S.W.  
 Lagerwall, Dr. Ivar, 63, Rue de Provence, Paris.  
 Laidler, C. P., 26, Noble Terrace, Gateshead-on-Tyne.  
 Laidler, T. S., Newcastle Chemical Works, Gateshead.  
 Laing, Jno., 3, Montone Terrace, Edinburgh.  
 Lake, D. E., 36, Mark Lane, London, E.C.  
 Lake, G., jun., 83, Primrose Lane, Glossop, Derbysire.  
 Lambert, Alan, Union Oil Mills, Ltd., 18, Bishopsgate Street Within, E.C.  
 Lampray, R. H., 24, Burghley Road, Highgate Road, London, N.W.  
 Lander, Albert H., 164, Edmund Street, Birmingham.  
 Lang, Jas. G., 5, Viewfield Terrace, Hillhead, Glasgow.  
 Lang, W. R., 9, Crown Gardens, Dowanhill, Glasgow.  
 Langbeek, H. W., The Park, Loughton, Essex.  
 Langdon, Dr. M. J., 3, Cooper Street, Manchester.  
 Lange, Dr. Martin, Amersfoort, Holland.  
 Langenbeck, Karl, c/o The A. E. Tiling Co., Zanesville, Ohio, U.S.A.  
 Langer, Dr. Carl, Eaton House, Willis Road, Leamington.  
 Larkin, T., St. Bede Chemical Works, South Shields.  
 Larned, J. N., Young Men's Library, Buffalo, N.Y., U.S.A.  
 Larsen, John, 18, Pine Street, Chicago, Ill., U.S.A.  
 Lascelles, Jno. H., Lynwood Villa, Beaconsfield Road, New Southgate, N.  
 Lassing, Henry, M.D., 19, Park Place, New York, U.S.A.  
 Latham, Baldwin, 13, Victoria Street, Westminster, S.W.  
 Latham, J. J., 157, Albert Road, Appleton, Widnes.  
 Laurie, A. P., 49, Beaumont Square, London, E.  
 Law, A. E., Donald's Chlorine Co., Ltd., Kilwinning, N.B.  
 Lawrance, H. A., 28, Grosvenor Road, Gunnersbury.  
 Lawrence, Jas., Repauno Chemical Co., Hannibal, Mo., U.S.A.  
 Laws, J. P., 32, Holborn Viaduct, London, E.C.  
 Lawson, Arthur J., Marsh Soapworks, Bristol.  
 Lawson, Dr. Thos. A., 15, Alexandra Road, London, N.W.  
 Lawton, Thos., Calthorpe House, Aldridge Road, Perry Bar, Birmingham.  
 Laycock, Dr. W. F., 2, Park Street, Dewsbury.  
 Lazarus, M. J., See Langdon, M. J.  
 Leach, Walter, 27, St. Andrew's Place, Bradford, Yorks.  
 Leathart, J., Lead Works, Newcastle-on-Tyne.  
 Leather, Dr. J. W., Dehra Dun, N.W.P., India.  
 Le Bouillier, Clement, c/o Taylor Iron and Steel Co., High Bridge, N.J., U.S.A.  
 Ledoff, Prof. A., Technological Institute, Kharkoff, Russia.  
 Lee, C. Tennant, 146, Franklin Street, Boston, Mass., U.S.A.  
 Lee, J. E.  
 Lee, J. W., Richmond, 70, St. Helens Gardens, North Kensington, W.  
 Lee, S. Wright, 6-10, Whitechapel, Liverpool.  
 Lee, Theo. H., Edgcombe Villa, Clevedon, Somerset.  
 Leech, F. S., 32, Plain Street, Cape Town, S. Africa.  
 Leeds, F. H., 26, East Bank, Stamford Hill, N.  
 Leese, Joseph, 3, Lord Street West, Southport.

- Leete, Jos., 19—25, Hermondsey Street, S.E.  
 Lettmann, Dr. H., 715, Walnut Street (Third floor front), Philadelphia, Pa., U.S.A.  
 Leigh, Cecil, Adderley Park Rolling Mills, Birmingham.  
 Lennard, F., Enfield Lodge, St. John's Road, Blackheath, S.E.  
 Lennox, Robt. N., Royal Institution, Albemarle Street, London, W.  
 Leonard, Wm. J., Hope Chemical Works, Hackney Wick, E.  
 Leon, J. T., 38, Portland Place, London, W.  
 Lequin, E., 9, Rue Ste. Cécile, Paris.  
 Lester, J. H., 51, Arcade Chambers, St. Mary's Gate, Manchester.  
 Lett, Stephen J., 25, Percy Street, Liverpool.  
 Lever, Jas. D., Port Sunlight, near Birkenhead.  
 Lever, Wm. H., Thornton House, Thornton Haugh, Cheshire.  
 Levinstein, Ivan, 21, Minshull Street, Manchester.  
 Lewes, Prof. Vivian B., Royal Naval College, Greenwich, S.E.  
 Lewinton, B., 14, Cleveland Street, Fitzroy Square, London, S.W.  
 Lewis, A. E., 94, Tritonville Road, Sandymount, Dublin.  
 Lewis, G. T., Room 50, S.E. corner, 4th and Walnut Streets, Philadelphia, Pa., U.S.A.  
 Lewkowsch, Dr. Julius, Whitehall Soapworks, Leeds.  
 Lichtenstein, Theodore, Chemical Works, Silvertown, London, E.  
 Liddle, W. T., Carr Bank, Walmersley, Bury.  
 Liddle, G. A., Carr Bank, Walmersley, Bury.  
 Liebert, Dr. M., c/o R. Dewhurst and Co., Lim., Birstal, near Leeds.  
 Liebmann, Dr. A., 61, Marsden Street, Manchester.  
 Liepmann, Dr. H., c/o Cracker Creek G.M. Co., Enreka, Baker Co., Oregon, U.S.A.  
 Lightfoot, T. E., 88, Arden Terrace, Acerington.  
 Lilly, Oliver M., The Croft, Spondon, Derby.  
 Limpach, Dr. L., Schweinfurt, Germany.  
 Lindemann, Dr. G., Thalstrasse 614, St. Pauli, Hamburg.  
 Lindley, Hubert E., 13, Dalmeny Avenue, Camden Road, London, N.  
 Lineff, A. L., c/o Goldenberg, 51, Tribune Buildings, New York, U.S.A.  
 Ling, Arthur R., 45, Lambton Road, Cottenham Park, Wimbledon.  
 Lishman, W. W. L., 36, Washington Street, Girlington, Bradford, Yorks.  
 Lister, Simeon, 70, High Street, Great Horton, Bradford, Yorks.  
 Little, Wm. G., Blendon Grove, Boxley, Kent.  
 Littlejohn, J., c/o African Banking Corporation, Johannesburg, S.A.R.  
 Liversidge, A. J., c/o Mirrlees, Watson, and Yaryan Co., Ltd., 45, Scotland Street, Glasgow.  
 Liversidge, Prof. A., F.R.S., The University, Sydney, New South Wales; and (Journals) c/o Kegan, Paul, & Co., 20—22, Charing Cross Road, W.C.  
 Livesey, Frank, South Metropolitan Gas Co., 709A, Old Kent Road, London, S.E.  
 Livingston, W. J., London County Council, Spring Gardens, London, S.W.  
 Lloyd, Fred. J., Agricultural Laboratory, 4, Lombard Court, London, E.C.  
 Lloyd, Herbert, c/o Electric Storage Battery Co., Drexel Building, Philadelphia, Pa., U.S.A.  
 Lockwood, Jos., 263, Argyle Street, Glasgow.  
 Lodge, A. S., Newchurch, near Manchester.  
 Lodge, Edw., 94, Bradford Road, Huddersfield.  
 Loewenthal, Dr. R., Steglitzerstrasse 56, Berlin, W.  
 Lomas, T., Cleveland, Minehead, Taunton, R.S.O.  
 Lombard, Emile, 10, Rue Breteuil, Marseilles, France.  
 Loughshaw, Jas., 3, Church Road, Scaforth, near Liverpool.  
 Lord, F. J., Brooklands, Millbrook, Stalybridge.  
 Lorenz, H., 7 and 8, Idol Lane, London, E.C.  
 Lorimer, J., Britannia Row, Islington, N.  
 Lorrain, J. G., Norfolk House, Norfolk Street, Strand, London, W.C.  
 Losanitsch, Prof. S. M., Belgrade, Servia.  
 Lott, F. E., The Laboratory, Bridge Chambers, Burton-on-Trent.  
 Loughton, J. P., Washington Chemical Works, co. Durham.  
 Louis, D. A., 77, Shurland Gardens, London, W.  
 Love, Dr. E. G., 69, East 54th Street, New York, U.S.A.  
 Lovett, W. Jesse, Sydney House, St. Anne's-on-Sea.  
 Lovibond, J. W., 26, St. Ann's Street, Salisbury.  
 Lovibond, T. W., Tyne Brewery, Newcastle-on-Tyne.  
 Lovibond, V. L., The Hermitage, North End, Fulham, S.W.  
 Low, Wilson H., c/o N. K. Fairbank & Co., 18th and Blackwell Streets, Chicago, Ill., U.S.A.  
 Lowe, C. W., Summerfield House, Reddish, near Stockport.  
 Lowe, Horace A., Halliwell Bleachworks, Smithills, near Bolton.  
 Lowe, Jas. S., Britannia Estate, Riviere Dragon, Mauritius.  
 Lowe, W. F., 9, Hough Green, Chester.  
 Lowman, Dr. Oscar, 124, Brush Street, Detroit, Mich., U.S.A.  
 Lowson, J. G. F., Beltonford Paper Mill, Dunbar, N.B.  
 Lucas, Bernard R., 32, Borough Road West, Middlesbrough.  
 Lucas, R. J., Alwinenstrasse 11, Wiesbaden, Germany.  
 Luck, A., Powder Mill, Dartford, Kent.  
 Luck, E., 68, Sumner Street, Southwark, S.E.  
 Ludlow, Lionel, c/o Cape Copper Mining Co., Ookiep, Namaqualand, South Africa.  
 Lund, Jas., 142, Hawthorne Street, Malden, Mass., U.S.A.  
 Lundholm, Carl O., Ardeer Factory, Stevenston, Ayrshire.  
 Lunge, Dr. G., Englisches Viertel, Hottingen, Zurich, Switzerland.  
 Lunn, C., 3, Osborne Road, Anfield, Liverpool.  
 Lupton, Sydney, Grove Cottage, Roundhay, Leeds.  
 Luthy, Edmund O., c/o Mellwood Distillery Co., Louisville, Ky., U.S.A.  
 Lüthy, Otto, c/o American Alumina Co., Barberton, Ohio, U.S.A.  
 Lye, W. T., Brachead, Cromwell Road, Luton, Beds.  
 Lyle, James, Plaistow Wharf, North Woolwich Road, London, E.  
 Lyle, Jno., 21, Mincing Lane, London, E.C.  
 Lyon, J. G., The Aire Tar Works, Knottingley, Yorks.  
 Lyte, F. Maxwell, 60, Finborough Road, Redcliffe Square, London, S.W.  
 Lytle, A. M., North of Ireland Chemical Co., Belfast.

## M

- Mabery, Prof. Chas. F., 9, Rockwell Street, Cleveland, Ohio, U.S.A.  
 Macadam, C. T., 116, Fenchurch Street, London, E.C.  
 Macadam, Herbert E., The Lake, Snarebrook, Essex.  
 Macadam, Dr. Stevenson, Surgeons' Hall, Edinburgh.  
 Macadam, Prof. W. Ivion, Surgeons' Hall, Edinburgh.  
 McAlister, R., Lawes' Chemical Manure Co., Limited, Barking Creek, Essex.  
 Macallan, J., 3, Charlemont Terrace, Clontarf, Dublin.  
 Macalpine, G. W., Parkside, Acerington.  
 Macara, Thos., jun., 6, West Bank Terrace, Hillhead, Glasgow.  
 McArthur, Jno., 196, Trinity Road, Wandsworth Common, S.W.  
 McArthur, J. B., Price's Patent Candle Co., Limited, Bromborough Pool, near Birkenhead.  
 Macarthur, J. G., 98, Dobbie's Loan, Glasgow.  
 McArthur, J. S., Cassel Gold Extracting Co., Limited, 13, West Scotland Street, Kinning Park, Glasgow.  
 McArthur, Thos., Cairndhu, Kirklee Road, Glasgow.  
 McBeath, J. W., c/o H. McBeath, Greatham Terrace, West Hartlepool.  
 McBretnay, E. G., Pontefract Road, Castleford, Yorks.  
 McCallum, J. M., South Park, Paisley, N.B.  
 McCalman, D., Waterside, Irvine, N.B.  
 McCann, Owen, 158, Tredegar Road, Bow, E.  
 McCombie, C., c/o C. Gross and Co., 9, Mincing Lane, London, E.C.  
 McCowan, W., The Brewery, Reading, Berks.  
 McCubbin, Wm. A., Mill Bank, West Derby, Liverpool.

- McCulloch, J., Oakleigh, Rose Street, Garnet Hill, Glasgow.  
 McCulloch, Norman, 7, Melrose Street, Glasgow.  
 McDaniel, J. J., Bandon, Ireland.  
 Macdonald, A., 72, Great Clyde Street, Glasgow.  
 Macdonald, J. W., c/o Messrs. H. Tate & Sons, Love Lane, Liverpool.  
 McDonald, T. M., Walilabo Estate, St. Vincent, West Indies.  
 McDougall, Arthur, Fallowfield House, Fallowfield, Manchester.  
 McDougall, J. T., Dunolly, Morden Road, Blackheath, S.E.  
 McElroy, Karl P., 1412, 16th Street, N.W., Washington, D.C., U.S.A.  
 MacEwan, Peter, 4, Gresley Road, Hornsey Lane, N. (Journals); and 42, Cannon Street, E.C.  
 McEwen, Atholl F., 43, Gilmore Road, Lewisham, S.E.  
 McEwen, Jas., Ruthven House, Bowden Lane, Marple, Cheshire.  
 Macfarlane, J. A., 917, Vender Street, Vancouver City, British Columbia.  
 Macfarlane, R. F.  
 Macfarlane, Thos., Inland Revenue Dept., Ottawa, Canada.  
 Macfarlane, Walter, Garnockside, Glangarnock, R.S.O., N.B.  
 Macfarlane, Walter, Holmeview, Mansefield, Pollokshaws, Glasgow.  
 Macfarlane, W. W., Riverside Mills, Chester, Pa., U.S.A.  
 McGeorge, A. J. (subs.), 78, Mount Pleasant, Liverpool; and (Journals) 69, West 88th Street, New York City, U.S.A.  
 McGhie, T. Burns, 10, St. James', New Cross, S.E.  
 McGill, Dr. J. T., Vanderbilt University, Nashville, Tenn., U.S.A.  
 McGlashan, John, Woodneuk, Gartcosh, near Glasgow.  
 McGowan, John, Ash House, Talke near Stoke-upon-Trent.  
 MacIndoe, G. D., Hall's House, North Woolwich Road, Victoria Docks, E.  
 MacIntosh, C. J., 54, Leadenhall Street, London, E.C.  
 MacKenzie, Wm., Incandescent Gas Light Co., Ltd., 14, Palmer Street, Westminster, S.W.  
 McKechnie, D., Eccleston Grange, Prescot, Lancashire.  
 McKechnie, D. M., Greystone House, Penketh, near Warrington.  
 McKellar, W. G., c/o United Alkali Co., Ltd., Eglington Works, Irvine, N.B.  
 McKenny, C., Railway Cottage, Dublin Road, Drogheda, Ireland.  
 Mackenzie, Dr. G. S., Sydney Smelting Works, Pyrmont, Sydney, N.S.W.  
 Mackenzie, Jas., 24, Shuttle Street, Glasgow.  
 Mackenzie, T. E., Dalziel Iron and Steel Works, Motherwell, N.B.  
 Mackenzie, Dr. W. Cossar, Tewfikieh College of Agriculture, Ghizeh, Egypt.  
 McKerrrow, C. A., 41, Eccles Old Road, Pendleton, Manchester.  
 McKesson, John, 91, Fulton Street, New York City, U.S.A.  
 Mackey, W. McD., Victoria Chambers, Leeds.  
 McKillop, Jno., Puloah Brani Smelting Works, Singapore.  
 McKinlay, R. W., Ameliaville, Aytoun Road, Pollokshields, N.B.  
 Mackinnon, A. K., 108, Oxford Gardens, London, W.  
 MacLagan, R. C., 5, Coates Crescent, Edinburgh.  
 MacLean, Alex. S., 31, Bank Street, Greenock, N.B.  
 McLellan, J. Y., 34, Friedrich Strasse, Bernburg, Anhalt, Germany.  
 McLeod, Prof. H., F.R.S., Cooper's Hill, Staines.  
 McLeod, Jas., Alpine Villa, Cardross Road, Dumharton, N.B.  
 McMillan, W. G.  
 McMurtrie, J. M., 21, Princes Street, Pollokshields, Glasgow.  
 Macnab, C., Lillyburn, Milton of Campsie, N.B.  
 Macnab, W., jun., 14, Great Smith Street, Westminster, S.W.  
 Macnair, D. S., People's Palace Technical Schools, Mile End, E.  
 McNeight, W. J., Earlwell, Palmerston Park, Dublin.  
 Macnochie, J. R., Saracen's Head Buildings, Snow Hill, E.C.  
 McRoberts, G., Todhill, Newton-Mearns, Renfrewshire, N.B.  
 Maetear, J., 2, Victoria Mansions, Westminster, S.W.  
 McVie, Jas. P., Tennant's Works, Hebburn-on-Tyne.  
 Madden, W. J. H., 7, Montalto, South Parade, Belfast, Ireland.  
 Mahon, R. W., 1221, East Preston Street, Baltimore, Md., U.S.A.  
 Major, J. C., The Bhylls, Compton, Wolverhampton.  
 Major, L., Sencleates, Hull.  
 Makin, James, Wallhead Mills, Rochdale.  
 Mallafien, Thos. C., Anlage, 24 C., Heidelberg, Germany.  
 Mallinekrodt, Edw., Mallinekrodt Chemical Works, St. Louis, Missouri, U.S.A.  
 Mander, S. T., 17, Gracechurch Street, London, E.C.; and (Journals) Varnish Works, Wolverhampton.  
 Manbes, P., 3, Rue Sala, Lyons, France.  
 Mann, Harold H., 7, Minster Yard, York.  
 Maun, John C., 19, New City Road, Plaistow, E.  
 Manning, F. A., 18, Billiter Street, London, E.C.  
 Manning, Isaac H., c/o Chesapeake Guano Co., Baltimore, Md., U.S.A.  
 Mannington, H. T., Grove House, Halkyn Street, Flint, North Wales.  
 Mansbridge, Wm., 21, Rosenau Crescent, Battersea, S.W.; and (Journals), c/o Goodwin Manufacturing Co., St. Louis, Mo., U.S.A.  
 Marchlewski, Dr. L., 15, Peru Street, Higher Broughton, Manchester.  
 Markel, Dr. K., Lodge Lane, Warrington.  
 Markham, A. D., 71, Queen Street, Hull.  
 Marks, E. G., Belleville, N.J., U.S.A.  
 Marley, J. E., Hebburn-on-Tyne.  
 Marsh, W., Union Alkali Co., Soho Works, Manchester.  
 Marsh, J. T., British Alkali Works, Widnes.  
 Marsh, Walter, Mysore West Gold Co., Lim., Oorgaum, Mysore, South India.  
 Marshall, Dr. Hugh, Chemistry Department, The University, Edinburgh.  
 Marshall, John, Messrs. Marshall, Son, & Co., Cudbear Street, Hunslet, Leeds.  
 Marshall, Prof. T. Rymer, St. Mungo's College, Glasgow.  
 Marshall, Wm., 15, West Street, Rochdale.  
 Marshall, Wm. (Journals), c/o D. G. Rose, Samarang, Java; and (subs.) c/o D. R. Calder Marshall, 49, Queen Street, Edinburgh.  
 Martin, Alex. M., Douglas Villa, Dunbeth Road, Coatbridge, N.E.  
 Martin, Chas. D., 106, Addison Road, Heaton, Newcastle-on-Tyne.  
 Martin, H., Poole, near Wellington, Somerset.  
 Martin, N. H., 29, Mosley Street, Newcastle-on-Tyne.  
 Martin, W. H., 183b, King's Road, Chelsea, London, S.W.  
 Martindale, Wm., 19, Devonshire Street, Portland Place, W.  
 Martineau, Sydney, South Road, Clapham Park, S.W.  
 Martino, F. W., 107, Montgomery Road, Sharrow, Sheffield.  
 Martins, Dr. C. A., 28, Vosse Strasse, Berlin, Germany.  
 Martyn, T. Graham, Trevemper Bridge, New Quay, Cornwall.  
 Martyn, W., Tennant's Works, Hebburn-on-Tyne.  
 Mason, A. H., Seabury Buildings, 59-61, Maiden Lane, New York City, U.S.A.  
 Mason, J., Eynsham Hall, Witney, Oxon.  
 Mason, J., Francis, Eynsham Hall, Witney, Oxon.  
 Mason, Thos., Hyson Green Works, Nottingham.  
 Mason, W. B., 117, Derby Street, Bolton-le-Moors.  
 Masson, Prof. D. Orme, University of Melbourne, Victoria, Australia.  
 Master, Ardesheer B., 679, Tardeo, Bombay, India.  
 Mather, Colin, Salford Iron Works, Manchester.  
 Mather, J., Blaydon Chemical Works, Blaydon-on-Tyne.  
 Matheson, W. J., 178, Front Street, New York, U.S.A.  
 Mátos, Louis J., 3913, Fairmount Avenue, Philadelphia, Pa., U.S.A.  
 Matthews, C. G., Laboratory, Bridge Chambers, Burton-on-Trent.  
 Mawdsley, W. H., 8, Eaton Road, Chester.  
 Maxwell, Thos., 49, Harvie Street, Bridgeton, Glasgow (Journals); and Mosknove, Braeside Avenue, Rutherglen.

- Mayenfeld, Dr. E. von Salis. See under "Salis."
- Mayer, Dr. Nelson B., 215, North 8th Street, Philadelphia, Pa., U.S.A.
- Mayer, Wm. Turner, c/o Albany Chemical Co., 65-67, Green Street, Albany, N.Y., U.S.A.
- Mayfield, H. B., Heanor, near Nottingham.
- Mayhew, E. W. A., High Street, Freemantle, Western Australia.
- Meacham, Chas. S., Brewery House, Earl Street, Maidstone.
- Mead, Frank, Sutton Gasworks, Surrey.
- Meggitt, H. A., Chemical Works, Mansfield, Notts.
- Meikle, Jno., 4, Woodlands Road, Glasgow.
- Meldola, Prof. R., F.R.S., 6, Brunswick Square, London, W.C.
- Meldrum, Jas. Jones, Atlantic Works, City Road, Manchester.
- Mellen, Edwin D., c/o Curtis, Davis, & Co., 184, Broadway, Cambridgeport, Mass., U.S.A.
- Melliss, J. C., 232, Gresham House, Old Broad Street, London, E.C.
- Mellon, W. W., Howdenlyke, Howden, Yorks.
- Mellor, S., Magnesium Metal Co., Patricroft, Manchester.
- Melville, D., P.O. Box No. 1, Woodmere, Wayne Co., Mich., U.S.A.
- Mendeleeff, Prof. D., Cadet Line 9, Vassilieff Island, St. Petersburg.
- Menges, Dr. Franklin, Penna. College, Gettysburg, Pa., U.S.A.
- Mensching, Dr. C., Crumpsall Vale Works, Blackley, near Manchester.
- Menzies, R. C., Inveresk Mills, Musselburgh, N.B.
- Mercer, C. A., Dagmar Lodge, Cheam Road, Sutton, Surrey.
- Mercer, J. B., 330, Lower Broughton Road, Manchester.
- Mercer, F. M., 89, Bishopsgate Street, London, E.C.
- Mercer, Thos., The Brewery, Edenfield, near Bury, Lancashire.
- Merek, E., Darmstadt, Germany.
- Merrell, Geo., Lock Box 786, Cincinnati, Ohio, U.S.A.
- Messel, Dr. R., Silvertown, London, E.
- Metcalf, Jno., Moorfield, Altham, near Accrington.
- Metcalf, Wm., Aspin House, Oswaldtwistle, near Accrington.
- Mewburn, J. C., 55 and 56, Chancery Lane, London, W.C.
- Meyer Max E., 62, Corso Venezia, Milan, Italy.
- Meynier, J. A., 19, Rue Baudin, Paris.
- Milestone, W. C., Garrett Lane, Wandsworth, S.W.
- Miller, Dr. A. K., Ardwick Brewery, Manchester.
- Miller, A. Russell, The Cairns, Cambuslang, near Glasgow.
- Miller, E. V., c/o New Zealand Sugar Co., Auckland, New Zealand.
- Miller, Geo., c/o Widnes Alkali Co., Ltd., Widnes.
- Miller, Dr. Harry E., 1264, 14th Street, Oakland, Cal., U.S.A.
- Miller, Hugh L., 414, North Front Street, Wilmington, North Carolina, U.S.A.
- Miller, Dr. H. von, Beatrixgasse 32<sup>III</sup>, Vienna, Austria.
- Miller, Jno., Messrs. Jas. Black & Co., 23, Exchange Square, Glasgow.
- Miller, J. Carlie, 89, Rumford Street, Bridgeton, Glasgow.
- Miller, J. Hopkins, 23, McAshm Street, Glasgow.
- Miller, Jno. Poynter, Sandilands Chemical Works, Aberdeen.
- Miller, Dr. N. H. J., Harpenden, near St. Albans.
- Miller, T. Paterson, The Cairns, Cambuslang, near Glasgow.
- Miller, W. M., Pln. De Willem, Demerara, West Indies.
- Mills, Prof. E. J., F.R.S., 60, John Street, Glasgow.
- Milner, E., Hartford Manor, Northwich.
- Milnes, Edmund, Seedfield, Bury, Lancashire.
- Milnes, J. M., c/o G. C. Whitfield, 1, Hardshaw Street, St. Helens.
- Miniati, T., Kenwood, Broom Lane, Higher Broughton, Manchester.
- Mitchell, J. W., Wood Leigh, Clough Fold, near Manchester.
- Mitting, E. K., 423, Superior Street, Chicago, Ill., U.S.
- Moffatt-Johnston, J., The Birches, Midcalden, N.B.
- Mohr, Dr. B., 69A, Parliament Hill, Hampstead, N.W.
- Molesworth, F. H., Eton Street, Malvern, Adelaide, South Australia.
- Molineux, John, C.B., Selsley House, Albert Road, Battersea, S.W.
- Molmeux, Roland, 6, S. Jersey Street, Newark, N.J., U.S.A.
- Mond, Alf. M., 20, Avenue Road, Regent's Park, N.W.
- Mond, L., F.R.S., 20, Avenue Road, Regent's Park, London, N.W.; and 64, Via Sistina, Rome.
- Mond, Robt. L., 20, Avenue Road, Regent's Park, N.W.
- Moodie, W. E., Crofting Work, Alexandria, N.B.
- Moody, Samuel J., Casilla 288, Buenos Ayres, Argentina.
- Mook, Chas., Douglasshall, Westeregeln, Magdeburg, Germany.
- Mooney, M., Chemical Works, 74, Rogerson's Quay, Dublin.
- Moore, B. T., Longwood, Bexley, Kent.
- Moore, Chas. C., 125, Chester Road, Hartford, Cheshire.
- Moore, Dr. Geo. D., 25, Catherine Street, Worcester, Mass., U.S.A.
- Moore, R. T., 156, St. Vincent Street, Glasgow.
- Moore, Thos., "Le Nickel," Noumea, New Caledonia.
- Moore, Thos., Northwich, Cheshire.
- Moore, Wm. F., Lonsdale, Temple Road, Upper Rathmines, Dublin.
- Moorhouse, J. B., Cecil Mount, Horton Park, Bradford.
- Mordle, F. Dare, Fishpond Drive, The Park, Nottingham.
- Morgan, Albert J., 489, West Street, New York City, U.S.A.
- Morgan, Arthur F., Springfield, Carleton Road, Newport, Mon.
- Morgan, Jno. Jas., Milton Lodge, Brecon Road, Abergavenny.
- Morgan, Dr. Wm., Public Analyst's Laboratory, Nelson Terrace, Swansea.
- Morgans, Thos., The Guildhall, Bristol.
- Moritz, Dr. E. R., 72, Chancery Lane, London, W.C.
- Morley, Dr. H. Forster, 47, Broadhurst Gardens, South Hampstead, N.W.
- Morrell, Jno. B., Holdgate House, York.
- Morrell, Robt., Elmtree, Hooton, near Chester.
- Morrice, Jas. A., 1, Athole Gardens Place, Kelvinside, Glasgow.
- Morris, Dr. G. Harris, 72, Chancery Lane, London, W.C.
- Morris, Herbert N., 52, Manley Road, Manchester.
- Morris, J. H., 63 and 65, Blundell Street, Liverpool.
- Morris, R., Doncaster.
- Morrison, Geo. R., Richmond House, Plaistow, Essex.
- Morrison, J., St. Peter's Chemical Works, Newcastle-on-Tyne.
- Morson, T., 124, Southampton Row, Russell Square, London, W.C.
- Morson, T. P., 33, Southampton Row, Russell Square, London, W.C.
- Morton, Jas., Dalquhurn Works, Renton, N.B.
- Mosenthal, Henry de, 220, Winchester House, Old Broad Street, London, E.C.
- Moss, J., Wilson Street, New Cross Road, London, S.E.
- Moul, Frank, Aldersgate Chemical Works, Southall.
- Moult, J., Underhill, Low Fell, Gateshead-on-Tyne.
- Moulton, G. L., Soho Mills, Macclesfield.
- Mount, Edw., Oaklands, Aughton, near Ormskirk.
- Muir, J. P., 233, Camden Road, London, N.W.
- Muir, Jas. Stanley, Chemical Laboratory, The University, Glasgow; (Journals) to 27, Huntley Gardens, Kelvinside, Glasgow.
- Müller, Geo., c/o Puget Sound Reduction Co., Everett, Washington, U.S.A.
- Müller, Dr. H., F.R.S., 13, Park Square East, Regent's Park, London, N.W.
- Mummery, Albert F., 5, Castlemount Terrace, Dover.
- Munro, Dr. J. M. H., Churebfields, Salisbury.
- Munroe, Prof. Chas. E., Columbian University, Washington, D.C., U.S.A.
- Murdoch, H. R. M., 4, Nobel's Villas, Stevenston, Ayrshire.
- Muspratt, E. K., Seaforth Hall, near Liverpool.
- Muspratt, S. K., 24, Grove Park, Liverpool.
- Muter, Dr. J., Winchester House, Kennington Road, London, S.E.
- Myers, Wm. S., 98, Easton Avenue, New Brunswick, N.J., U.S.A.

## N

- Nadiein, Capt. M., 7, Dumskaya, St. Peter-burg, Russia.  
 Naf, Dr. P., Argentine, near Kansas City, Kansas, U.S.A.  
 Nahsen, Dr. R., Dömitz, Elbe, Germany.  
 Nakamura, Teikichi, 154, Shirokane-Sankocho, Shibaku, Tokyo, Japan.  
 Napier, J., 89, London Road, Ipswich.  
 Napier, Jas., 15, Prince's Square, Strathbungo, Glasgow.  
 Napier, Jno. W., Clippens Oil Works, Loanhead, near Edinburgh.  
 Nason, Prof. H. B., Troy, New York, U.S.A.  
 Naylor, Wm., 16, Walton's Parade, Preston, Lancashire.  
 Naylor, W. A. H., 38, Southwark Street, London, S.E.  
 Neill, Geo. D., 26, Forsyth Street, Greenock, N.B.  
 Neilson, James, 107, High John Street, Glasgow.  
 Neilson, Thos., c/o Oregon Iron and Steel Co., Oswego, Oregon, U.S.A.  
 Neilson, Wm., 23, Terrace Road, Upton Manor, E.  
 Neiman, H. S., P.O. Box 319, Albany, N.Y., U.S.A.  
 Nelson, Arthur J., c/o Clayton Aniline Co., Ltd., Clayton, Manchester.  
 Ness, T., Black Banks Chemical Works, Darlington.  
 Newall, F. S., Washington, co. Durham.  
 Newall, J. F., 8, Market Place, Manchester.  
 Newberry, Spencer B., Sandusky Portland Cement Co., Sandusky, Ohio, U.S.A.  
 Newlands, B. E. R., 27, Mincing Lane, London, E.C.  
 Newlands, W. P. R., 1, Mildenhall Road, Lower Clapton, N.E.  
 Newsholme, G. T. W., 74, Market Place, Sheffield.  
 Newton, A. H., Belsize Court, Hampstead, N.W.  
 Newton, A. H., jun., 4, Croftdown Road, Highgate Road, N.W.  
 Newton, H. C., 47, Calcott Road, Brondesbury, N.W.  
 Newton, Jno., Park Green, Macclesfield.  
 Newton, Jno., Manor Works, Rotherhithe New Road, London, S.E.  
 Nichols, J. A., Hurstfield, New Mill, near Stockport.  
 Nichols, W. H., 45—47, Cedar Street, New York, U.S.A.  
 Nicholson, J. C., Chemical Works, Hunslet, Leeds.  
 Nickolls, John B., The Laboratory, Grange, Guernsey.  
 Nicol, W. W. J., Mason College, Birmingham.  
 Nimmo, J., Peushurst, Stanger Road, South Norwood, S.E.  
 Nishigawa, T., Chemical Works, Kawa Kitamura, Osaka, Japan.  
 Nolting, Dr. E., Ecole de Chimie, Mulhouse, Alsace, Germany.  
 Norman, F. J., Lyndhurst, Higher Runcorn, Cheshire.  
 Norman, J. T., 78, Chelverton Road, Putney, S.W.  
 Norris, Wm. M., Princeton, N.J., U.S.A.  
 North, E. Gordon N., Bella Vista 14, Minas de Rio Tinto, Huelva, Spain.  
 Northing, J., 96, Tritonville Road, Sandymount, Dublin.  
 Norton, Dr. S. A., 363, East Town Street, Columbus, Ohio, U.S.A.  
 Norton, Dr. T. H., University of Cincinnati, Ohio, U.S.A.

## O

- O'Beirne, W. G., British Chemical Works, Clydebank, Glasgow.  
 Oddie, Jas., School of Mines, Ballarat, Australia.  
 Oddy, Robert W., 60, Waterhouse, Toad Lane, Rochdale.  
 Odling, Dr. W., F.R.S., 15, Norham Gardens, Oxford; and 38, Ladbrooke Grove Road, North Kensington, W.  
 Oehler, K., Offenbach-am-Main, Germany.  
 Ogata, Saburo, c/o T. Hirano, 11, Ginza Shichome, Tokyo, Japan.  
 Ogston, G. H., Junior Athenæum Club, Piccadilly, London, W.  
 Ogura, K., Army Survey Department, General Staff, Tokio, Japan.

- Oliver, F., 70, Winchester Street, South Shields.  
 Oliver, Wm. Letts, 1110, 12th Street, Oakland, Cal., U.S.A.  
 Ollershaw, S., Sutton Alkali Works, St. Helens.  
 O'Neill, E. H., Johnson's Saccharum Co., Limited, Stratford, London, E.  
 Orme, J., 65, Barbican, London, E.C.  
 Orr, A. (subs.), 19, Albion Crescent, Dowanhill, Glasgow; and (Journals) 109, Pitt Street, Sydney, New South Wales.  
 Orr, J. B., Blantyre Lodge, Westcombe Park, London, S.E.  
 Orr, Robert, 79, West Nile Street, Glasgow; and Falkirk, N.B.  
 Orsman, W. J., Roburite Explosives Co., Gathurst, near Wigan.  
 Osborne, Jas., c/o Rio Tinto Co., Ltd., 30, St. Swithin's Lane, E.C.  
 Osgood, E. R., Cossipore Sugar Factory, Calcutta, India.  
 Ostersetzer, J., Balcarra House, Serpentine Avenue, Ballsbridge, Dublin.  
 Ostlere, Edward, Messrs. Barry, Ostlere, & Co., Kirkcaldy, N.B.  
 O'Shea, L. T., Firth College, Sheffield.  
 O'Sullivan, C., F.R.S., 149, High Street, Burton-on-Trent.  
 O'Sullivan, J., 71, Spring Terrace, Burton-on-Trent.  
 Oswald, Benj. L., 71, Spring Terrace Road, Burton-on-Trent.  
 Oushkoff, P. K., Elabouga, Govt. of Viatka, Russia.  
 Overbeck, Baron Gustavus de, 23, Ryder Street, St. James', London, S.W.  
 Overtoun, Lord, 7, West George Street, Glasgow.  
 Owen, Thos., Westbury-on-Trym, Bristol.  
 Owens, Caradoc, 88, Great Clowes Street, Lower Broughton, Manchester.  
 Oxland, Robert, 32, Portland Square, Plymouth.

## P

- Packard, E., jun., Bramford, near Ipswich.  
 Paddon, A. M., Drymma, Neath, South Wales.  
 Page, F. J. M., 54, Sutherland Street, Pimlico, S.W.  
 Pagés, Albert, 34, Boulevard Henri IV., Paris.  
 Paine, Augustus G., 60, Times Building, New York, U.S.A.  
 Paine, S., 7, Exchange Street, Manchester.  
 Palmer, J., Chalkley, Box 19, Chester, Pa., U.S.A.  
 Palmer, T., c/o Mon Van Cutsem & Co., Soignies, Belgium.  
 Palmer, Thos. C., 98, Commercial Road East, London, E.  
 Park, J., c/o Steven-on, Carlile & Co., Milburn Chemical Works, Garngad Hill, Glasgow.  
 Parker, Chas. E., Vine House, Penketh, Warrington.  
 Parker, Edw., Laburnum House, Rushford Park, Levenshulme, Manchester.  
 Parker, Gerald L., The Paddocks, Eccleston, Chester.  
 Parker, Thos., Newbridge, Wolverhampton.  
 Parkinson, J., Howarth, Stretford, Manchester.  
 Pass, A. C., The Holmes, Stoke Bishop, Bristol.  
 Pasmore, Dr. F. W., 27, Kennington Park Gardens, London, S.E.  
 Paterson, Dr. Jas. H. R., 10, Millerfield Place, Edinburgh.  
 Paterson, John, Belle Isle Place, Workington, Cumberland.  
 Paton, J. M. C., Messrs. Manlove, Alliott, Fryer & Co., Nottingham.  
 Paton, W. Grant, Greenbank Alkali Co., Limited, St. Helens, Lancashire.  
 Patberg, J. C. H., Valleyfield, Helensburgh, near Glasgow.  
 Patterson, G., c/o The Maubré Saccharine Co., Ltd., Hammersmith, W.  
 Patterson, Harry J., College Park, Prince George's Co., Md., U.S.A.  
 Patterson, T. L., Messrs. J. Walker & Co., Sugar Refinery, Greenock, N.B.  
 Pattinson, H. L., jun., 7, Windsor Crescent, Newcastle-on-Tyne.  
 Pattinson, Dr. H. Salvin, 75 Side, Newcastle-on-Tyne.  
 Pattinson, J., 75, The Side, Newcastle-on-Tyne.

Pattison, J., 83, North Oswald Street, Glasgow, N.B.  
 Pattison, Percy J., 11, Park Road, West Ham, E.  
 Paul, Jus. H., 123, Palace Road, Tulse Hill, S.W.  
 Pauli, Dr., Hochst, Germany.  
 Payne, J. B., 15, Mosley Street, Newcastle-on-Tyne.  
 Peece, Frank K., Monton Grange, Keele, Manchester; and (Journals) G.P.O., Sydney, N.S.W.  
 Peacock, Saml., Finley's Post Office, N.J., U.S.A.  
 Peak, C. P., Bridgewater Chemical Works, Wigan.  
 Pearce, W., Bow Common, London, E.; and Brent House, Brentwood, Essex (for Journals).  
 Pears, Andrew, jun., Spring Grove, Isleworth.  
 Pearson, Frank P., Arnold Printworks, North Adams, Mass., U.S.A.  
 Pechiney, A. R., Salindres (Gard), France.  
 Peden, Jno. jun., Blair Terrace, Greenock, N.B.  
 Pedler, Prof. A., F.R.S., Presidency College, Calcutta, India.  
 Pedler, J. R., Woodbank, Lordship Lane, Dulwich, S.E.  
 Pemberton, Henry, jun., 1947, Locust Street, Philadelphia, Pa., U.S.  
 Pemberton, Thos., South Brook Street, Leeds.  
 Peniston, Alex. H., Lyndhurst, Theydon Bois, Essex.  
 Pennoek, J. D., c/o Solvay Process Co., Syracuse, N.Y., U.S.A.  
 Pentecost, S. J., Nottingham Road, New Basford, Nottingham.  
 Pentermann, H. T., 37, Clifton Crescent, Peckham, S.E.  
 Peplow, D. H. T., 24, Sloane Gardens, London, S.W.  
 Perkin, A. G., 8, Montpelier Terrace, Hyde Park, Leeds.  
 Perkin, Dr. W. H., F.R.S., The Chestnuts, Sudbury, Harrow.  
 Perkin, Dr. W. H., jun., F.R.S., Fairview, Wilbraham Road, Fallowfield, Manchester.  
 Perkins, T. S., 39, Garden Place, Brooklyn, N.Y., U.S.A.  
 Perry, D., Forth and Clyde Chemical Works, Kirkintilloch, N.B.  
 Pethybridge, Walter, 29, Methley Street, Kennington Cross, S.E.  
 Pettigrew, J., 6, St. Helen's Place, Bishopsgate, London, E.C.  
 Pettigrew, Robt., Tinto View, Airdrie, N.B.  
 Petty, A., Silvertown, London, E.; and (Journals) 33, Clapton Common, E.  
 Peyton, E. P., Chemical Works, Lister Street, Birmingham.  
 Philip, Arnold, 43, Onslow Road, Richmond, Surrey.  
 Phillips, A. G., 18, Fopstone Road, South Kensington, S.W.  
 Phillips, George Brinton, 622, Race Street, Philadelphia, Pa., U.S.A.  
 Phillips, H., 183, Moss Lane East, Manchester.  
 Phillips, Thos., Birthright View, Cornholme, Todmorden.  
 Phipson, Dr. T. L., Laboratory, 8, Rotham Villas, Putney, London, S.W.  
 Picard, Wm., Stafford Villa, Norfolk Park, Sheffield.  
 Pick, Dr. S., Direction der Soda Fabrik, Szezakowa, Galizien, Austria.  
 Pickles, H., Prussiate Works, Droylsden, Manchester.  
 Pielsticker, Carl M., 43, Connaught Road, Harlesden, N.W.  
 Pilkington, G., Old Market Place, Bury, Lancashire.  
 Pilley, Thos. W., 33, Grove Hill Road, Denmark Hill, S.E.  
 Pinkney, Robert, 18, Bread Street Hill, London, E.C.  
 Pipe, Jas., Messrs. Wm. Henderson & Co., Irvine, N.B.  
 Pitblado, L., 69, Newhouse, Stirling, N.B.  
 Pitt, T., 16, Coleman Street, London, E.C.  
 Pittock, F. W., 15, Stratford Grove, Newcastle-on-Tyne.  
 Platts, Jno. C., 25, Ilarecourt Road, Crookesmoor, Sheffield.  
 Playfair, David J., 12, Woodside Terrace, Glasgow.  
 Pocklington, Hy., 41, Virginia Road, Leeds.  
 Pollitt, R. B., 12, Mark Lane, London, E.C.  
 Pollock, A., Dillichip Turkey-red Dyeworks, Bonhill, Dumbartonshire.  
 Pomeroy, Dr. Chas. T., 18, Pennington Street, Newark, N.J., U.S.A.  
 Pond, J. A., 99, Queen Street, Auckland, New Zealand.  
 Poole, Thos., 25, Water Street, Liverpool.  
 Pooley, T. A., 121, The Grove, Denmark Hill, S.E.  
 Pope, Frank, 40, Pearl Street, Cambridgeport, Mass., U.S.A.  
 Pope, S., Camden Works, Runcorn.

Pope, Wm. J., 43, Hartismere Road, Wallam Green, S.W.  
 Pott, W. Hamilton, 68, Sumner Street, Southwark Bridge Road, London, S.E.  
 Potter, Chas. E., Love Lane, Salt R. Refinery, Liverpool.  
 Potter, Chas. J., Heaton Hall, Newcastle-on-Tyne.  
 Potter, E. P., Hollinhurst, Bolton & Moors.  
 Potts, Joseph T., Price's Patent Gun Co., Bromborough Pool, near Birkenhead.  
 Powell, A. Ernest, 59, Egerton Road, Fallowfield, Manchester.  
 Powell, L. S., 5, Campden Hill Square, London, W.  
 Pratt, Walter E., Chemical Laboratory, Midland Railway Co., Derby.  
 Prentice, Manning, Stowmarket, Suffolk.  
 Prescott, Dr. Albert B., Ann Arbor, Mich., U.S.A.  
 Preston, Alf., 1, Market Street, Bury, Lancashire.  
 Preston, E. S., 10, Holyrood Terrace, The Hoe, Plymouth.  
 Preston, R., Hill End, Hodecombe, near Manchester.  
 Price, A. F., 524, Sacramento Street, San Francisco, Cal., U.S.A.  
 Price, T. Spiers, 15, Seething Lane, London, E.C.  
 Prinz, Dr. Otto, Sesto Fiorentino, Italy.  
 Pritchard, Edgar J., Burrows, Swansea.  
 Pritchard, W. S., Farnworth, Widnes.  
 Procter, H. R., Yorkshire Coll., Leeds; and (Journals) 4, Montpellier Terrace, Hyde Park, Leeds.  
 Procter, J. W., Skeldergate Bridge, York.  
 Procter, Miss Anne J., Free Library, Widnes.  
 Procter, B. S., 11, Grey Street, Newcastle-on-Tyne.  
 Procter, C., Government Laboratory, Somerset House, London, W.C.  
 Procter, W. W., 33, The Side, Newcastle-on-Tyne.  
 Pullar, R., Pullar's Dyeworks, Perth, N.B.  
 Pullar, R. D., Pullar's Dyeworks, Perth, N.B.

## Q

Quibell, Oliver, Magnus Lodge, Newark-on-Trent.  
 Quinke, Dr. F., Chemische Fabrik Rhénania, Stolberg bei Aachen, Germany.  
 Quinn, J. Cardwell, Kennet Wharf, 67, Upper Thames Street, E.C.

## R

Rademacher, H. A., General Delivery, Lawrence, Mass., U.S.A.  
 Rae, G., Tharsis Sulphur and Copper Co., Widnes.  
 Ramsay, Dr. W., F.R.S., University College, Gower Street, London, W.C.; Journals to 12, Arundel Gardens, W.  
 Ramsay, W., Chemical Laboratory, University College of Wales, Aberystwith.  
 Ramsden, Alf. A., 11, Fairfield Terrace, West Park Street, Dewsbury.  
 Ramsden, Edw., 11, Cecil Avenue, Horton Park, Bradford.  
 Ramsden, J., Lion Brewery, Belvedere Road, Lambeth, London, S.E.  
 Ratcliff, F. D., 71, Pershore Road, Birmingham.  
 Rawcliffe, H., Gillibrand Hall, Chorley, Lancashire.  
 Rawlins, T. Beilby, 2, Leven Street, Pollokshields, Glasgow.  
 Rawson, C., 2, Melbourne Place, Bradford.  
 Rawson, Dr. S. G., Westbourne, Westbourne Grove, West Kirby, Cheshire.  
 Ray, Wm., 33, Sloane Street, Brooks Bar, Manchester.  
 Raymond, C. W., 65, Clova Road, Forest Gate, E.  
 Rayner, J. A. E., High Wray Bank, Anneside.  
 Reade, Thos., Oakleigh, Compton, near Wolverhampton.  
 Readman, Dr. J. B., Chemical Laboratory, 4, Lindsay Place, George IV. Bridge, Edinburgh.  
 Reddrop, J., Laboratory, L. & N. W. Railway, Crewe.  
 Redfern, G. F., 4, South Street, Finsbury, London, E.C.  
 Redgate, J. G., Traffic Street, Nottingham.  
 Redwood, B., 4, Bishopsgate Street Within, London, E.C.



- Redwood, I. J., 104, Kent Street, Brooklyn, N.Y., U.S.A.  
 Redwood, Robt., 4, Bishopsgate Street Within, London, E.C.  
 Redwood, T. Horne, 2, Fisher Street, Red Lion Square, W.C.  
 Rée, Dr. A., 1, Brighton Grove, Rusholme, Manchester.  
 Reed, Albert E., Selwood, Overcliffe, Gravesend.  
 Reekie, J. A., 5, Simmondley Lane, Glossop, Derbyshire.  
 Reeks, T. H., 39, Tierney Road, Streatham Hill, S.W.  
 Reibstein, Dr. Thisko, Engelbostelerdamm 61B, Hanover.  
 Reid, Wm., jun., Bombay Dyeworks, Dadur, Bombay, India.  
 Reid, W. F., Fieldside, Addlestone, Surrey.  
 Reid, W. G., 3, Findhorn Place, Edinburgh; and Soap Works, Beaconsfield Diamond Fields, South Africa (Journals).  
 Remfry, H. H., 5, Fancy Lane, Calcutta, India.  
 Renant, F. W., The Brambles, Elmbourne Road, Tooting Common, S.W.  
 Rennie, Dr. E. H., University of Adelaide, South Australia.  
 Rennoldson, W. L., St. Bede Chemical Works, East Jarrow.  
 Reoch, R., River Point, Rhode Island, U.S.A.  
 Reynolds, Henry C., Thorncliff, Lansdown Road, Cheltenham.  
 Reynolds, Dr. J. Emerson, F.R.S., Trinity College, Dublin.  
 Reynolds, R., 13, Briggate, Leeds.  
 Rhodes, E., c/o Thos. Vickers & Sons, Widnes.  
 Rhodes, Jos., Blane Cottage, Blanefield, near Glasgow.  
 Richards, E., 1621 H. Street, Washington, D.C., U.S.A.  
 Richards, W. A., Sandbach, Cheshire.  
 Richardson, Clifford, Office of District Commissioners, Washington, D.C., U.S.A.  
 Richardson, C. T., 9, Rosetti Mansions, Cheyne Walk, Chelsea, S.W.  
 Richardson, D. B., Hartfield, Cove, Dumbartonshire, N.B.  
 Richardson, Col. J. G. F., Elmfield, Stoneysgate, Leicester.  
 Richardson, F. W., 2, Farelife Place, Bradford, Yorkshire.  
 Richardson, G. E., Howley Park, Batley, Yorks.  
 Richardson, S. M., 115, Main Street, Bonhill, N.B.  
 Richardson, Walter W., 1, Montpellier Terrace, Cliff Road, Leeds.  
 Richmond, H. D., 50, Clanricarde Gardens, Bayswater, W.  
 Richmond, W. H., Liver Alkali Co., Limited, Ditton Road, Widnes.  
 Riddell, Robert, 86, Derby Street, Burton-on-Trent.  
 Rideal, Dr. Samuel, Chemical Laboratory, St. George's Hospital, London, W.  
 Ridsdale, C. B., Hutton Grange, Guisboro', Yorks.  
 Rigby, John S., 35, Bagot Street, Wavertree, Liverpool.  
 Riker, Jno. J., 43, Cedar Street, New York City, U.S.A.  
 Riley, E., 2, City Road, Finsbury Square, London, E.C.  
 Riley, Jas., 150, Hope Street, Glasgow.  
 Riley, J. E., Arden Hall, near Acerington.  
 Riley, J., Hapton Chemical Works, Acerington.  
 Riley, Jno., Thornliebank, near Glasgow.  
 Riley, James, Laboratory, Brinseall Works, near Chorley, Lancashire.  
 Riley, W. G., Hapton Chemical Works, near Acerington.  
 Riley, Wm., Castleton, Manchester.  
 Rintoul, Wm., 43, King William Street, Greenwich, S.E.  
 Ripley, H., Bowling Dyeworks, Bradford, Yorkshire.  
 Ritchie, Robt., Shawfield Works, Rutherglen, near Glasgow.  
 Ritson, T. N., Gas Works, Kendal.  
 Rix, W. P., Doulton & Co., Lambeth Art Pottery, London, S.E.  
 Robbins, Herbert, Chemical Works, Gibraltar Walk, Bethnal Green Road, N.E.  
 Robbins, J., 147, Oxford Street, London, W.  
 Roberts, C. F., Linfitts, Delph, via Oldham.  
 Roberts, Ernest H., 15, Herndon Road, East Hill, Wandsworth, S.W.  
 Roberts, F. A., Cornbrook Chemical Works, Manchester.  
 Roberts, F. G. Adair, Lion House, Amhurst Park, Stamford Hill, N.  
 Roberts, J. H. M., Laboratory, 6, Trinity Square, Tower Hill, E.C.  
 Roberts, R. Wightwick, 22, Calle Arturo Prat, Valparaiso, Chili.  
 Robertson, Alex. A., c/o Australian Kerosene Oil Co., Ltd., Gresham Street, Sydney, N.S.W.  
 Robertson, Geo. H., 30, Hemstall Road, West Hampstead, N.W.  
 Robertson, Robt., Royal Gunpowder Factory, Waltham Abbey, E.  
 Robertson, R. A., 8, Park Street East, Glasgow.  
 Robinson, G. C., Royal Institution, Hull; and Laboratory, Bond Street, Hull (for Journals).  
 Robinson, H. H., Imperial College, Hankow, China.  
 Robinson, Jos., Farnworth, Widnes.  
 Robinson, Jno., 81, Albert Road, Widnes.  
 Robinson, Thomas, 401, West Street, Glasgow.  
 Robeson, Wm. S., 1112 Dodge Street, Omaha, Neb., U.S.A.  
 Rodda, Edw. D., 6, Gold Street, Roath, Cardiff.  
 Rodger, Edw., 1, Clairmont Gardens, Glasgow, W.  
 Rogers, Harry, 5, Stoke Newington Common, London, N.  
 Rogers, Capt. Jno. Martin, Mount Hawke, Scorrier, Cornwall.  
 Roger-on, W. J., 38, Southwark Street, London, S.E.  
 Rollin, J. C., St. Bede Chemical Co., Limited, Newcastle-on-Tyne.  
 Roques, Adolphe, 36, Rue Sainte Croix de la Bretonnerie, Paris.  
 Roscoe, Sir Henry, M.P., F.R.S., 10, Bramham Gardens, South Kensington, S.W.  
 Roscow, Jno. F., 25, Church Street, Littleborough, near Manchester.  
 Rosell, Claude A. O., U.S. Patent Office, Washington, D.C., U.S.A.  
 Ross, Alex. J. J., Tayavalla, Falkirk, N.B.  
 Ross, Arthur, 1, Glengall Road, Old Kent Road, London, S.E.  
 Ross, J. G., 30, Brownlow Street, Liverpool.  
 Rothwell, C. F. Seymour, 88, George Street, Moss Side, Manchester.  
 Rottenburg, Paul, c/o Messrs. Leisler, Bock & Co., 130, Hope Street, Glasgow, N.B.  
 Rouillard, R. A., 5, Dundonald Road, Wimbledon; and Mauritius.  
 Rowell, W. A., Hedworth Barium Works, East Jarrow.  
 Rowland, W. L., 4800, Chester Avenue, Philadelphia, Pa., U.S.A.  
 Rowntree, B. Seeborn, The Cocoa Works, York.  
 Roxburgh, J. W., Levenbank Works, Jamestown, Dumbartonshire, N.B.  
 Royle, T., Dalton House, Upton Lane, Forest Gate, E.  
 Royle, S. W., St. Andrew's Chambers, Albert Square, Manchester.  
 Royston, Ernest R., 15, Water Street, Liverpool.  
 Rudolf, Norman S., Sewan, Sarun, Bengal, India.  
 Rueff, Emil, 426—436, East 19th Street, New York City, U.S.A.  
 Ruffle, Jno., Gnoll Brook, Neath, Glamorganshire.  
 Rumble, C., Belmont Works, Battersea, London, S.W.  
 Ruscoe, Jno., Albion Works, Henry Street, Hyde, near Manchester.  
 Russell, D., Cadham, Markinch, Fife, N.B.  
 Russell, Jno., Anchor Brewery, Britten Street, Chelsea, London, S.W.  
 Russell, Dr. W. J., F.R.S., St. Bartholomew's Hospital, London, E.C.  
 Russell, Wm., Summerlee Ironworks, Coatbridge, N.B.  
 Rutter, Chas. H., The Gas Co., Hove, Sussex.  
 Ryder, C. E., c/o Messrs. Elkington, Newhall Street, Birmingham.  
 Ryland, Howard P., The Cedars, Gravelly Hill, Birmingham.

- Sach, Robt., c/o Señor César García, Honda, Rep. of Columbia, South America.  
 Sadler, A. E., Sand Hall, Ulverston, Lancashire.  
 Sadler, S. A., Middlesbrough-on-Tees.  
 Sadtler, Dr. S. P., 145, North 10th Street, Philadelphia, Pa., U.S.A.



- Saint, W. Johnston, 11, Queen's Road, Aberdeen; and  
 (Journals) Kurtheimstrasse 5, Bonn-Poppelsdorf,  
 Germany.  
 Salamon, A. G., 1, Fenchurch Avenue, London, E.C.  
 Salamon, Jno., Ralham, S.O. Essex.  
 Salis-Mayenfeld, Dr. E. von, c/o Hohenhausen & Co., Ltd.,  
 Blackley, near Manchester.  
 Salmon, E. H. R., Brazilian Extract of Meat Factory,  
 Cachoeira, Rio Grande do Sul, S. Brazil.  
 Saltmarsh, J. G., 147, Shrewsbury Street, Brooks Bar,  
 Manchester.  
 Samuel, W. Colden, 337, Norwood Road, West Norwood,  
 S.E.  
 Samuelson, Sir Bernard, Bart., M.P., F.R.S., 56, Prince's  
 Gate, London, S.W.  
 Sanderson, T. C., 53, Church Road, Norwich.  
 Sandon, R., 21, Archibald Road, Tufnell Park, N.  
 Sanford, J. Reintram, El Toro Fabrica de Jabon, Box 68,  
 Monterey, Mexico.  
 Sanford, P. Gerald, Blandford Lodge, Streatham, S.W.; and  
 (Journals) 20, Culham Street, Fenchurch Street, E.C.  
 Saniter, E. H., c/o The Wigan Coal and Iron Co., Wigan.  
 Sankey, Chas. H., Hillshore Lodge, Dulwich Grove, North  
 Dulwich, S.E.  
 Savage, W. W., 109, St. James's Street, Brighton.  
 Savary, W. J. H., 39, Lombard Street, E.C.  
 Sayers, Jos. J., Nobel's Explosives Co., Ardeer, Stevenston,  
 Ayrshire.  
 Schad, Julius, 15, Cooper Street, Manchester.  
 Schad, Dr. Philip, 4, Dyar Terrace, Widdington, North-  
 wich.  
 Schüppi, Dr. H., Mithöli, Canton Glarus, Switzerland.  
 Schellhaas, H., 38, Navigation Road, Northwich.  
 Scheurer-Kestner, Dr. A., 8, Rue Pierre-Charron, Paris,  
 France.  
 Schleicher, Francis J., 250, Van Alst Avenue, Long Island  
 City, N.Y., U.S.A.  
 Schlesinger, Dr. Jas., P.O. Box 368, Pretoria, S.A.R.  
 Schlichter, H. C., c/o Stöhr & Co., Kammgarn Spinnerei  
 Leipzig, Germany.  
 Schiller, Louis J., The American Sugar Refining Co., Granite  
 Street, South Boston, Mass., U.S.A.  
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 Schofield, H. E., Greenwood, Victoria Park, Wavertree,  
 Liverpool.  
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 Schulze, Dr. Karl E., Chemische Fabrik Ländenhof, Waldhof  
 bei Mannheim, Germany.  
 Schunck, Dr. E., F.R.S., Kersal, near Manchester.  
 Schupphaus, Dr. Robt. C., 118, Pennsylvania Avenue,  
 Brooklyn, N.Y., U.S.A.  
 Schwab, Dr. L. C., Sedanstrasse 53, Bernburg, Anhalt.  
 Schweich, Emil, 47, Victoria Street, Westminster, S.W.  
 Schweitzer, Dr. H., 159, Front Street, New York City,  
 U.S.A.  
 Seagrie, Prof. J., Poona Villa, King's Gate, Aberdeen.  
 Scott, Andrew, 2, Teviot Terrace, Kelvinside, Glasgow.  
 Scott, Andrew R., Cruntyte Chemical Works, Parkhead,  
 Glasgow.  
 Scott, Chas. K., Caixa 32, Pernambuco, Brazil.  
 Scott, Ernest G., 67, Lord Street, Liverpool.  
 Scott, F., Messrs. Fred. Scott & Co., 'Littleboro', near  
 Manchester.  
 Scott, F. Walter, 44, Christian Street, London, E.  
 Scott, G. H., Bally, Fairfield, near Manchester.  
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 Scott, Wm., 2, Creek Terrace, Albert Road, Silvertown, E.  
 Seovell, M. A., Lexington, Kentucky, U.S.A.  
 Seutton, Willis, Robinson's Ferry, Calaveras Co., Cal.,  
 U.S.A.  
 Strymeour, A., (Journals) Box 1283, Johannesburg,  
 S.A.R.; and (subs.) Shore Road, Stevenston, N.B.  
 Sandler, P., 29, Elmfield Road, Balham, S.W.  
 Searl, Albert, Phoenix Mills, Dartford, Kent.  
 Segner, P., 26, Princess Street, Manchester.  
 Seinet, Louis, 217, Chaussée de Vleurgat, Brussels.  
 Seutter, Dr. Erhard von, Via Trere 4, Gorz, Austria.  
 Sevin, C., c/o Dollman & Pritchard, 3, Laurence Pountney  
 Hill, E.C.

- Small, Wm., jun., c/o Londonderry Iron Co., Londonderry, Nova Scotia.
- Small, Evan W., Sunnyside, West Park, Newport, Mon.
- Smetham, A., 18, Brunswick Street, Liverpool.
- Smiles, Jas., Blandfield Chemical Works, Canonmills, Edinburgh.
- Smith, Alfred, Excelsior Chemical Works, Clayton, Manchester.
- Smith, Anthony, Hughenden, Castle Avenue, Clontarf, Dublin.
- Smith, A. J., 220, Rock Street, Pittsboro, Sheffield.
- Smith, Edgar B., c/o Messrs. Bart, Boulton, and Haywood, Selzacte, Belgium.
- Smith, Prof. Edgar F., University of Pennsylvania, Philadelphia, Pa., U.S.A.
- Smith, Edgar F., 35, Amptill Square, Hampstead Road, London, N.W.
- Smith, Ernest A., Metallurgical Laboratory, Royal College of Science, South Kensington, S.W.
- Smith, Francis P., 1, S. Navy Yard, New York, U.S.A.
- Smith, Fred., Box 1324, Johannesburg, S.A.R.
- Smith, G., Polmont Station, Scotland.
- Smith, Geo., 14, The Pass, Ramsay Street, Rochdale.
- Smith, Geo. F., Cromwell Lodge, Purney Hill, S.W.
- Smith, Harry, 33, Withington Road, Whalley Range, Manchester.
- Smith, Harry E., 133, 26th Street, Milwaukee, Wis., U.S.A.
- Smith, H. R., 1, Aubert Park, Highbury, London, N.
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- Smith, R. W., The Grange, Kirkburton, near Huddersfield.
- Smith, R. Watson, Young's Oil Co., Ltd., Chemical Works, Bathgate, N.B.
- Smith, S., 35, Amptill Square, Hampstead Road, London, N.W.
- Smith, Watson, University College, London, W.C.; and 34, Upper Park Road, Haverstock Hill, N.W.
- Smith, Wilfred, 182, West Street, Glasgow.
- Smith, W., 10, Corn Street, Bristol.
- Smithells, Prof. A., Yorkshire College, Leeds.
- Smithers, F. O., Dashwood House, 9, New Broad Street, London, E.C.
- Smithson, J., Park Printworks, Halifax.
- Smithson, Saml., Ravensthorpe, near Dewsbury, Yorks.
- Snap, Dr. H. Lloyd, University College, Aberystwith.
- Soames, J. K., Thames Soap and Candle Works, Greenwich, S.E.
- Sohn, Chas. E., 25, Beverley Road, Anerley, S.E.
- Solvay, Alfred, 25, Rue de Prince Albert, Brussels.
- Solvay, Ernest, 43, Rue des Champs Elysées, Brussels.
- Sommer, Adolf, corner 1st and Binney Streets, East Cambridge, Boston, Mass., U.S.A.
- Sommer, Dr. G. Schaack, 323, Vauxhall Road, Liverpool.
- Sonstadt, Edw., Church Fields, Chesshurst, Herts.
- Southern, Thos., Jr., Jr., Wheatthill Chemical Works, St. Simon Street, Salford.
- Soward, A. W., 141 Friern Road, East Dulwich, S.E.
- Sowerby, Thos. H., Sherwell, Dartmouth Place, Blackheath, S.E.
- Sowerby, W. M., Stanley Villa, Higher Runcorn, Cheshire.
- Sowler, Alf., Fairview, The Knoll, Beckenham, Kent.
- Spackman, Chas., 12, Beacon Road, Loughborough.
- Speakman, Jas., Penhold P. O., Calgary, Alberta, Canada.
- Speakman, J. J., Stanley Villas, Greenway Road, Runcorn.
- Spence, D., Alum Works, Manchester.
- Spence, F., Alum Works, Manchester.
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- Spiegel, Dr. Adolf, Messel, bei Darmstadt, Germany.
- Spies, Adolph, 102, Fenchurch Street, London, E.C.
- Spies, Hermann, 102, Fenchurch Street, London, E.C.
- Spiller, A., The Buckeye Electric Co., 1925, Broadway, Cleveland, Ohio, U.S.A.
- Spiller, J., 2, St. Mary's Road, Canonbury, London, N.
- Spoor, John L., Ga's Hill, Higham, Kent.
- Sprengel, Prof. H., Ph.D., F.R.S., Savile Club, 107, Piccadilly, London, W.
- Squire, E. L., Coalbrookdale, Shropshire.
- Squire, Percy, 11, Wine Office Court, Fleet Street, London, E.C.
- Squire, P. W., 413, Oxford Street, London, W.
- Squire, Dr. W. S., Clarendon House, St. John's Wood Park, N.W.
- Stacey, H. G., 300, High Holborn, London, W.C.
- Stahl, Dr. K. F., 57th Street and A. V. R. R., Pittsburgh, Pa., U.S.A.
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- Stead, W. H., 23, Boundary Street, Liverpool; and (Journals) Orchard Place, Blackwall, E.
- Stearns, Fred. K., Detroit, Mich., U.S.A.
- Stebbins, J. H., 114, Pearl Street, New York, U.S.A.
- Steedman, R. M., Sprengfield House, Dalmeir, N.B.
- Steel, R. Elliott, Science, Art, and Technical Schools, Plymouth.
- Steel, Thos., Colonial Sugar Refinery, Sydney, N.S.W.
- Stein, Sigmund, 323, Vauxhall Road, Liverpool.
- Steinhart, Dr. Oscar J., c/o May and Baker, Ltd., Garden Wharf, Battersea, S.W.
- Stenhouse, T., 8, West Street, Rochdale.
- Stephens, H. O'Reilly, 20, Brunswick Gardens, Kensington, W.
- Stephens, M. E., Avenue House, Finchley, N.
- Stephens, H. Chas., M.P., Avenue House, Finchley, N.
- Stephenson, Chas., 25, Cecil Street, Greenheys, Manchester.
- Stear, Arthur L., 170, Ashby Road, Barton-on-Trent.
- Stewart, D. R., Broxburn, near Edinburgh, N.B.
- Stevens, Jno. H., The Celluloid Co., Newark, N.J., U.S.A.
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- Stevens, W. J., 24, New Walls Road, Totterdown, Bristol.
- Stevenson, Jas., 23, West Nile Street, Glasgow; and The Bromfields, Largs, N.B.
- Stevenson, J. C., M.P., 33, Devonshire Place, W.
- Stevenson, Dr. T., Guy's Hospital, London, S.E.
- Stevenson, W., Standard Works, 95A, Southwark Street, London, S.E.
- Stewart, Alex. F., Box 1283, Johannesburg, S.A.R.
- Stewart, Ebenezer, Oil Seed Mills, Rochester, Kent.
- Stewart, Jeffrey, 15, Claremont Road, Forest Gate, Essex.
- Stewart, Robt., c/o Bonke, Roberts, and Co., Warton Road, Stratford, E.
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- Stoddart, F. Wallis, Western Counties Laboratory, Bristol.
- Stoer, J., 6, Hanover Quay, Dublin.
- Stoker, G. N., Laboratory, Somerset House, London, W.C.
- Stone, F. H., Eardley Villa, Picardy Hill, Belvedere, Kent.
- Stone, Thos. W., Chemical Works, St. George, Bristol.
- Stone, W., 345A, Swanston Street, Melbourne, Victoria.
- Storer, Dr. John, 29, Bligh Street (G.P.O. Box 220), Sydney, New South Wales.
- Storey, J. H., Haverbreaks, Lancaster.
- Stott, Jno. W., 2, Prince of Wales Road, Swansea.
- Stowe, W. T., Laboratory, Somerset House, Strand, W.C.
- Strangman, J. Pim, 28, Rue Desbordes-Valmore, Passy, Paris.
- Sträuli, Emil, Winterthur, Switzerland.
- Strong, Colin R., 18, Exchange Street, Manchester.
- Stuart, C. E., 29, Mosley Street, Newcastle-on-Tyne.
- Stuart, E. H., 3,900, Cottage Grove Avenue, Chicago, Ill., U.S.A.
- Stuart, Jas., 22, High Street, Hull.
- Stuart, T. W., 15, Windsor Terrace, Newcastle-on-Tyne.
- Studer, Dr. A., 10, Marsden Street, Manchester.
- Studer, Simon J., 17, Lovely Lane, Warrington.
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- Sutherland, D. A., 2, Victoria Mansions, Westminster, S.W.
- Sutherland, Jas., Ballyclare, co. Antrim, Ireland.
- Sutherland, Jno., Elsternwick, near Melbourne, Victoria.
- Sutherland, R. M., Lime Wharf Chemical Works, Falkirk, N.B.
- Sutton, F., London Street, Norwich.
- Sutton, F. Napier, 6, Grosvenor Gardens, Willesden Green, N.W.
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- Swinburne, Geo., c/o J. Coates & Co., Planet Chambers, 8, Collins Street East, Melbourne, Australia; (subs.) Suffolk House, Laurence Pountney Hill, E.C.
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- Sykes, E., 111, Victoria Street, Newton, Hyde, Cheshire.
- Sykes, James, 76, Lockwood Road, Huddersfield.
- Syme, W. B., c/o Young's Paraffin Oil Co., Addicwell, West Calder, N.B.
- T
- Takamatsu, T., Tokyo University, Japan.
- Takamine, J., 2111, North Jefferson Street, Peoria, Ill., U.S.A.
- Takayama, Jintaro, Geological Survey Office, Department of Agriculture, Tokio, Japan.
- Tasker, G., 1048, Cathcart Road, Mount Florida, Glasgow.
- Tate, E., 1, Collingham Gardens, South Kensington, S.W.
- Tate, F. H., 9, Hackins Hey, Liverpool.
- Tate, H., jun., Allerton Beeches, Allerton, near Liverpool.
- Tatlock, J., 40, Renfrew Street, Glasgow.
- Tatlock, R. R., 156, Bath Street, Glasgow.
- Tatton, Reginald, A., 44, Mosley Street, Manchester.
- Taubman, R., 124, Southampton Row, London, W.C.
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- Taylor, J. Scott, c/o Winsor and Newton, Limited, 38, Rathbone Place, London, W.
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- Taylor, Thos., Walmersley Bleach and Dyeworks, near Bury, Lancashire.
- Taylor, W. Ambrose, Royal Geological Society of Cornwall, Penzance.
- Taylor, W. G. H., 3, Vernon Road, North Baw, E.
- Taylor, W. J., 55, Forsyth Street, Greenock, N.B.
- Tchaykovsky, N. W., Langham House, Harrow-on-the-Hill.
- Teauby, G. W. A., Laboratory, 65, Bath Row, Birmingham.
- Teed, Dr. F. L., 15, Victoria Street, Westminster, London, S.W.
- Tennant, Sir Chas., Bart., 35, Grosvenor Square, S.W.; and Glen, Peebleshire, N.B. (Journals to St. Rollox, Glasgow.)
- Tennant, Jas., Dartmouth Lodge, Saltwell, Gateshead-on-Tyne.
- Terry, Albert, Verulam, Mount Albert Road, Balwyn, near Melbourne, Victoria.
- Terry, Hubert L., 14, Herbert Street, Moss Side, Manchester.
- Tervet, R., 54, Peshurst Road, South Hackney, E.
- Tetley, C. E., Messrs. Jos. Tetley and Son, The Brewery, Leeds.
- Thew, Walter H., 47, Castle Street, Liverpool.
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- Thomas, Philip A., Cornwall Buildings, 35, Queen Victoria Street, London, E.C.
- Thomas, S. Percy, 15, Ham Park Road, Stratford, E.
- Thomasson, Wm., 167, North Road, St. Helens, Lancashire.
- Thompson, Prof. Claude M., University College, Cardiff.
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- Thompson, W., jun., Larkfield, Earlestown, Lancashire.
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- Thomson, G. Carruthers, 23, Kersland Terrace, Hillhead, Glasgow.
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- Thomson, John, 70A, Grosvenor Street, London, W.
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- Thomson, Dr. Murray, 44, Victoria Road, Gipsy Hill, S.E.
- Thomson, Robt. T., 156, Bath Street, Glasgow.
- Thomson, W., Royal Institution, Manchester.
- Thomson, Wm. Garth, 41, Mitchell Street, Glasgow.
- Thomson, Wm. Thos., Royal Gunpowder Factory, Waltham Abbey, Essex.
- Thorne, Dr. L. T. S., Dynevor Road, Richmond-on-Thames.
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- Thorp, Thos., Moss Bank, Whitefield, Manchester.
- Thorp, W., 24, Crouch Hall Road, Crouch End, N.
- Thorpe, Prof. T. E., F.R.S., Royal College of Science, South Kensington, S.W.
- Tichborne, Dr. Chas. R. C., 15, North Great George Street, Dublin.
- Tilden, Prof. W. A., F.R.S., 77, Harborne Road, Birmingham.
- Timmius, A., Argyll Lodge, Higher Runcorn.
- Timmis, T. Sutton, Widnes.
- Tobey, C. M., Collingwood, Ontario, Canada.
- Tocher, Jas. F., 1, Chapel Street, Peterhead, N.B.

- Todd, A. M., 204, North Rose Street, Kalamazoo, Mich. U.S.A.
- Tompkins, H. K., 52, Whiteley Road, Gipsy Hill, S.W.
- Toms, F. Woodland, States Analyst's Office, St. Heliers, Jersey.
- Tonks, E., Packwood, Knowle, near Birmingham.
- Torsell, C. T., McKees Rocks, Allegheny Co., Pa., U.S.A.
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- Towers, J. W., Grosvenor Buildings, Victoria Road, Widnes.
- Townsend, Chas. F., 31, Elms Road, Clapham Common, S.W.
- Townsend, Chas. W., 19, Crawford Street, Port Dundas, Glasgow.
- Townsend, Oliver C., Port Dundas, Glasgow.
- Travers, Morris W., 2, Phillimore Gardens, Kensington, W.
- Trechmann, A. O., Tinnoth House, Halling, near Rochester.
- Trechmann, C. O., 10, Cliff Terrace, Hartlepool.
- Trench, G., Standard House, Faversham, Kent; and the Cotton Powder Co., Limited, Faversham.
- Trewby, Herbert, Radnor House, Ribblesdale Road, Horsey, N.
- Tribe, P. C. M., Oswego, New York, U.S.A.
- Trimble, Prof. H., 115, North 10th Street, Philadelphia, U.S.A.
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- Trubshawe, Wolstan, 6, St. Benet Place, London, E.C.
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- Tsukiyama, S., Printing Department, Ministry of Finance, Tokyo, Japan.
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- Tuer, Arthur H., Thornhill, near Wigan.
- Tulloch, John, 12, Victoria Terrace, Jarrow-on-Tyne.
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- Turnbull, G. W., 10, Northgate, Darlington.
- Turnbull, Wm., 14, Wilberforce Terrace, Gateshead-on-Tyne.
- Turnbull, W. S., Place of Bonhill, Renton, Dumbartonshire.
- Turnbull, Samuel G., 10, Rue de Bas Trevois, Troyes (Aube), France.
- Turner, H. B. H., Trinity College, Cambridge.
- Turner, P. R., Tar Works, Rothwell Haigh, near Leeds.
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- Turner, W. Spencer, 227A, Oxford Street, Manchester.
- Turney, Sir J., Springfield, Alexandra Park, Nottingham.
- Turri, G. G., San Buildings, Queen Street, Melbourne, Victoria.
- Tweedie, G. R., 54, Hawley Square, Margate, Kent.
- Tweedy, Jas., 77, Western Road, Jarrow-on-Tyne.
- Twitchell, E., 10, Bellevue Avenue, Mount Auburn, Cincinnati, Ohio, U.S.A.
- Twynam, T., Elmhurst, Egham Hill, Surrey.
- Typeke, P. G. W., Ravenhurst, Norbiton Park, New Malden, Surrey.
- Tyrer, Chas. T., Stirling Chemical Works, Stratford, E.
- Tyrer, T., Stirling Chemical Works, Stratford, E.
- Tyzack, Stuart, Beekford Lodge, Williamson Road, Sheffield.
- U
- Umney, C., 50, Southwark Street, London, S.E.
- Underhill, Thos. J., H.M. Victualling Yard, Deptford, S.E.
- Underwood, G. R., Box 460, Peabody, Mass., U.S.A.
- Usmar, J. H., 34, Palmerston Buildings, London, E.C.
- Utsumi, S., Hokkaido Cement Works, Hakodate, Japan.
- V
- Vandenberg, Dr. Frank P., 32-33, Lewis Block, Buffalo, N.Y., U.S.A.
- Vandenberg, Dr. Horace C., University of the City of New York, 410, East 26th Street, New York, U.S.A.
- Van Gundy, Chas. P., Laboratory, B. & O. R. R., Baltimore, Md., U.S.A.
- Vargas-Vergara, J. M., Apartado No. 237, Bogota, Republic of Colombia, S. America.
- Vary, G. M. P., 1319, O'Farrell Street, San Francisco, Cal., U.S.A.
- Vasey, T. E., 6, South Parade, Leeds; Journals to P.O. Box 1777, Montreal, Canada.
- Vaughan, J. L., 329, Norwood Road, Tulse Hill, S.E.
- Vautin, Claude T. J., 42, Old Broad Street, London, E.C.
- Veitch-Wilson, J., 8, Earlsfield Road, Wandsworth Common, S.W.
- Venables, T., 23, Smith Street, Hillhead, Glasgow.
- Verel, W. A., 136, West George Street, Glasgow.
- Vickers, W., Rose Hill, Smedley Lane, Manchester.
- Voeleker, E. W., 22, Tudor Street, London, E.C.
- Voeleker, Dr. J. A., 20, Upper Phillimore Gardens, Kensington, W.
- Vorster, Fritz, 71, Bayenstrasse, Cöln a/Rhein, Germany.
- Voss, Hermann, 19, Beckenham Road, Beckenham, Kent.
- Vulté, Hermann T., School of Mines, Columbia College, New York, U.S.A.
- W
- Waché, Alf., 27, Rue Morel, Douai, France.
- Wade, Jas. L., 28, West Kensington Gardens, London, W.
- Wade, W. L., 28, West Kensington Gardens, W.
- Wadman, W. E., 102, Lord Avenue, Bayonne, N.J., U.S.A.
- Wagner, W. G., Glyndhurst, Ealing Common, W.
- Wainwright, Dr. J. H., 22, West 46th Street, New York, U.S.A.
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- Walker, A., Messrs. Alex. Walker and Co., Alkali Works, Irvine, N.B.
- Walker, Archibald, 8, Crown Terrace, Glasgow.
- Walker, E. Robinson, 18, St. Ann's Street, Manchester.
- Walker, Jno. C., 156, West Regent Street, Glasgow.
- Walker, S. R., 19, Wolsey Street, Radcliffe, Manchester.
- Walker, T., Eccleston Park, Prescott, Lancashire.
- Wallace, Robert, 1, Coates Place, Edinburgh.
- Waller, Dr. E., School of Mines, Columbia College, 50th Street, 4th Avenue, New York, U.S.A.
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- Walton, F., 114, Holborn, E.C.
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 Wyndham, Dr. Stanley, c/o Mrs. Jamieson, 4, Marloes Road, Kensington, W.

## Y

Yates, F., 64, Park Street, Southwark, London, S.E.  
 Yates, R., 64, Park Street, Southwark, London, S.E.  
 Yoshida, Hiokorokuro, Imperial University, Hongo, Tokyo, Japan.  
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 Young, Brongham, Home Lyn, Woodberry Down, London, N.  
 Young, Dr. Geo., Firth College, Sheffield.  
 Young, Jno., 2, Montague Terrace, Kelvinside, Glasgow.  
 Young, Jno., Gasworks, Etruria, near Stoke-on-Trent.  
 Young, Jno., The Linlithgow Oil Co., Ltd., 123, Hope Street, Glasgow.  
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 Young, W. C., 22, Windsor Road, Forest Gate, E.  
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## Z

Zimmermann, A., 67, Cross Lane, St. Mary-at-Hill, London, E.C.  
 Zinkeisen, Dr. W., 508, New City Road, Glasgow.

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## NOTICES.

Notice is hereby given in accordance with Rule 51 of the By-Laws that the Council will propose to the forthcoming Annual Meeting the amendment of the Society's By-Laws as follows:—

1. That *Rule 7* be amended by the deletion of "and" in the first line thereof, and by the addition of the words "and 47" in the same line.

2. That under *Rule 18* the form of nomination of candidates for the Council be amended by transferring the candidate's declaration to the end thereof, and that the words "and by the nominee" be added in the third line after the above-mentioned form after the word "subscriptions."

3. That the proviso at the end of *Rule 22* be deleted.



4. That in *Rule 26* the Nomination Form for candidates for Membership be amended to read as follows:—

"We, the undersigned members of the Society of Chemical Industry, hereby propose  
(*candidate's name in full*) as a fit and proper person to become a member of the Society.

(*For names only of members  
recommending the candidate.*)

"And I, the said candidate for membership, hereby declare that if elected I will do my best to support the Society and will observe its By-Laws.

(Signature) \_\_\_\_\_ (Profession) \_\_\_\_\_

(Address) \_\_\_\_\_ (Date) \_\_\_\_\_

*Extract from By-Laws (Rules 27, 29, and 30).*

5. That *Rule 27* should read as follows:—

"Each member shall pay an annual subscription of twenty-five shillings, due on the 1st January in each year; except that any member may pay a life composition fee of twenty pounds in lieu of an annual subscription. He shall also pay on receipt of notice of election an entrance fee of one guinea in addition to his first year's subscription; except that should a member who has paid an entrance fee resign and subsequently seek re-election the Council may dispense with a further entrance fee."

6. That in the second line of *Rule 28* the words after "and" read as follows:—"issued to the members once every year."

7. That *Rule 29* read as follows:—

"No person shall be entitled to the privileges of membership who has not paid his subscription for the current year. He shall not attend meetings nor vote, nor shall he receive gratuitously the publications of the Society. Should the subscription remain unpaid eight months after it is due, the Secretary shall give the member notice that unless payment be made within four months after the date of such notice, his name will be struck off the register. At the expiry of the said period, if the member should still remain in default, the Council may order his name to be removed from the register, whereupon he shall cease to have any right, privilege, or interest in the Society, and the Council may notify the same in the Journal and recover the arrears of subscription at law."

8. That *Rule 30* read as follows:—

"Any member may withdraw from the Society at the end of any year by giving previous notice to the General Secretary of his intention to withdraw; always provided that such notice to be valid must be accompanied by the payment of all subscriptions, if any, which may be due from him to the Society at the date of the notice. By such notice and payment he shall be released from all further liability as a member."

9. That the following Rule be added after *Rule 53* of the present Rules:—

"All communications between Local Sections and Public Bodies or Societies shall be conducted through or be at the discretion of the Council; and no publication shall be issued by any Section to the general public without the previous consent of the Council."

10. That *Rules 54* and *55* be placed after *Rules 34* and *38* respectively, and that the succeeding Rules be renumbered accordingly."

#### BANKERS' ORDERS.

For the convenience of Members, the Treasurer has arranged with the Bankers of the Society that they shall collect subscriptions from Bankers in town and country; and Members, who have not already done so, are invited to fill up and sign the Banker's Order enclosed with this number of the Journal, which should then be sent to the Honorary Treasurer, Mr. E. Rider Cook, East London Soap Works, Bow, E.

Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under *Rule 41* of the bye-laws, the Society has the right of priority of publication for three months of all such papers. Infringement of this bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. EYRE and SPOTTISWOODE, the Society's printers and publishers, to whom all communications respecting them should be addressed.

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Barracloagh, T. C., 20 Bucklersbury, London, E.C., engineer.

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Carswell, Thos. R., Day House, Day Street, Newcastle-on-Tyne, analyst and teacher.

Charlier, Andrew, 107, Breadalbane Terrace, Hill Street, Glasgow, manager and chemist.

Chatard, Dr. T. M., 1410 G. Street, Washington, D.C., U.S.A., chemical engineer.

Cheney, J. P., c/o Cheney Bros., South Manchester, Conn., U.S.A., silk chemist.

Clayton, Robt. H., 762, Rochdale Road, Manchester, chemist.

Crossley, Dr. Arthur W., The Owens College, Manchester, hon. demonstrator in organic chemistry.

Ede, Henry E., c/o Rev. Moore Ede, The Rectory, Gateshead-on-Tyne, analytical chemist.

Fuller, Chas. J. P., Laboratory, L. & Y. Ry. Works, Horwich, near Bolton, analytical chemist.

Gurney, J. Clare, Fabrica Roma, La Union, Prov. de Murcia, Spain, analytical chemist.

Heal, Carlton, Leather Department, Yorkshire College, Leeds, student.

Hett, Stephen J., 25, Percy Street, Liverpool, analytical chemist.

Lockwood, Jos., 263, Argyle Street, Glasgow, artist and patent agent.

McCann, Owen, 158, Tredegar Road, Bow, E., printing ink and varnish maker.

Martin, Alex. M., Douglas Villa, Dunbeth Road, Coatbridge, N.B., analytical chemist.

Martyn, T. Graham, Trevemper Bridge, Newquay, Cornwall, mining engineer and metallurgist.

Miller, J. Carlie, 89, Rumbold Street, Bridgeton, Glasgow, tar distiller.

Pattinson, Dr. H. Salvin, 75, Side, Newcastle-on-Tyne, analytical chemist.

Peden, Jno., jun., Blair Terrace, Greenock, N.B., analytical chemist.

Rodda, Edw. D., 6, Gold Street, Roath, Cardiff, engineer.

Schad, Dr. Philip, 4, Dyar Terrace, Winnington, Northwich, analytical chemist.

Schryver, Dr. S. B., University College, Liverpool, assistant lecturer on chemistry.

Schweitzer, Dr. H., 159, Front Street, New York City, U.S.A., analytical chemist.

Skinner, C. Weeding, Wansfell, Theydon Bois, Essex, manufacturing chemist.

Sonstadt, Edw., Church Fields, Cheshunt, Herts, chemical technologist.

Stevens, Jno. H., The Celluloid Co., Newark, N.J., U.S.A., manufacturing chemist.

Thomas, H. Russell, Broad Plain Soap Works, Bristol, soap manufacturer.

Wade, W. Luvian, 28, West Kensington Gardens, London, W., chemist.

Wareing, Arthur E., Bedford House, Appleton, Widnes, analytical chemist.

White, B. Newport, Manor Hill, Marple, Cheshire, consulting brewer.

Wimpenny, John, c/o Dublin and Wicklow Manure Co., Lim., Wicklow, Ireland, mauager.

### CHANGES OF ADDRESS.

Barrie, D. McL., Journals to c/o Langhaagte G. M. Co., P.O. Box 98, Johannesburg, S.A.R.

Billing, H. S., Journals to 5, Regent Terrace, Plympton, South Devon.

Bowing, John, Journals to Fuel Works, Tilbury, Essex.

Breen, G., Journals to c/o United Alkali Co., Lim., 204, St. Vincent Street, Glasgow.

Brown, J. Henry, 1/o North Wales; c/o Bowman, Thompson, and Co., Lim., Northwich.

Burton, Wm., 1/o The Potteries; Clifton Junction, near Manchester.

Campbell, Arch., 1/o Thornton; 42, Whittaker Road, Upton Park, E.

Coleman, J. B., 1/o University College; Burley House, Carlton, Nottingham.

Collins, C. S. A., Journals to c/o Mrs. Stevens, 56, Elmu Row, Leith Walk, Edinburgh.

Corbett, J., M.P., 1/o Stoke Prior; Impney, Droitwich.

Davis, Herbert J., 1/o Wall Street; 255, West 71st Street, New York, U.S.A.

Dixon, M. T., 1/o Johannesburg; 5, Brandling Park, Newcastle-on-Tyne.

Duffield, Dr. S. P., 1/o Detroit; Dearborn, Wayne Co., Mich., U.S.A.

Duncan, Jas., 1/o Mentone; Maison Ganich, Biarritz, France.

Edgell, G. E., 1/o 5; 3, Queen's Terrace; Gateshead-on-Tyne.

Ekman, C. D., 1/o Italy; Paper Mills, Northfleet, Kent.

Fenwick, Jas., 1/o Huelva; 23, Ronald Street, Glasgow.

Fiebing, J. H., Journals to 644, 28th Street, Milwaukee, Wis., U.S.A.

Fleming, J. Arnold, 1/o Glasgow; Woodland Pottery, Tunstall, Staffs.

Garrett, F. C., 1/o Newcastle; The Polytechnic, Woolwich, S.E.

Girdwood, Dr. G. P., 1/o Beaver Hall Terrace; 82, University Street, Montreal, Canada.

Hawliczek, J., 1/o Queensgate; Claremont Park, Blackpool.

Hulme, Jas., 1/o Hollingworth; Newton Chemical Works, Hyde, Manchester.

Hunt, Bertram, 1/o Glasgow; P. O. Box 1606, Denver, Col., U.S.A.

Hunt, W., Journals to Hampton House, Wednesbury

Hunzinger, A., Journals to Dinting Vale Printworks, Dinting, Derbyshire.

Inglis, R. A., 1/o Arden; Culrain, Bothwell, N.B.

Isaac, T. W. Player, 1/o Marine Parade; 11, Cavendish Place, Brighton.

Johnson, J. Henry, 1/o Lincoln's Inn; Mountains, Tonbridge, Kent.

Kay, Dr. P.; Journals to 196, Heerenstraat, Leiden, Holland.

Kibble, W. Oakes, 1/o Buckhurst Hill; Tavern Street, Stowmarket, Suffolk.

Laurie, A. P., 1/o Cambridge; 49, Beaumont Square, London, E.

Liepmann, Dr. H., 1/o Glasgow; c/o Cracker Creek G. M. Co., Ltd., Eureka, Baker Co., Oregon, U.S.A.

Lunn, C., 1/o Rotterdam; 3, Osborne Road, Antfield, Liverpool.

McBeath, J. W., 1/o West Hartlepool; (Journals) Kimberley, South Africa; and (subscription) c/o H. C. McBeath, Greatham Terrace, West Hartlepool.

McFarlane, J. A.; delete Glasgow address for subscriptions.

McKellar, W. G., Journals to c/o United Alkali Co., Lim., Eglinton Works, Irvine, N.B.

Mansbridge, Wm., Journals to c/o Goodwin Manufacturing Co., St. Louis, Mo., U.S.A.

Marshall, Prof. T. Rymer, 1/o Edinburgh; St. Mungo's College, Glasgow.

Mercer, J. B., 1/o 322; 330, Lower Broughton Road, Manchester.

Miller, Dr. H. von, 1/o Hruschau; Beatrixgasse 32 III., Vienna, Austria.

Miller, Dr. Harry E., 1/o San Francisco; 1264, 14th Street, Oakland, Cal., U.S.A.

Morris, Dr. G. Harris, 1/o Burton; 72, Chancery Lane, London, W.C.

Moss, John, Journals to Wilson Street, New Cross Road, S.E.

Oddie, Jas.; delete London address for subscriptions.

Pettigrew, John, 1/o 18; 6, St. Helen's Place, Bishops-gate, E.C.

Powell, A. Ernest, 1/o Cross Street; 59, Egerton Road, Fallowfield, Manchester.

Prinz, Dr. Otto, 1/o Bavaria; Sesto Fiorentino, Italy.

Ramsden, Edw., 1/o Great Horton; 11, Cecil Avenue, Horton Park, Bradford.

Ray, Wm., 1/o Sale; 33, Sloane Street, Brooks Bar, Manchester.

Raymond, C. W., 1/o Poplar; 65, Clova Road, Forest Gate, E.

Rintoul, Wm., 1/o Poplar, 43, King William Street, Greenwich, S.E.

Russell, D., 1/o Leven; Cadham, Markinch, Fife, N.B.

Sach, Robt.; Journals to c/o Señor César García, Honda, Republic of Colombia, South America.

Schweich, E., 1/o Regent's Park; 47, Victoria Street, Westminster, S.W.

Small, Evan W.; Journals to Sunnyside, West Park, Newport, Mon.

Sykes, E., 1/o Bolton; 111, Victoria Street, Newton, Hyde, Cheshire.

Tetley, C. F., 1/o Headingley; Messrs. Jos. Tetly and Son, The Brewery, Leeds.

Turner, W. Spence, 1/o 225; 227A, Oxford Street, Manchester.

Twynam, T., 1/o Chiswick; Elmhurst, Egham Hill, Surrey.

Voss, H., 1/o Penge Road; 19, Beckenham Road, Beckenham, Kent.

Wadman, W. E.: Journals to 102, Lord Avenue, Bayonne, N.J., U.S.A.

Waterhouse, R., 1, o London; Earncliffe, Beech Hill Road, Sheffield.

White, J. Campbell; all communications to "Overtoun, Lord," 7, West George Street, Glasgow.

### CHANGE OF ADDRESS REQUIRED.

Jarman, G. S., 1, o Croft House, Marsh, Huddersfield.

### Deaths.

Brown, Jas. T., 7, Beaufort Street, Chelsea, S.W.

Button, H., Rainham, Essex.

Eastlake, T. Lloyd, 23, Great George Street, Westminster, S.W.

Miller, Robt., 79, West Nile Street, Glasgow.

Neil, W., 126, Turner's Road, Burdett Road, E.

Sharpley, H., Limber Magna, Ulceby, Lincolnshire.

Upward, W., Albert Road, Widnes.

### ERRATUM.

In the December changes of address, the name of J. Carter Bell was given in error for that of Percy Carter Bell.

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Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

### SESSION 1899.

Feb. 7th:—

Professor E. Divers, F.R.S. "The Manufacture of Calomel in Japan."

Mr. J. Beveridge. "The Manufacture of Straw Pulp."

March 7th:—

Mr. F. H. Tate. "Methods for the Rectification of Vitriol."

Mr. Max Muspratt. "The Quantitative Estimation of Cadmium."

April 4th:—

Mr. J. Henry Brown. "The Treatment of Cupreous Iron Pyrites as carried out at Portuguese Mines."

Meeting held Wednesday, December 6th, 1893.

MR. EUSTACE CAREY IN THE CHAIR.

## THE MANUFACTURE AND INDUSTRIAL VALUE OF ALUMINIUM ALLOYS.

BY J. H. J. DAGGER, F.I.C., F.C.S.

IN the two papers I have had the honour of reading before this Society, and in a paper read before the British Association in 1889, I gave some account of the present day methods for the production of this industrially new metal, and, expressing my views as to its value, based upon an especial experience of the metal in the laboratory, foundry, and workshop, I then stated that "the greatest value of aluminium is as yet in its alloys"; that these words hold true, and that the alloys of aluminium have a value that can hardly be over-estimated, I will endeavour to set forth this evening. Aluminium alloys readily with all the common metals, with one or two exceptions, and with gold, silver, and platinum. These alloys may be conveniently considered as divided into two classes, simple and complex: aluminium alloyed with one metal, or with two or more.

*The Simple Alloys.*—The most important are the copper alloys; these form two series:—Aluminium bronzes with 1 to 11 per cent. of aluminium, and aluminium copper alloys with 90—99 per cent. of aluminium. The intermediate alloys form a series differing widely in colour and other physical characteristics of great scientific interest, but, owing to their crystalline brittle nature, valueless in the arts. The 10 per cent. aluminium bronze is perhaps the best known; it was first prepared by Dr. Percy, of the Royal School of Mines, in 1855, and as early as 1859 a series of experiments were made with it, a mountain gun being cast of it under the direction of the Committee of Artillery of France, and in 1860 further experiments in this direction were made by the Bavarian military authorities, both the French and Bavarian experts reporting that the cost of aluminium bronze was the only thing then prohibiting its use for guns. In his report on the Augsburg artillery trials, Colonel Weber states that the aluminium bronze resisted a pressure of 97,012 lb. per square inch, whilst the tin bronze then used gave only a pressure resistance of 39,601 lb. At that time, pure aluminium was 38s., aluminium 10 per cent. bronze 4s. 11d., and tin bronze 1s. 3d. per lb.

Aluminium bronze is prepared by two methods. 1. By direct reduction of aluminium ore, such as corundum or rich bauxite in presence of metallic copper in the electric furnace; this method I have previously described.\* Since that time, however, the old longitudinal furnace has been replaced by an upright one; the carbon walls now form the negative electrode, the positive being a bundle of carbon rods, plates, or a single carbon cylinder. The positive electrode is coupled to the cable as before, and is capable of being raised or lowered through an opening in the furnace cover; this cover is of iron and is kept cool by the circulation of cold water within it. At the bottom of the furnace is a tap hole closed by a carbon rod. By this method, a rich alloy containing 20 to 30 per cent. of aluminium is obtained, which is checked by analysis and standardised by the addition of copper. The following is a typical analysis of this crude bronze or "furnace product":—

Copper .....	71.517 (by difference)
Aluminium .....	20.147
Silicon .....	4.116
Iron .....	4.220

By using prepared alumina almost free from silica and iron, a very pure alloy can be obtained. 2. The better and more general method is by direct mixture of the metals in the crucible. The process is as follows:—The copper is first melted, and then the necessary quantity of aluminium added in portions, using a perforated ladle to facilitate mixture. For the first addition of aluminium there will be great evolution of heat rising to incandescence; this gradually

\* This Journal, September 1889.

diminishes as the remainder of the aluminium is added, care being taken to prevent overheating of the ladle and tools used during the mixing. The tools and ladle must be perfectly clean, free from scale, and it is best to change the hot rod for a fresh one. Blacklead pots must be used, and the greatest care taken to prevent the molten metal coming into contact with sand or siliceous matter, for aluminium readily reduces silicon from its compounds. Fluxes must not be used, but carbon may be placed over the molten metal if thought needful. Much trouble and failure to obtain good results in the foundry has arisen from using clay pots, neglecting these precautions, and using too high a mixing temperature. If a bronze requires to be as free as possible from silicon, crucibles lined with carbon must be used. This bronze is of a bright golden colour, its density is 7.69, it gives fine castings, is malleable, but requires frequent annealing if worked in the cold. It works well under the tool and takes a high polish.

The alloy containing 11 per cent. of aluminium is higher in tensile, transverse, and torsional strength; a small round cast bar tested by the Leeds Forge Co. Ltd. gave T.S. 57.27 tons, or 128,000 lb. per square inch.

This alloy is exceedingly hard and trying to the tools, and in it we have reached the highest useful limit of aluminium in low per cent. alloys. The alloys containing less than 10 per cent. aluminium are all of very great value. The tensile, transverse, and torsional strengths and resistance to compression decreases as the aluminium is lowered; this is accompanied by a proportional increase of elongation. Their colour varies from pale yellow to red gold; they can be readily cast, rolled, and drawn in the cold, and can be worked easily at bright red heat. Sheets, rods, bars, and wire are rolled at a bright heat and finished cold. The effect of working and rolling is shown in some of the tests given below. The fracture of the 10 and 11 per cent. bronzes are crystalline, that of the others fibrous.

You will notice that they far surpass ordinary bronze and gun-metals, and compare favourably even with steel.

The destructive effect of heat on gun metal (tin bronze) is one of the most serious objections to the use of that alloy. The aluminium bronzes, on the contrary, show no sign of liquation on melting, and there is no separation of the metals on slow cooling, and so sounder castings can be

TABLE I.  
ALUMINIUM BRONZE.—AVERAGE TESTS.

Aluminium Per Cent.	T.S. in Lbs. per Square Inch.	Elongation Per Cent.
10	69—100,000	4—14
8½	55—65,000	12—28
7½	35—45,000	8—32
5—5½	35—40,000	30—40
2	30,000	40—50
1	20,000	10—55
11	50—100,000	0—87.0

made from them. They retain their tenacity through a high range of temperature. Professor Unwin obtained the following result with a bar of this bronze:—

Specimen.	Heated to F.	T.S. in Lbs. per Sq. Inch.	Per Cent. of Elongation.
10 per cent. aluminium	350°	86,240	26.3
Bronze, rolled bar.....	500°	81,828	27.5

Other temperature tests made at Cowles' Company's laboratory are given in my paper in the Journal for 1889.

*Complex Alloys of Aluminium.*—Of these the most important contain zinc and copper with varying proportions of aluminium, forming the so-called aluminium bronzes. The composition of these alloys varies from 1 to 6 per cent. of aluminium combined with 12 to 43 per cent. of zinc, the remaining constituent being copper.

The following tables indicate the composition and mechanical properties of this class of alloy.

# ALUMINIUM BRASS.

Notes.	Composition of Alloy.				T.S. per Square Inch.		Elongation per Cent.	Authority.
	Cu.	Al.	Si.	Zn.	Lbs.	Tons.		
Chill .....	63.0	3.5	0.33	33.3	82,200	36.7	2.33	United States Naval Department.
Castings.....	63.0	3.3	0.33	33.3	70,400	31.4	0.40	
Sand castings .....	63.0	3.5	..	..	67,200 to 76,160	30 to 34	6 to 11	Average tests at Milton Works laboratory.
Roller bar.....	63.0	3.5	..	..	84,672	37.8	9.7	
Duplicate of above, rolled hot from 3 square inch pillar to 1½ inch, then when cold to 1 inch square pillar.	63.0	3.5	..	..	87,360	39.0	12.5	Pittsburg Aluminium Company, U.S.A.
	57.0	1.0	..	42.0	52,800	23.5	..	
	70.0	2.0	..	28.0	68,600	30.6	..	
	67.4	5.8	..	26.8	96,900	43.2	..	
Cast bars cut from propeller blade castings:— 6" × 1".....	..	..	..	..	76,187	34.0	1.5	Tested at Lloyd's proving house, Netherton, for Cowles Company, Limited.
12" × 2" .....	..	..	..	..	72,912	32.5	1.0	
18" × 3" .....	..	..	..	..	66,752	29.7	Slight	Neuhausen Company's laboratory tests.
	66.0	1.0	..	33.0	58,240	26.0	50.0	
	65.0	2.0	..	33.0	69,440	31.0	30.0	
	64.0	3.0	..	33.0	89,480	39.5	7.0	

TABLE II.  
COMPARISON OF ALUMINIUM BRONZE WITH GUN BRONZE, IRON, AND STEEL.

Metal or Alloy.—Description.	Tensile Strength.	Elastic Limit.	Elongation.	Authority.
	Lb. per Sq. In.	Lb. per Sq. In.	Per Cent.	
Steel, average of 137 specimens of accepted gun steel, oil tempered and annealed, hoops, jackets, and tubes, same grades of steel, 13 specimens not tempered or annealed.	96,150	51,611	19.93	United States Government tests of ordnance metal.
Cast gun steel, German soft.	88,000	38,140	18.70	
Cast gun steel, German soft.	70,784	33,392	.. ..	Holley ("Ordnance and Armour").
Wrought iron forgings:—				
small bars { Highest.	73,000	30,000	.. ..	.. ..
Mean.	53,000	18,000	26.0	.. ..
Heavy forgings, average of 700 tests.	48,160	23,760	.. ..	Sir W. G. Armstrong's Works tests.
Cast iron, average of four specimens of accepted iron.	30,000	17,000	0.166	Watertown Arsenal United States Government tests.
Gun bronze, average of 14 tests:—				
Copper 88.0, tin 10.0, zinc 2.0.	38,995	13,214	33.6	Watertown Arsenal tests.
" 92.0 " 8.0	20,008	.. ..	.. ..	Anderson.
" 91.7 " 8.3	31,001	.. ..	.. ..	"
" 91.0 " 9.0	32,995	.. ..	.. ..	"
" 90.0 " 10.0	37,900	.. ..	.. ..	"
" 92.0 " 8.0	43,200	5,672	40.0	General Uchatius.
" 88.0 " 10.0, zinc 2.0.	18,000	10,000	2.5	United States Navy Department tests.
" 82.0 " 10.0 " 2.5.	24,300	11,000	8.2	
" 82.0 " 10.0 " 2.0.	23,750	13,000	3.7	
Gun steel, average results of tests of Firminy, St. Etienne steel for French artillery.	98,134	57,796	16.0	Tests at gun factory of the Forges et Chantiers de la Méditerranée.
Aluminium bronze:—				
Al 10.0, Si 1.0, Cu 89.0	114,514	.. ..	0.15	United States ordnance tests.
" " "	109,823	79,894	0.05	
" " "	105,000	.. ..	6.0	
		Report reads probable elastic limit		
Aluminium bronze, Al 10, Si 1, Cu 89, rolled hot from 2 inch billet to $\frac{1}{2}$ inch round bar.	111,400	84,000	6.5	United States Government Watertown Arsenal test.
Aluminium bronze, Al 10, Si 1, Cu 89	128,400	.. ..	Nil.	Leeds Forge Company's test.
" " "	90,680	.. ..	5.7	C.S. Company's Works, Milton.
" " 10 per cent. Al	89,743	39,737	33.26	Professor Umoim.
" " cast in sand.	87,510	.. ..	17.0	Watertown Arsenal test.
Aluminium bronze, $\frac{7}{8}$ per cent. Al, Si 0.75 per cent., Cu 91.75 per cent.	69,800	21,500	32.8	" "
Cast in chill mould	68,000	24,000	18.2	
Aluminium bronze, Al 7.5 per cent., Si 0.75, Cu 91.75	60—65,000	.. ..	20.0	Otis Iron Works test.
Same after rolling into rod.	83,000	.. ..	39.0	Otis Iron and Steel Works, Cleveland, Ohio, U.S.
Aluminium bronze, Al 7.75 per cent., Si 0.75 per cent., Cu 91.5 per cent.	60,700	18,000	23.20	United States Government, Navy Department tests.
Aluminium bronze, Al 7.75 per cent., Si 0.75 per cent., Cu 91.5	67,600	24,000	13.0	
Aluminium bronze, Al 7.5 per cent., Si 0.5 per cent., Cu 92.0.	60,550	17,000	7.8	
Aluminium bronze bar, Al 5 per cent., rolled.	82,880	.. ..	60.0	Tested at Phoenix Works, Ruhort, Germany.
" " 10 per cent.	91,392	.. ..	1.5	Tests made at South Boston Iron-works by Mr. E. D. Self, on six-inch long bars.
" " 10	92,512	39,808	2.5	
" " 10	96,320	85,120	1.0	
" " 9	77,056	51,744	9.0	
" " 9	71,680	43,904	9.0	
" " 9	71,680	.. ..	8.5	
" " 8	57,792	.. ..	12.5	" "
" " 8 $\frac{1}{2}$	71,904	.. ..	28.5	E. D. Self, at South Boston Iron-works.
" " 7 $\frac{1}{2}$	60,700	45,472	6.0	

The percentage of aluminium in aluminium brass depends upon the amount of zinc present, since the aluminium must decrease in quantity with an increase of zinc, otherwise the alloy will become brittle and unworkable. With 40 per cent. of zinc the aluminium should not exceed 1 per cent., with 30 per cent. of zinc, 2 per cent. of aluminium, with 11 per cent. of zinc 6 per cent. of aluminium may be added. This alloy is best made by melting 10 per cent. aluminium bronze with the quantity of copper required, and when the ingots are melted and thoroughly mixed in the crucible, adding the zinc. The metal is left in the furnace until small test bars taken from it and broken show the required tensile strength and ductility; it is then ready for pouring.

Other alloys of this class contain small percentages of manganese and tin, usually not exceeding 0.5 of either metal.

The following is an example of this alloy

Specimen.	Composition.	Tensile Strength.	Elongation.
Cast bar of aluminium-manganese-bronze.	Aluminium ... 2.000	85,300 lb.	14.25 per cent.
	Zinc..... 34.000		
	Manganese... 0.300		
	Tin..... 0.500		
	Copper..... 63.200		
	100.000		

COMPARATIVE TRANSVERSE TESTS, ON BARS 1 IN. x 1 IN. x 12 IN.

Gun Metal.				Manganese Bronze.				Aluminium Brass.			
Stress Applied.	Deflection under Stress.	Permanent Set.	Resilience.	Stress Applied.	Deflection under Stress.	Permanent Set.	Resilience.	Stress Applied.	Deflection under Stress.	Permanent Set.	Resilience.
Cwts. 8	0.03			Cwts. 8				Cwts. 8			
10	0.045			10				10	0.02	0	
12	0.06	0.015	0.015	12				12			
14	0.09	0.04	0.05	14	0.06			14			
15	..	..	..	15	..	..	..	15	0.04	0	
16	0.15	0.09	0.06	16	0.07			16			
18	0.27	0.20	0.07	18	0.08	0.065	0.075	18	0.06	0	
20	0.46	0.38	0.08	20	0.09	0.01	0.08	20			
22	0.75	0.65	0.10	22				22	0.08	0	
24	1.08	0.97	0.11	24				24			
25	..	..	..	25	..	..	..	25	0.10	0	
26	1.50	1.46	0.13	26				26			
Slipped through supports.				28				28			
Permanent deflection, $3\frac{1}{2}$ in.				30	0.20	0.075	0.125	30	0.12	0.04	0.11
				32	0.23	0.105	0.125	32			
				34	0.28	0.140	0.140	34	0.15	0.02	0.13
				35	..	..	..	35			
				36	0.34	0.19	0.15	36	0.18	0.03	0.15
				38	0.41	0.26	0.15	38	0.19	0.03	0.16
				40	0.49	0.33	0.16	40	0.20	0.04	0.16
				42	0.59	0.425	0.165	42	0.21	0.05	0.16
				44	0.70	0.53	0.17	44	0.23	0.06	0.17
				45	..	..	..	45	0.24	0.08	0.16
				46	0.81	0.63	0.18	46	0.28	0.10	0.18
				48	1.16	0.96	0.20	48	0.31	0.12	0.19
				50	1.39	1.17	0.22	50	0.35	0.16	0.19
				52	1.70	1.47	0.23	52	0.40	0.20	0.20
				54	Fractured.			54	0.46	0.26	0.20
					Permanent deflection, $3\frac{1}{2}$ in.			56	0.54	0.32	0.22
								58	0.63	0.40	0.25
								60	0.67	0.45	0.22
								62	0.7	0.50	0.23
								64	0.79	0.56	0.23
								66	Break.		
								68	Permanent deflection, $\frac{1}{2}$ in.		

Metal for rolling into sheets and rods has often 0·5 per cent. of lead added to the alloy. Aluminium brass can be forged and worked readily under the hammer; the working heat must be lowered with the decrease in the percentage of aluminium. The 3 per cent. aluminium alloy with 33 per cent. of zinc can be worked at a dark red heat, contrasting in this respect with common brass. These alloys all give sharp, clean, tough castings, and from their high tensile and

transverse strength and their corrosion resistance they ought to be most valuable to the marine and mechanical engineer. They have already been tried as a material for propeller blades, and, judging from results obtained, ought to displace other bronzes in this class of work.

The following are comparative tests of transverse strength:—

COMPARATIVE TESTS OF TRANSVERSE STRENGTH.

Material.	Cross Section of Bar in Inches.	Length of Bar between Supports.	Breaking Load.	Limit of Elasticity.	Permanent Deflection.	Resilience.		Percentage Composition.
						At Elastic Limit.	At Ultimate Strength.	
Cast iron.....	1	12	Cwt. 16 to 20	..	..	..	..	..
Gun metal.....	1	12	26	18	3½	0·07	0·13	..
Manganese bronze.....	1	12	54	38	3½	0·15	0·23	..
Aluminium-Hercules bronze..	1	12	56	50	3¼	0·18	0·24	Al ½ - 1 per cent. Zn 40 - 42.
Aluminium-manganese bronze.	1	12	61	56	1·8	0·22	0·25	Al 2½, Mn ½, Zn 34½, Sn ½.
Aluminium brass.....	1	12	72	64	!	0·2	0·23	Al 3½, Zn 33½.

*The Casting of Aluminium Bronze and Brass.*—The following are the melting points of aluminium bronze, as compared with the pure metal and other alloys and metals:—

Metal or Alloy.	Melting Point.
	° F.
Aluminium.....	1,300
Copper.....	1,900
Iron.....	Cast, above 2,780
	Wrought, above 1,000
Steel.....	1,000
Steel bronze (gun bronze).....	1,100—1,200
Aluminium bronze (aluminium brass).....	1,000—1,700

In making castings from aluminium bronze and brass the difficulties, as in other metals and alloys, arise from oxidation, contraction, shrinkage, and the enclosing of air bubbles, but no gases are produced during the melting of the alloy, and there is no trouble from the occlusion of gases, as in the case of other metals.

The liquid bronze on exposure to air is instantly covered with a thin film of oxide, which protects the under metal from any further oxidation; in pouring, care must be taken to prevent any of this film getting into the casting; this is best done by making a "pouring gate" or receiver, into which the metal is first poured.

The molten bronze is kept back until the impurities have risen to the surface, when the plug is withdrawn and the clean metal enters the mould. Instead of a "pouring gate" a secondary pot or ladle can be used.

Contraction must be guarded against by having cores of a yielding character, avoiding as far as possible core-rods or irons; coarse sand mixed with rosin, which softens on heating, makes a good core. To diminish shrinkage the gates must be made as large as possible and the pouring done quickly, and at no higher temperature than is necessary to ensure a good casting.

CORROSION RESISTANCE OF ALUMINIUM ALLOYS. NEUBAUER LABORATORY TESTS.

Specimen.	Weight in Grams.	Surface.	Reagent.	Temperature.	Time of Exposure.	Loss Per Cent.
2·4 per cent. aluminium bronze.....	9·346	50 cm. sq.	5 per cent. acetic acid.	Ordinary.	504 hours.	0·440
10 per cent. free from silicon.....	..	Equal surfaces.	5 per cent. acetic acid and 3 per cent. solution of common salt.	80°—90° C.	14 "	1·0
10 per cent. aluminium bronze with 2·8 per cent. of silicon.*					14 "	2·1
Aluminium brass, 3·5 per cent. aluminium.						4·4

\* A specimen tested in the Milton Laboratory indicates the influence of silicon on corrosion resistance. A polished bar of 10 per cent. bronze, normal colour, strength, and elongation, gave following result:—

17·432	22 cm. sq.	Pure conc. H <sub>2</sub> SO <sub>4</sub>	6°—11° C.	336 hours.	0·401
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The action of the sulphuric acid developed a dark band in form of a spiral round the bar; this showed minute corrosion pits under lens, which gave off sulphuretted hydrogen; the corrosion was apparently confined altogether to this dark patch. Silicon alloyed with iron is often 1·5 as a black insoluble powder on dissolving the bronze in aqua regia.

## HIGH PER CENT. ALLOYS.

Composition.		Specific Gravity.	T.S. in Tons per Sq. Inch.	T.S. in Lbs. per Sq. Inch.
Al.	Cu.			
98	2	2.71	19.6	43,904
96	4	2.77	19.90	44,576
94	6	2.82	21.70	55,328
92	8	2.86	22.70	50,848
Aluminium	..	2.67	12.0	26,880
Al, 97-98	Titanium, 2-3	..	17.8	40,000
Langley's patent.	Langley's patent.		26.7	60,000

**High per cent. Aluminium Alloys.**—Very many experiments have been made to increase the tensile strength and hardness of aluminium, and with considerable success, though at some slight sacrifice of lightness and corrosion resistance. For ordinary foundry and workshop castings the addition of 1-6 per cent. of copper is the most ready and serviceable method. The mechanical tests of such an alloy are shown in the table. The addition of only 2 per cent. of copper increases the tensile strength of rolled aluminium from 12 to 19½ tons per sq. inch, while 6 per cent. doubles it; a further addition of copper increases both the hardness and brittleness, but the alloys containing between 12 and 90 per cent. of aluminium are industrially valueless.

**The Alloys with Tin.**—From my own experiments I find the addition of not more than 2½ per cent. of tin to aluminium gives a hard serviceable alloy with a slightly improved colour. Richer alloys are crystalline and brittle.

(Samples containing 2 per cent. and 50 per cent. of tin were exhibited.)

**The Alloys with Titanium.**—For information as to these alloys I am indebted to Dr. J. W. Richards, Professor of Metallurgy at Letrigh University, U.S.A. The alloy containing 2 to 3 per cent. (Langley's patent) gives a tensile strength of 40,000 to 60,000 lb. per sq. inch in forgings; this alloy is especially useful for horse shoes, stirrups, bits, and small, strong fittings.

**Alloys with Nickel and Zinc.**—The addition of nickel lowers the melting point, and increases the hardness and elasticity, if added in quantities not exceeding 2 per cent.; above this figure the alloy is brittle and useless.

With zinc, aluminium forms hard, close-grained, brittle alloys having a lower melting point than that of the constituent metals. A small ingot containing 10 per cent. of aluminium and 90 per cent. of zinc is among the samples on the table. A very useful alloy contains 3 per cent. of German silver. It has double the tensile strength of aluminium itself. Professor Richards had samples of it tested at Watertown Arsenal, and while not quite equal to the titanium alloy in tensile strength, its elasticity was greater; this alloy is suitable for knives, spatulas, paper cutters, spectacle frames, and all uses where elasticity is essential.

**Alloys with Silver.**—Aluminium alloys readily with silver in all proportions. With up to 6 per cent. of silver the elasticity and hardness increase, beyond this proportion the brittleness increases, but alloys containing up to 30 per cent. of silver are serviceable. These alloys, owing to their hardness, lightness, bright lustre, and permanency, are excellent for art work.

**Chromium Alloys.**—For a description of these alloys I have to thank Mr. H. N. Yates. He finds from experiment that the addition of chromium increases the hardness, while it reduces the strength and ductility of the aluminium.

**Cadmium Alloys.**—Aluminium is said to form malleable fusible alloys with cadmium in all proportions, but I have not been able to verify this statement.

**Alloys with Iron, &c.** Aluminium alloys readily with iron in all proportions. Aluminium containing more than 1 to 2 per cent. of iron is hard and brittle; with 8 per cent. the alloy crystallises in needles.

Iron containing 15 to 16 per cent. of aluminium cast in the chill is marked by the file with difficulty; the fracture is fine and crystalline.

An alloy of steel containing 1 per cent. of manganese with 7 per cent. of aluminium will just scratch glass. Although aluminium is said by some writers to diminish hardness in iron, used in even small proportions, it is to my mind sufficiently clear that this depends entirely upon the change in the state of the contained carbon.

Above 5 per cent. of aluminium rapidly destroys the magnetic properties of iron, and some time ago experiments were carried out in the Milton laboratory to determine whether this phenomenon would furnish any clue to the percentage of aluminium present in iron alloys, but the results were not precise enough for quantitative indications.

**The Use of Aluminium in Melting and Casting other Metals.**—Very large quantities of the aluminium produced go for use in iron and steel founding in this country, and Professor Richards and Mr. Yates report that there is hardly a steel melter in the States that tries to do without it, and that they are the largest users. This brings me to the consideration of the influence of minute quantities of aluminium on other metals. The percentage used is so small that such mixtures cannot be truly termed alloys. The action of aluminium is probably twofold: chemical and physical. Chemical, owing to its affinity for oxygen and the stable character of the oxide when formed; physical, causing changes in the structure of the alloy, crystalline to non crystalline.

The effect of aluminium is due to—

First, Its action on the combined carbon, changing it to the graphitoidal form; the carbon is apparently liberated at the moment of solidifying and uniformly. 0.25 per cent. of aluminium added to white iron renders it perceptibly darker in colour, 0.5 per cent. darker still, and 0.75 per cent. renders it grey with no sign of white. Up to 4 per cent. the effect is similar; the castings are softer and greyer as the aluminium increases.

Secondly, Its action on the dissolved oxides of iron and silicon. These are reduced with the formation of alumina, iron, and free silicon.

Thirdly, Decomposition of gaseous and solid compounds of carbon with oxygen and hydrogen entangled in the molten metal. This is why aluminium is so valuable in steel casting. Some experiments carried out by Professor Langley, of Pittsburg, explains this action clearly. Blow holes in steel are due to non-oxidising occluded gases, hydrogen, carbonic oxide, and nitrogen separating, under a pressure of many atmospheres, from steel just before solidification. It has been proved by repeated experiments that aluminium readily decomposes, carbonic oxide below steel melting heat, forming alumina and free carbon. Professor Langley confirmed this by blowing 40 gallons of pure carbonic oxide through a crucible of molten steel containing aluminium, and obtained an increase of 35 per cent. on the carbon present before passing the gas. The quantities of aluminium used vary with the quality of the steel. For open hearth metal with less than 0.5 per cent. of carbon, 5 to 10 oz. per ton. For steel over 0.5 per cent. of carbon, aluminium must be used cautiously, the quantity usually being 4 to 8 oz. per ton.

These quantities, you will notice, are very minute; 4 oz. per ton being 0.0125 per cent., 16 oz. would be 0.05 per cent. Professor Richards and Mr. A. E. Hunt have patented an alloy made by adding 10 to 20 per cent. of aluminium to ferro-manganese. The addition of the aluminium causes a separation of carbon, and an alloy with less carbon than ordinary ferro-manganese is obtained, admitting of melting softer steel with down to 0.05 per cent. of carbon. As might be expected from its influence on iron and steel, aluminium is useful in the preparation of other metals. It is much used by American galvanisers and brass founders for adding to molten zinc; its action is to reduce the oxide disseminated through the bath. Its effect on the galvanised sheets is at once perceptible. The amount used



is 0.005 to 0.010 per cent. of aluminium, usually in the form of the 4 per cent. aluminium alloy of zinc; one firm in the United States puts 2,000 tons of aluminium zinc on the market yearly. The process is patented in this country. A small quantity added to copper, 0.05 to 0.5 per cent., decomposes entangled oxides and enables sounder and more ductile castings and ingots to be produced without hardening the metal or affecting its electrical conductivity.

In conclusion I would say that the true value of aluminium is not yet known as it ought to be to those engaged in the metallurgical industries of this country, and in its use and applications we are behind the Continent and the United States. In the arsenals, shipbuilding yards, machine shops, and factories of France, Germany, Switzerland, Italy, Denmark, Russia, and the United States, aluminium and its alloys are taking the place of the older tin, zinc, and copper alloys, of cast iron, and in some cases that of steel itself.

I have to express my thanks to Professor Richards and Mr. H. N. Yates for communication of facts as to experiments and use of the metal in the United States; to the Cowles Syndicate Co. for specimens of alloys; to Messrs. Blackwell for specimens of bauxite, to Mr. Lloyd Barnes, of the Liverpool School of Science, for a test of the electrical conductivity of aluminium; to Dr. Bailey for assistance in preparation of the diagrams; and to Mr. C. Boundy for the use of his metallurgical laboratory.

#### DISCUSSION.

Mr. W. P. THOMPSON asked if Mr. Dagger had seen any purple alloys of aluminium, and what was his knowledge with regard to them.

Dr. HURTER remarked that what had struck him very much was the considerable increase in the strength of the metals obtained by adding trifling quantities of aluminium. Taking, for instance, a cast with 3 per cent. of aluminium with 33 per cent. of zinc, there would be 64 of copper. If they divided the several weights by the molecular weights they would find on 0.11 molecules aluminium about 1.6 atoms of other metals. That would be on one atom of aluminium about 15 of copper and zinc. But if they considered that as a cube with so many molecules in it, they would find that on the linear dimensions there would be 1 of aluminium to 3 of the other metal. It struck him that, looking only at the percentages, it did not give one a just idea of the amount of aluminium present. He was pleased that Mr. Dagger brought forward those papers from time to time. He was lately in London, and a gentleman undertook to show him a few experiments, and he was surprised to learn a good many things from them. He had been shown the enormous affinity which aluminium had for sulphur, a thing which one did not altogether realise. It would take sulphur away from nearly every other metal. Aluminium sulphide would be a boon in the laboratory, if only cheap enough, for the preparation of sulphuretted hydrogen. One only needs to pour water upon it when an abundant stream of sulphuretted hydrogen is given off. Aluminium had many properties which rendered it valuable to the chemist, and, comparatively speaking, it was still an unknown substance, and would, no doubt, play an important part in metallurgy.

Professor CAMPBELL BROWN said the subject was full of interest, but there was one alloy mentioned about which he would like more information. Could Mr. Dagger give them any information of how 2 per cent. of titanium was alloyed with aluminium?

The CHAIRMAN said they were all very much struck with the progress made in the metallurgy of aluminium and the immense variety of uses to which it and its alloys could be put. What would a casting of aluminium brass cost per lb. or per cwt. as compared with other alloys of equal tensile strength, or with the best alloy that was known? Mr. Dagger had mentioned screws for steamers; and, assuming the aluminium brass to be very much stronger than another material, a screw, although made of the more expensive alloy, might be really cheaper per lb. or cwt. than the best

made of some other metal. He would also ask what was the present price of aluminium, and what prospect Mr. Dagger thought there was of a reduction of cost?

Mr. A. E. FLETCHER asked where aluminium was produced in the largest quantities.

Mr. DAGGER, in reply, said he was sorry he had not seen any purple alloys of gold and aluminium, but Professor Roberts Austen's description of it was exceedingly interesting, and it made them think how very little they really knew as to the true nature of alloys. Regarding Dr. Hurter's questions, he could not remember with any accuracy the tensile strength of pure copper as compared with aluminium. The figures, however, were given in his last paper, and published in the Journal (this Journal, 1892, 125). Dr. Hurter had compared bulk with per cent., and looking at it that way, it was not so startling after all, because, weight for weight, they would use almost three times the amount of aluminium as they would of copper. The reducing properties of aluminium led one to suppose it would be useful to the chemist in laboratory work, and a great deal had to be done in that direction, as there was plenty of scope for chemists to try how far it would replace the metals that were at present used. Aluminium did not alloy with lead. He had before him a sample that he had made—a very carefully prepared alloy of 50 per cent. tin and lead. It was carefully mixed in the casting, and he had no doubt at all that the tin and lead were thoroughly alloyed together. He then added 50 per cent. of aluminium to that mixture, taking the same precautions, and poured it into a mould, and the aluminium separated the lead from the tin; the two layers could be very distinctly seen. Such a property would render it very useful in the separation of silver from lead. Professor Campbell Brown had raised the question of titanium in alloys. He had not seen that alloy, but understood that it was prepared by reducing titanous oxide with alumina in the furnace. By that means a rich alloy was obtained. Regarding the cost of aluminium alloys, he was sorry to say that was a question he could not answer right off, having no figures with him. The present price of aluminium had not been reduced since he read his last paper nearly two years ago. It remained practically at about 2s. per lb. in ingots of 98.7, or say 99 per cent. in a ton lot. In reply to Mr. Fletcher, the largest factory was undoubtedly that of the Cowles' Company in Lockport, N.Y., U.S.A. Next came the Neuhausen Company, in Switzerland.

## Manchester Section.

CHEMICAL THEATRE, OWENS COLLEGE.

Chairman: Ivan Levinstein.

Vice-Chairman: Edw. Schunck.

Committee:

G. H. Bailey.  
F. H. Bowman.  
R. F. Carpenter.  
G. E. Davis.  
C. Dreyfus.  
H. Grimshaw.

P. Hart.  
J. M. Irving.  
E. Knecht.  
W. H. Perkin, jun.  
Sir H. E. Roscoe, M.P.  
C. Truby.

Hon. Local Secretary:

J. Carter Bell,

Bank House, The Cliff, Higher Broughton, Manchester.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

SESSION 1893-94.

Feb. 2nd, 1894:—

Mr. Wm. Thomson, F.R.S.E. "The Protection of Metallic Iron from Corrosion."  
Mr. J. Carter Bell. "Notes on the Fertilisers and Feeding Stuffs Act, 1883."

Meeting held Friday, January 5th, 1894.

DR. CHARLES DREYFUS IN THE CHAIR.

### THE MANCHESTER SHIP CANAL.

The CHAIRMAN referred to the importance of the opening of the Ship Canal in its bearing on the chemical trades, and submitted a motion congratulating the directors and shareholders of the Canal Company on the completion of the canal, and wishing success to the undertaking.

The motion was seconded by Mr. Peter Hart and adopted.

Mr. HARRY GRIMSHAW, in supporting the resolution, said, that in fixing the rates for the carriage of chemicals, there seemed to be an amount of uncertainty and ignorance, which ought not to exist amongst railway companies and other carriers. They failed to realise the magnitude of the chemical industries of this country. The quantity of iron ore, sulphur, and the various forms of alkali and other heavy chemicals that came into this district, would run into hundreds of thousands of tons per annum, and it behoved the members of the Society to impress upon the Ship Canal Company and the carriers on the canal the magnitude of the trade, and the importance of the rates for freight being reduced, in order to induce a greater development of the trade in the district.

The CHAIRMAN stated that the matter was already under the consideration of the chemical section of the Manchester Chamber of Commerce, but possibly good might be done by the appointment of a committee to take action on behalf of the Society, and thereby strengthen the hands of the Chamber of Commerce. Liverpool shipowners looked upon chemicals with great disfavour, and this placed this country at a very great disadvantage as compared with other countries. There was no difficulty whatever in shipping any kind of chemicals from such places as Antwerp, Rotterdam, and Havre, or Hamburg, where the charges were not so prohibitive as they were at Liverpool or any place on the east coast. This was mainly owing to the ignorance which prevailed amongst British shippers as to the name and nature of certain chemicals, for the moment one called goods chemicals the Liverpool shippers were frightened. Copperas, for instance, was charged differently from sulphate of iron, which was exactly the same thing. He hoped they would succeed in getting facilities in Manchester such as they had not been able to obtain from Liverpool.

It was agreed to postpone to a future meeting any action in the direction suggested by the Chairman.

### ON THE VULCANISATION OF INDIA-RUBBER.

BY CARL OTTO WEBER, PH.D., F.C.S.

THE chemical nature of india-rubber is very little better known to-day than about 60 years ago, when Gregory\* first examined the products of the dry distillation of this remarkable substance, and found them to consist of a mixture of hydrocarbons boiling between  $-18^{\circ}\text{C}$ . and  $360^{\circ}\text{C}$ . These experiments were repeated later on by Dalton,† Himly,‡ Williams,§ and subsequently by Bouchardat|| with the result that these hydrocarbons were found to belong to the class of the terpenes or polyterpenes. The most important feature of these investigations, no doubt, was the discovery of isoprene  $\text{C}_5\text{H}_8$  (hemiterpene), and the observation that the greater part of the mixture of hydrocarbons obtained, consists of a hydrocarbon, boiling

between  $168^{\circ}\text{C}$ . and  $176^{\circ}\text{C}$ ., and which was found to be identical with di-isoprene or dipentene, a terpene very closely related to citrene.

At present isoprene must be considered the most interesting of the products of the dry distillation of india-rubber. Bouchardat\* observed that by heating it in sealed tubes to about  $300^{\circ}\text{C}$ ., or by treating it with fuming hydrochloric acid it could be reconverted into india-rubber. Tilden† obtained isoprene by distilling turpentine through porcelain tubes at a dull red heat, and discovered 'the poorly' that this compound, on standing, spontaneously polymerises into india-rubber. I am in a position to confirm this observation with respect to isoprene obtained from india-rubber by dry distillation. About 300 grms. of this substance were found after nine months standing to be converted into a very viscid, treacly mass, from which by treatment with methyl alcohol, a solid spongy substance of almost white colour could be separated. This substance, after drying, was of a light-brown colour, and in all respects identical with india-rubber. The methyl alcohol contained about 82 grms. of a thick oil, boiling at about  $215^{\circ}\text{C}$ ., and combining with bromine with formation of a tetrabromide. The weight of india-rubber thus obtained was 211 grms.

Whether in the future we shall succeed to manufacture india-rubber artificially by means of isoprene appears very doubtful as yet. The manufacture of cheap isoprene is certainly an exceedingly difficult task to start with, and on the other hand we have not succeeded so far to completely polymerise it into india-rubber. The by-products of this polymerisation consist of dipentene and polyterpenes, which will always remain comparatively worthless, there being any amount of cheap natural terpenes. At the present time the constitution of isoprene itself is very doubtful. We only know that it contains two ethylene bonds, and for that reason must be a derivative either of allene,  $\text{CH}_2=\text{C}=\text{CH}_2$ , or of vinyl ethylene,  $\text{CH}_2=\text{CH}-\text{HC}=\text{CH}_2$ . Also dipentene contains these two ethylene bonds as is proved by its behaviour against hydrochloric acid, or hydrochloric acid and nitrosyl chloride. Isoprene very easily polymerises into dipentene, and as both these bodies contain two ethylene bonds it is evident that the polymerisation is effected by the resolution of one ethylene bond in each of two molecules of isoprene. Isoprene is, however, able to form a number of higher polymerisation products, of which  $\text{C}_{10}\text{H}_{16}$  and  $\text{C}_{15}\text{H}_{24}$  have been known for some considerable time. The oil I obtained besides india-rubber by the spontaneous polymerisation of isoprene contains about 20 per cent. of dipentene, the rest is a mixture of two substances which so far I failed to separate.§ The estimation of the depression of the freezing point in Beckmann's apparatus strongly indicates this mixture to consist of condensation products of the formula  $\text{C}_{33}\text{H}_{56}$  and  $\text{C}_{40}\text{H}_{64}$ . All these condensation products of isoprene are terpenes or polyterpenes, and the fact that the results of the analysis of india-rubber leads to the empirical formula  $\text{C}_{10}\text{H}_{16}$ , which is that of the terpenes might induce us to pronounce also india-rubber a terpene, or possibly a polyterpene  $(\text{C}_{10}\text{H}_{16})_n$ . Gladstone and Hibbert|| showed that for  $\text{C}_{10}\text{H}_{16}$  india-rubber contains three pairs of carbon atoms doubly linked, and this seems to be confirmed by the existence of a chlorinated derivative  $\text{C}_{10}\text{H}_{14}\text{Cl}_8$ , which corresponds to an addition product  $\text{C}_{10}\text{H}_{12}\text{Cl}_6$ . But terpenes with three ethylene bonds are quite unknown at present, so that unless further evidence be adduced we are scarcely justified to describe india-rubber as a terpene. The above mentioned facts leave very little doubt that the constitution of india-rubber differs very essentially from that of the terpenes and polyterpenes as at present understood. What we know with certainty is the formation of india-rubber from isoprene by polymerisation, and we further know that the molecular weight of it is much higher than  $\text{C}_{10}\text{H}_{16}$ . Under these circumstances it appears very much preferable to describe india-rubber as a polyterpene  $(\text{C}_{10}\text{H}_{16})_n$ . We shall have to bear in mind at the same time that india-rubber

\* Journ. pr. Chem., 9, 387.

† Journ. pr. Chem., 10, 131.

‡ Clap. Chem., 16, 161.

§ Phil. Trans. 1860, 241.

|| Comptes rend., 87, 654 and 89, 361, 1117.

\* Loc. cit.

† Journ. Chem. Soc., 45, 410.

‡ Chem. News, 65, 265.

§ Fractional distillation does not avail.

J. C. S., 1888, 679.

is an unsaturated hydrocarbon, a fact which must find expression throughout in the chemical behaviour of india-rubber, and which must be taken into consideration in the chemical influences we subject it to.

The chemical treatment of india-rubber in the manufacture of india-rubber articles is very limited at present and consists exclusively in the treatment known as vulcanisation, which is effected by subjecting the india-rubber either to the action of sulphur at temperatures above the melting point of sulphur, or with chloride of sulphur in the cold.\* The chemical reaction taking place in these treatments has so far scarcely been investigated, still less satisfactorily explained. The authors of the various papers on india-rubber express themselves all with more or less reserve on this point, describing vulcanisation as an "absorption" of sulphur by the india-rubber without explaining the meaning of this term. They are almost unanimous in stating that india-rubber "absorbs" about 2 per cent. of sulphur, leaving however the nature of this absorption undecided. Much more unsatisfactory even are the terms in which the nature of the vulcanisation with chloride of sulphur is described. On the one hand we are told that this treatment also results in an absorption of sulphur by the india-rubber, while on the other hand we are assured that the vulcanising action of the chloride of sulphur is due to its chlorine exclusively, one author going so far as to say chloride of sulphur was merely chlorine in a diluted form. As far as these conflicting assertions are supported by experimental proofs they are certainly deserving consideration. Unfortunately the proofs adduced in either case are, as we shall see presently, of a very unsatisfactory description. Moreover, the chemical reactions which are supposed to take place in these vulcanisation processes are of such an extraordinary kind that they are utterly irreconcilable with each other and still more so with the well-established laws and facts of organic chemistry. I cannot illustrate this better than by giving a selection of extracts from the various papers and other publications bearing upon this subject.

In the first instance the unanimity with which the vulcanisation process is described as a substitution process is greatly surprising in view of the fact that the hydrogen which is being substituted, as far as those authors account for it at all, generally behaves in a manner which is utterly at variance with all we know about substitution processes in organic compounds. With regard to the vulcanisation with sulphur Burghardt† says that "it consists in all probability in the alteration of the caoutchouc resin through the production in it of a 'sulpho-compound,' a certain amount of hydrogen in the caoutchouc being replaced by sulphur."

Burghardt evidently entirely overlooks the fact that in this case sulphuretted hydrogen would be evolved. As a matter of fact whether we vulcanise with sulphur on a small or on the large scale, not a trace of sulphuretted hydrogen, still less hydrogen is given off. Terry‡ also considers vulcanisation a substitution process, accompanied by evolution of sulphuretted hydrogen, but in which manner and under which circumstances he observed the appearance of this gas is not stated. Peculiarly his own is his view of the function of lead oxide in the vulcanisation process. This oxide is added to take up the excess of sulphur which is necessary in the vulcanisation with sulphur and which would prove very detrimental to the durability of the vulcanised articles if remained in the free state in the rubber. Terry§ remarks that no other metallic oxide can replace the oxide of lead for this purpose, and then goes on to say: "Owing to the formation heat of most of the metallic sulphides being above the melting point of sulphur, the sulphide of lead may be taken as the only one formed during ordinary vulcanising processes."

I have not been able yet to fathom the meaning of this sentence and came in the meantime to the conclusion that

\* I am well aware that it has been suggested to vulcanise india-rubber with vaporised chloride of sulphur, *etc.* at higher temperatures, and this process was indeed at one time, especially in America, largely used. But the characteristic property of chloride of sulphur as a vulcaniser consists in its capability to exert its full effect in the cold, even when strongly diluted with indifferent solvents, such as carbon bisulphide, benzene or the light petroleum hydrocarbons.

† Thorpe, *Dict. of appl. chem.*, Vol. II., 312.

‡ Terry, *this Journ.* 1892, 970-974.

§ *Loc. cit.*

Terry has never grasped the meaning of the term formation heat, but seems to consider it as a quality comparable with temperature.

The statements given above contain the whole of our present knowledge of the so-called dry vulcanisation process with sulphur. Our present notions regarding the so-called cold vulcanisation process with chloride of sulphur are, if possible, in even a greater state of confusion. Fawsitt\* maintains that vulcanisation in this case consists in the substitution of sulphur for hydrogen, which latter, according to him, escapes as a gas. What happens to the chlorine of the chloride of sulphur is left to the imagination of the reader. Burghardt† in the main adopts Fawsitt's explanation, but he deals in a more rational manner with the chlorine, which he says is given off in the form of hydrochloric acid. Diametrically opposed to these statements is Thomson, who gives it as his opinion‡ that the vulcanising action of chloride of sulphur is due to the chlorine and not to the sulphur of this compound. He asserts that india-rubber vulcanised with chloride of sulphur contains only minute quantities of sulphur as an impurity, but large quantities of combined chlorine and that the same is the case with the oils converted into india-rubber substitute by means of chloride of sulphur. Thomson gives us no information, whether he tried to vulcanise india-rubber with chlorine alone, or whether he obtained india-rubber substitute by treating suitable fatty oils with chlorine. Thomson's results were considered rather startling when first published, but I think his analytical method by which he obtained them is more startling still, and I consider it a matter of great surprise that it never has been challenged. This method consists in fusing the vulcanised rubber or rubber substitute, as the case might be, with caustic potash, dissolving the melt in water, oxidising the "lower sulphur compounds" with peroxide of hydrogen, and precipitating the sulphuric acid with barium chloride. It is a matter of surprise that by means of this method Thomson should have found any sulphur at all in these compounds. At any rate, before applying it to the analysis of these unknown compounds, its suitability for the purpose ought to have been tested on other sulphuretted hydrocarbons of known composition. After this neither the method, nor the startling results it furnished, would ever have got into print. Thomson's view respecting vulcanisation with chloride of sulphur is evidently shared by Terry, on grounds about as remarkable as Thomson's analytical method. Terry expresses himself in the following manner‡:

"With regard to the action of this body (chloride of sulphur), I think there can be no doubt that it is due to the chlorine, and that although some sulphur must necessarily be absorbed at the same time by the rubber, yet that this latter does not play any part in the reaction. Sulphur in the cold has no effect on rubber, and chloride of sulphur must be looked upon as merely chlorine in a diluted form. The action of chlorine in whatever form applied may be measured by the amount of hydrochloric acid produced."

From this we should have to conclude, that since free sulphur in the cold has no action upon rubber, the sulphur of the chloride of sulphur can have no vulcanising action, this vulcaniser being even effective in the cold. A criticism of this argument would be a mere waste of time: the great chemical energy and activity even the most inert elements acquire in many of their combinations is a subject which is treated of in every elementary text book of chemistry. From the last part of the above quoted sentence from Terry's paper it is evident that he also considers the chloride of sulphur vulcanisation a substitution process. Accordingly of one molecule of chloride of sulphur (135 parts) used for vulcanisation only one atom of chlorine (35.5 parts) could remain in the rubber. It is difficult to understand why Terry omitted to ascertain the correctness of his views by estimating experimentally, at least the amount of chlorine combining with rubber from a definite weight of sulphur chloride.

No more fortunate is Terry in his explanation of the action of Fawsitt's new vulcanising agent antimony iodide.

\* *This Journ.* 1886, 638.

† *Loc. cit.*

‡ *Industries*, 9339.

He writes\*—

"As far as I have gone into the matter myself I think that some substitution of hydrogen by iodine certainly does take place" and further on.

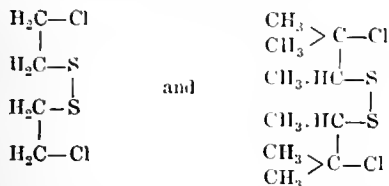
"The formation of hydriodic acid cannot be taken as proof that substitution of hydrogen in the rubber has taken place, because hydrogen would be liberated by the action of sulphur present."

From this we must assume that iodine can substitute hydrogen in organic compounds with evolution of hydriodic acid, and further, that sulphur can be substituted for hydrogen, the latter simply being set free. These lines are in complete opposition to our entire knowledge on substitution.

The above quotations are a summary of all that chemical literature contains on the subject of the vulcanisation of india-rubber, and after critical examination, we can only come to the conclusion, that neither the vulcanisation with sulphur, nor that with chloride of sulphur are in the least understood. Even such a simple question as that regarding the minimum quantity of sulphur required for vulcanisation, or the equally simple one whether the vulcanising action of chloride of sulphur is due to the sulphur or to the chlorine, are still objects of controversy. The work on which I am going to report to you to-night has been undertaken with a view to arrive at a definite settlement of these points and at the same time to clear up the chemical mechanism of the vulcanisation processes in general. Historically, the vulcanisation with sulphur preceded the vulcanisation with chloride of sulphur. Notwithstanding this, I first took up the investigation of the last named process, for the only reason that in this last case the vulcanisation is effected under conditions which are much more easily kept under control than is the case with the first process.

#### 1.—Vulcanisation with Chloride of Sulphur.

The action of chloride of sulphur upon hydrocarbons has been studied very little. Whether it is at all capable to react with saturated hydrocarbons is doubtful, and the chemical literature affords no information on this point. But, on the other hand, there are a few compounds known, obtained by the action of this substance upon unsaturated hydrocarbons, and these compounds appear especially interesting if we remember that the nature of india-rubber, as that of an unsaturated hydrocarbon, is one of the few well-established facts regarding the chemical constitution of this product. Guthrie† obtained ethylene chlorosulphide,  $(C_2H_4)_2.S_2Cl_2$ , and amylene chlorosulphide,  $(C_5H_{10})_2.S_2Cl_2$ , by acting upon ethylene and amylene respectively with chloride of sulphur. Both these compounds are heavy oily liquids, which readily give up their chlorine to various reagents without suffering any change in their constitution. The removal of the sulphur, however, seems to be almost impossible without destroying the molecule. Indeed, it appears that the sulphur can only be got at by treating those compounds with strong oxidising agents, the action of which invariably results in a complete breaking up of the molecule. These chlorosulphides are saturated substances, and their constitution is respectively—



We observe now the important fact that these compounds are addition products pure and simple. This fact becomes of the greatest significance if we remember that india-rubber must be chemically described as a poly-isoprene (poly-prene), and that isoprene itself is one of the five isomeric

pentenes which on their part are derived from five isomeric amylenes.

These chlorosulphides, as already stated, are perfectly saturated bodies, but we see at the same time that the characteristic feature of the reaction between the unsaturated hydrocarbon and the chloride of sulphur is not so much the production of a saturated addition product, but essentially the condensation, or rather conjugation, of two molecules of the hydrocarbon by means of the double sulphur atom. As long as this double atom maintains its function, we may remove the chlorine from these addition products or substitute other elements or radicals for it without in the least altering the general character of the compound.

The products of the action of chloride of sulphur upon the triglycerides of the unsaturated fatty acids are evidently compounds of a similar order. It has been impossible so far to isolate from these products well-defined chemical individuals, chiefly in consequence of the almost absolute insolubility of these compounds in all solvents. But it has been known for some time that these products, which are extensively used as india-rubber substitutes, contain both sulphur and chlorine as essential constituents. Quite lately Ulzer and Horn,\* and especially Henriques,† proved conclusively that these india-rubber substitutes contain sulphur and chlorine in the same proportions in which they form chloride of sulphur. These results are of course directly opposed to Thomson's figures, which, however, have been obtained by such a questionable method that they cannot be taken seriously.

Though limited, our present knowledge on the action of chloride of sulphur upon the before-mentioned unsaturated compounds affords some important hints regarding the chemical process of the vulcanisation of india-rubber by chloride of sulphur. It seemed to me important to investigate this vulcanisation process under such conditions that the total quantity of the rubber employed for each experiment would take part in the reaction at the same rate and to the same degree, and to carry out the experiments in such a manner as to preclude secondary reactions due to impurity of the materials employed. All former experiments on this subject did not comply with these conditions, the experiments always being carried out by immersing thin sheets of india-rubber in solutions of chloride of sulphur in carbon bisulphide. However thin the sheets so treated might be, their conversion into a homogeneous compound will be always doubtful, if only because it is uncontrollable; and further, in withdrawing the sheet so treated from its vulcanising solution almost instant volatilisation of the carbon bisulphide takes place, and this is always accompanied by the formation of a film of moisture or dew upon the surface from which the volatilisation takes place. The result is that the excess of sulphur chloride which is contained in the carbon bisulphide adhering to the rubber sheet, is decomposed by this moisture into free sulphur and hydrochloric acid. I proceeded therefore in this manner that I dissolved in 1,000 cc. of pure benzene, which had been twice distilled over sodium wire, 50 grms. of best Pará rubber which had been purified by fractionate precipitation from its solution in chloroform by means of alcohol. This solution was filtered through a fluted filter in a hot-water funnel, and to the beautifully bright solution so obtained a solution of 50 grms. of twice distilled chloride of sulphur in 200 cc. of pure benzene was added, the mixture thoroughly shaken and the flask containing it connected with a mercury barometer by means of a stopper and tube. After about five minutes standing the reaction began to set in, the mixture becoming somewhat gelatinous, but otherwise exhibiting no outward appearance of any change taking place. After 20 minutes the reaction had evidently reached its end; the contents of the flask being converted into a moderately stiff jelly, the firmness of which did not show any further increase. During this time the temperature of the mass had risen about 4° C., and the barometer registered only a barely corresponding higher pressure, which disappeared as soon as the contents of the flask had cooled

\* Loc. cit.

† Ann. Chem. 113, 270.

\* Mittheil. d. techn. Gewerbemus., 1899, 43.

† See present number of this Journal, page 47.

down to their original temperature. Not the faintest trace of the evolution of any gas appeared during the whole of this time, the contents of the flask remaining clear and absolutely undisturbed from beginning to end. This experiment was several times repeated with the same result. Already now it can no longer be doubted that Fawcitt's observation of an evolution of hydrogen in this reaction must be erroneous, as well as the statements of Burghardt, Terry, and Thomson respecting evolution of hydrochloric acid. Evolution of hydrogen in this reaction is in itself almost a chemical impossibility, but hydrochloric acid gas is indeed given off, if not every trace of water has been removed from the india-rubber and the benzene from which the solution is prepared. The evolution of hydrochloric acid in this case is simply caused by the decomposition of the chloride of sulphur by the water, but has no connection whatever with the reaction which takes place between the chloride of sulphur and the india-rubber.

The purification of the product of the above reaction was carried out in the following manner:—The gelatinous contents of the flask were found to be absolutely insoluble in all available solvents, *i.e.*, solvents which will not cause the decomposition of the excess of chloride of sulphur which is present. The whole mass was therefore placed in a bag of fine calico, this was immersed in pure benzol, and the contents of the bag then squeezed through the meshes of the fabric. In this manner the jelly was easily converted into a pulp, consisting of very fine flakes of the product of the above reaction. The excess of chloride of sulphur present was dissolved in the benzene. It is scarcely necessary to remark that the benzene used for this last operation must also be absolutely free from water to prevent decomposition of the chloride of sulphur. The whole is then filtered and the filter residue washed with warm benzene until the filtrate is free from every trace of chloride of sulphur. The filter residue was then quickly pressed between filter paper, transferred to a large Soxhlet apparatus, and during 12 hours extracted with pure carbon bisulphide. The extracted product, still saturated as it was with carbon disulphide, was then quickly transferred into a flask containing absolute alcohol, the flask connected with a condenser and so much alcohol distilled off until the distillate no longer gave the phenyl hydrazine reaction for carbon bisulphide. The contents of the flask were then filtered, the filter residue pressed between filter paper and dried *in vacuo*. A mass of fine yellowish white and very friable flakes was thus obtained. If before drying this substance every trace of benzene or carbon bisulphide has not been removed the substance dries in the form of horny granules of enormous toughness, which resist all attempts to reduce them to a powder.

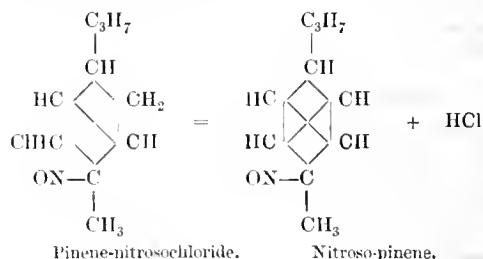
The substance so obtained is perfectly insoluble in all solvents, although in its behaviour against them it exhibits marked differences. Methyl alcohol, ethyl alcohol, glycerol, acetone, ethyl ether, ethyl acetate, amyl acetate, glacial acetic acid, and acetic anhydride have no effect whatever upon it. The coal-tar hydrocarbons, chloroform, carbon tetrachloride, carbon bisulphide, all the terpenes, eymene, and the etheral oils cause it to become transparent and swell it more or less considerably. But neither in the cold, nor by gently heating the solvents, are the faintest signs of solution observable. On prolonged heating to temperatures above 140° C. more or less complete solution takes place with mesitylene, eumene, eymene, the heavy petroleum hydrocarbons, and all terpenes.\* But there is little doubt that these solutions do not contain the original substance, but merely products of its decomposition at these high temperatures. This conclusion is justified by the fact that on heating the substance in question in the drying oven to a temperature of about 80° C., decomposition sets in with evolution of large quantities of hydrochloric acid. Moreover, the original substance cannot be recovered from the above-mentioned solutions.

The analysis of this substance gave the following figures:—

	I.	II.	Calculated for $C_{10}H_{16}S_2Cl_2$
C.....	43.61	43.51	44.28
H.....	6.21	6.14	5.90
S.....	23.88	23.81	23.62
Cl.....	25.97	26.11	26.19

The most remarkable result of these analyses is the fact that this substance contains S and Cl in the identical proportion in which they occur in chloride of sulphur, just as we found them in the chlorosulphides of ethylene and amylene, and also in the products obtained by the action of chloride of sulphur upon the triglycerides of the unsaturated fatty acids (india-rubber substitutes).

From these results we arrive at the conclusion that india-rubber vulcanised with chloride of sulphur is an addition product of these two compounds, polyprrene-sulphochloride. As already mentioned, the chlorine is very easily removed from this compound and escapes in the form of hydrochloric acid at a temperature of between 80° and 120° C. This removal of the hydrochloric acid may also be effected by boiling the sulphochloride with alcoholic potash. This is a reaction of the same kind as the one Henriques\* observed in treating india-rubber substitutes with alcoholic potash, in which case he obtained unsaturated fatty acids which still contained sulphur, but no chlorine. Of the same order is evidently the elimination of hydrochloric acid from the nitroschlorides of the terpenes:—



The analysis of polyprrene sulphochloride boiled with alcoholic potash for several hours, gave the following figures:—

	I.	II.	Calculated for $C_{10}H_{16}S_2$
C.....	59.88	59.93	60.60
H.....	7.21	7.47	7.07
S.....	32.11	32.04	32.22

The product thus obtained is, therefore, evidently polyprrene sulphide, all the chlorine of the sulphochloride being eliminated. According to Thomson\* and Terry,\* who assign the vulcanising action of chloride of sulphur to its chlorine, we should expect now this polyprrene sulphide to have lost all the characteristic properties of vulcanised india-rubber and to differ very widely in its properties from polyprrene sulphochloride. But as a matter of fact this elimination of the chlorine alters the properties of our vulcanisation product so little, that without the analytical data we could not form any opinion as to whether alcoholic potash has any action at all upon the sulphochloride. In all its physical and chemical properties polyprrene sulphide cannot be distinguished from perfectly vulcanised india-rubber, especially its absolute insolubility and chemical indifference have remained the same as we found them in the sulphochloride. While it is thus shown that the removal

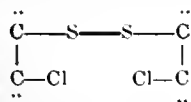
\* See also Henriques, this Journal, 1893, 468.

\* Loc. cit.

of the chlorine from polyprene sulphochloride can be effected with the greatest ease and without causing any structural alteration of this compound, any attempt to remove the sulphur is either unsuccessful, or results in a total destruction of the substance. These observations leave no room for any doubt as to whether the vulcanising action of chloride of sulphur is due to the sulphur or the chlorine of this compound, especially if we find that the state of vulcanisation remains the same with or without the chlorine. Therefore, although the chlorine and the sulphur of chloride of sulphur act simultaneously upon the india-rubber, it is the sulphur to which the vulcanising must be attributed. It is somewhat strange that Thomson, Terry, and Hurgbardt pronounced the vulcanising action of chlorine without supporting it by further experimental evidence, at least by a closer investigation of the halogen derivatives of india-rubber. They would then have found that although these derivatives in outward appearance very closely resemble vulcanised india-rubber, they are clearly distinguished from it by their great and complete solubility in benzene or chloroform. But these halogen derivatives do not even possess the chemical stability of vulcanised india-rubber and readily lose all their halogen under a variety of conditions. These halogen derivatives will be specially treated in the second part of this paper.

The above quoted analyses lead to the formulae  $C_{10}H_{16}S_2Cl_2$  and  $C_{10}H_{14}S_2$  for polyprene sulphochloride and polyprene sulphide respectively. Whether these formulae indeed represent the respective molecular weights of these compounds is a most important question, but all the means usually employed for the determination of this factor are quite unavailable in this case owing to the impossibility to vaporise these compounds without decomposition or to dissolve in some indifferent solvent. On the other hand, however, it can be shown from purely chemical reasons that those formulae do not even approximately represent the molecular weights of polyprene sulpho-chloride and polyprene sulphide, as if they did the molecular weight of india-rubber itself would appear to be  $C_{10}H_{16}$ , which certainly is not the case, as we shall see presently.

I have been referring above to the addition products obtained from ethylene, or amylene, and chloride of sulphur, and I showed that these products are formed by the coupling together of two molecules of the hydrocarbon by the sulphur of the chloride of sulphur, thus—



Consequently in acting upon terpenes with chloride of sulphur compounds must be obtained, the molecular weight of which cannot be lower than  $C_{20}H_{32}$ . Lately Henriques,\* in his excellent studies on the analysis of india-rubber articles, showed that of two sheets of pure Pará rubber which were cured by immersion in solutions of chloride of sulphur in carbon bisulphide, the sheet containing 5 per cent. of sulphur was over vulcanised, consequently rather stiff and hard, while the second sheet, which contained 0.5 per cent. of sulphur, was underdone, and therefore still somewhat tacky. But our above described polyprene sulpho-chloride contains 23.8 per cent. of sulphur, and practically consists of equal parts of india-rubber and chloride of sulphur. In practical working, therefore, such a vulcanisation product is never produced, as it would be almost equal to a complete destruction of the india-rubber for practical purposes. But at the same time, we have to consider the fact that india-rubber will combine with its equal weight of chloride of sulphur, forming a vulcanisation product with over 23 per cent. of sulphur, so that the question arises as to whether india-rubber vulcanised for practical purposes and containing a possible maximum of 5 per cent. of sulphur, may represent a homogeneous compound, or must under all circumstances be regarded simply as the solution of a corresponding quantity

(about 20 per cent.) of polyprene sulphochloride  $C_{10}H_{16}S_2Cl_2$  in unaltered india-rubber.

To decide this point, 5 grms. of purest Pará rubber were dissolved in 1,000 cc. of pure benzene, and 5 grms. of chloride of sulphur were added to this solution. In consequence of the great dilution the reaction proceeded very slowly, but after three hours standing all the chloride of sulphur had disappeared. The solution was left standing overnight, and on the following morning 100 cc. of it, representing 2 grms. of the india-rubber used, were treated with absolute alcohol and the polyprene sulphochloride, which separated out purified in the manner before described. An analysis of this compound again showed 23.78 per cent. of sulphur, so that there is no doubt about the conversion of the india-rubber in the sulphochloride  $C_{10}H_{16}S_2Cl_2$ , even in such dilute solution, provided there be the required amount of chloride of sulphur. To the remaining 600 cc. of the above-mentioned solution, a solution 22.5 grms. of pure Pará rubber in 200 cc. of benzene was now added, and the mixture strongly agitated. The gelatinous character of the mixture rapidly changed into that of an ordinary india-rubber solution, and on pouring this solution into absolute alcohol a voluminous precipitate very much like polyprene sulphochloride was obtained. This precipitate was separated from the supernatant liquid by filtration, the filter residue repeatedly washed with warm alcohol, and then dried *in vacuo*. In this manner a rather elastic substance was obtained, which, on analysis, was found to contain 4.96 per cent. of sulphur, or about as much as Henrique's high vulcanised india-rubber sheet. The question is now whether this new product really possesses the properties of commercially vulcanised india-rubber. To determine this point the substance was placed in benzene. After 18 hours standing, complete solution had taken place; the solution appeared somewhat turbid. It was strongly diluted, and filtered through a hot-water funnel. The filtrate obtained was beautifully clear. On the filter remained a residue, which was repeatedly washed with warm benzene and then dried. It formed a whitish, easily friable mass, weighing 5.8 grms., and on analysis it was found to contain 23.41 per cent. of sulphur. There is no doubt that this substance represents the total quantity of polyprene sulphochloride we mixed with india-rubber, and this is further confirmed by the fact that the above-mentioned filtrate contains not the faintest trace of either chlorine or sulphur. This result is a conclusive proof that a mixture of polyprene sulphochloride and india-rubber only possesses the properties to be anticipated for such a mixture, but not those of vulcanised india-rubber. This being so, we must at once come to the conclusion that india-rubber vulcanised with chloride of sulphur and containing a maximum of 5 per cent. of combined sulphur, must be a homogeneous compound in the sense that every molecule of india-rubber present is combined with sulphur and chlorine, and that there is no uncombined india-rubber. But this, of course, is only possible if the molecule of india-rubber is very much larger than  $C_{10}H_{16}$ .

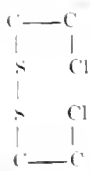
Before proceeding any further it was important to prove that india-rubber homogeneously vulcanised with such a quantity of chloride of sulphur as to produce a product containing again 5 per cent. of combined sulphur, behaved distinctly different from the above mixture. For this purpose 25.5 grms. of purest Pará rubber were dissolved in 1000 cc. of pure benzene. To this solution 3 grms. of chloride of sulphur were added, the mixture vigorously stirred and then left to stand. Very soon the mass began to thicken, the reaction reaching its maximum after about 15 minutes. After several hours standing the jelly was thoroughly broken up by stirring, and the resulting pulp poured into absolute alcohol, the precipitate was filtered off, adhering and occluded benzene driven off in the well

\* Of course in vulcanising, say, waterproof cloth, with chloride of sulphur, it is well known that only the surface of the india-rubber coating becomes vulcanised, the lower strata receiving very little, if any, of the vulcanising agent. This has, however, nothing to do with the above question, the point being as to whether that part of the india-rubber which did get vulcanised possesses the composition,  $C_{10}H_{16}S_2Cl_2$ , or is a vulcanisation product with less sulphur. And if so, whether such a vulcanisation product is a homogeneous compound or a mixture of the above-mentioned description.

\* See page 70, this Journal, present number.



known manner, and the substance finally dried *in vacuo*. The dry substance contained 5.11 per cent. of sulphur, and is therefore in this respect identical with the mixture of polyprène sulpho-chloride and india-rubber. The elasticity of the new substance is, however, greatly superior to that of the mixture, from which it further differs essentially by its absolute insolubility in all solvents. Even an immersion of weeks at a time utterly fails even to soften it, although considerable swelling of the immersed substance takes place. This, however, is also the case with our sulpho-chloride of the formula  $C_{100}H_{180}S_2Cl_2$ . It is evident that this substance, containing only 5 per cent. of sulphur, does not contain any unaltered india-rubber, and must be considered as sulpho-chloride, distinctly different from  $C_{10}H_{16}S_2Cl_2$ , which contains nearly 24 per cent. of sulphur. The only possible explanation of this observation is that the molecule of india-rubber possesses a considerable number of ethylene bonds which react successively with chloride of sulphur in the already explained manner:—



every fresh molecule of chloride of sulphur producing such a sulphur bond between two molecules of india-rubber. With the number of such bonds in one molecule of the vulcanisation product the hardness and indifference of this product must increase. This unavoidable inference appears to me of considerable interest for the explanation of the process of manufacturing ebonite, which is altogether in the dark as yet.

A very simple calculation shows now, that a vulcanisation product containing 5 per cent. of sulphur must possess a molecular weight of about 1,300, and this almost exactly corresponds to a compound  $C_{100}H_{180}S_2Cl_2$ . Taking into account that one molecule of chloride of sulphur will copulate two molecules of india-rubber, in the manner repeatedly alluded to, the above formula would have to be dissolved into—



Accordingly the molecular weight of india-rubber has to be taken as  $C_{50}H_{90}$  or  $(C_{10}H_{18})_5$ , and accordingly our first described polyprène sulpho-chloride must possess the formula  $[C_{50}H_{90}]_2 \cdot 10S_2Cl_2$ . If we calculate according to this formula the lowest vulcanisation product (sulpho-chloride) india-rubber is capable to form, we find that it would be one containing 4.20 per cent. of sulphur. Such a product was prepared by dissolving 90.97 grms. of pure Para rubber in 4,500 cc. of absolutely dry benzene and adding to this solution 9.03 grms. of chloride of sulphur dissolved in 100 cc. of dry benzene. The reaction proceeded very rapidly, but the mass was left standing for several hours after the reaction had come to a standstill. The rubber solution was then found to be converted into an exceedingly viscous jelly, which was perfectly free from stickiness. This jelly was squeezed through very fine silk gauze into alcohol, where it separated out in the form of very small and elastic pieces of a whitish colour. The substance so obtained was freed from every trace of benzene in the manner before mentioned and then dried.

On analysis this product was found to contain 4.26 per cent. of sulphur, and 4.71 per cent. of chlorine. It possesses all the properties of well vulcanised india-rubber, especially very great elasticity and great indifference against changes of temperature. In the well-known solvents for india-rubber this vulcanisation product swells very considerably, without, however, even on very prolonged treatment, ever exhibiting the faintest sign of solubility. This leaves no doubt that it contains no free india-rubber, a fact which can only be explained by attributing

to it the formula  $C_{100}H_{180}S_2Cl_2$ , or rather  $(C_{50}H_{90})_2 \cdot S_2Cl_2$ . As before mentioned, our old formula for polyprène sulpho-chloride,  $C_{10}H_{16}S_2Cl_2$ , must accordingly, be altered to  $(C_{50}H_{90})_2 \cdot 10S_2Cl_2$ . If we now examine the formulae of these two compounds we see at once that while each contains two molecules of india-rubber of the formula  $C_{50}H_{90}$ , there is a wide difference between the amount of chloride of sulphur they contain, and it is evident that between these two polyprène sulpho-chlorides eight others must exist containing from two to nine molecules of  $S_2Cl_2$ , and from the vast difference in the physical properties of these two end products of the series we may safely conclude that every one of these ten possible vulcanisation products will have distinct properties which distinguish it from any of the other nine members of the series. It will be a matter of the greatest practical importance to define clearly the specific properties of each of these ten vulcanisation products. There is no doubt that the degree of vulcanisation of an india-rubber article must vary, and ought to be regulated according to the very varying uses these articles are put to, and certainly the manufacturers are fairly well aware of this truth, although I do not think that they have given it half the attention yet which it deserves. This is probably and in a large measure due to the fact that the *rationale* of the vulcanisation process so far has not been properly understood, although it certainly has been misunderstood. Consequently our present means and ways to carry vulcanisation always to the same stage, or to some definite stage, are in a very crude state as yet. Indeed I am justified in saying that vulcanisation so far is still on materially the same point to which the labours of Goodyear, Hancock, and Parkes advanced it, and there is ample room for improvement.

It would be beyond the scope of the present paper to enter into a closer investigation of the properties and technical applicability of the ten vulcanisation products above named. This may in itself be the subject of a probably rather extensive paper, as it will be necessary to devise altogether new methods for the practical vulcanisation of india-rubber articles. In the present paper I have not directly been aiming at practical results in regard to vulcanisation, but rather restricted myself to the scientific side of the question, which is the only ground upon which technical points of this kind can be satisfactorily discussed and advanced. I may, however, perhaps point out that at the present moment the vulcanisation of india-rubber articles with chloride of sulphur is of a rather limited applicability, the vulcanising through of anything like a substantial thickness of india-rubber, or even the homogeneous vulcanisation of thin rubber films, being impossible with this vulcaniser, on account of its rapid and energetic action upon india-rubber. Nevertheless, I feel justified in already now expressing the opinion, proof of which I shall furnish in the next paper, that the vulcanisation process of the future for india-rubber articles of every description, will be some process based upon the chemical theory of the chloride of sulphur vulcanisation as above propounded. No doubt there are considerable difficulties in the way of this improvement, but they are not insurmountable, and from certain observations in advance of the present paper, I infer that by such a new process the ordinary chloride of sulphur cure (cold cure), as well as the sulphur cure (dry heat and steam cure), will be altogether superseded, and I really doubt very much whether the latter will be able to hold its own even in the manufacture of ebonite. Indeed I have no hesitation in saying that the sulphur cure as in use at present is an exceedingly crude, unreliable, antiquated and unscientific process, which is being kept alive only by our ignorance of the chemistry of india-rubber.

I can hardly expect that the molecular weight for india-rubber, which I deducted in a somewhat unusual manner from the polyprène sulpho-chlorides as  $C_{50}H_{90}$  will be accepted without comment. While maintaining that on the one hand the methods and arguments I used for the purpose cannot be objected to, I am on the other hand bound to admit that there is still a possibility of the size of the india-rubber molecule being even greater than I stated it, namely, twice  $C_{50}H_{90}$ , or even more. To settle this point definitely only one of the physical methods for the estimation of the

molecular weight can be relied upon. Hibbert and Gladstone\* have already made attempts in this direction by determining the depression of the freezing points of solutions of india-rubber. The results these authors obtained were so unsatisfactory that they themselves regard them with a great deal of doubt, and I may at once say that my own results by that method are no better. There was still the possibility that the boiling point method might prove successful, but the figures obtained by its means soon dispelled this illusion. This, no doubt, in both cases is due to the colloidal state of solutions of india-rubber. Ostwald,† Luedeke,‡ and others found that solutions of colloidal substances show almost immeasurably small depressions of the boiling and freezing point. Until we are able to obtain easily soluble and non-colloidal derivatives of polyprène, the probability to confirm or correct by physical means the above propounded molecular formula of polyprène is very remote. For this reason the formula  $C_{50}H_{80}$  strictly speaking can be considered to represent a minimum only, as the real molecular weight of polyprène might be a multiple even of that formula. I expect to be shortly in position to settle this point definitely.

The results of this investigation may be shortly summed up as follows:—

1. The action of sulphur monochloride upon polyprène is identical with the action of sulphur monochloride upon the unsaturated hydrocarbons of the ethylene series; that is to say, two molecules of polyprène are linked together by one or more double atoms of sulphur. Every molecule of sulphur monochloride entering into reaction dissolves one ethylene bond in each of the two molecules of polyprène.

2. At least ten different polyprène sulpho-chlorides exist, containing from one to ten double atoms of sulphur.

3. The vulcanisation of india-rubber consists in the formation of one or more of these polyprène sulpho-chlorides. The state of vulcanisation is due to the presence of these sulphur bonds, by which a condensation or conjugation of two molecules of polyprène into one molecule is effected. The presence of the chlorine in the vulcanisation product is without influence upon the state of vulcanisation. It is merely the means, in the chloride of sulphur, which enables us to act upon polyprène with a double atom of sulphur *in statu nascendi*.

4. The practical process of vulcanising with chloride of sulphur is most imperfect, as it does not admit of a homogeneous vulcanisation of the india-rubber. A process for the homogeneous vulcanisation of india-rubber by means of a method based upon the reaction between polyprène and sulphur monochloride, would be of very great practical value, and by far preferable to any of the vulcanisation processes now in operation.

#### DISCUSSION.

The CHAIRMAN gathered from the statements of Dr. Weber that no scientific progress had been made during the past fifty years with regard to the important industry of vulcanising india-rubber. This was because neither solvents nor other agents seemed to have any great chemical action upon that substance, and there had been great difficulties in the way of anyone starting a theoretical research on the possible combinations of this substance, and he was glad that Dr. Weber had resolved the moot points as to what chemical action did take place when india-rubber was subjected to the action of chloride of sulphur. The author had shown that a combination was formed of a definite compound containing both sulphur and chlorine, and that chlorine did not play an active part in the end on the properties of the vulcanised substance. This in itself was a wonderful result, and differed from those admitted by former searchers in this field.

Mr. TERRY admitted that he had perhaps been too ready to accept the explanation as to the theory of vulcanisation advanced by Burghardt, but he had never looked on the matter as at all settled. In regard to the passage objected to by Dr. Weber, the opinions then expressed were never intended to be more than a provisional hypothesis to form

the basis of future inquiry. Such opinions, founded on analogy alone, must of course give way before the practical results of Dr. Weber, though in the light of the known action of chlorine on rubber it was not so very far fetched to attribute a somewhat similar action to chloride of sulphur. It was not easy to discuss such a paper without seeing it in print, and he should defer any further remarks until the occasion of the reading of the second part.

Dr. WEBER, in reply, quite agreed with Mr. Terry's remarks. He did not propose to do away with sulphur vulcanising and do it all with chloride of sulphur. What he said was that he could see the possibility of devising some method for the vulcanisation of india-rubber, based upon the same reaction as that taking place between chloride of sulphur and india-rubber. He quite saw a way to getting at a substance that would be capable of the same reaction without the drawbacks of chloride of sulphur. Before the chloride of sulphur had time to penetrate the articles to be vulcanised, its chemical action had already been exhausted by combination with the superficial strata. Mr. Terry would be able to bear him out, that on the vulcanising of cloth with chloride of sulphur, they would always find that the india-rubber on the back was absolutely unvulcanised. This precluded altogether the idea of homogeneous vulcanisation. The importance of chloride of sulphur lay in the fact that it allowed them to vulcanise at ordinary temperatures. With sulphur they had to use high temperatures. He had actually found that direct harm was done to the india-rubber through the excessive heat which was necessarily employed. It was quite true the temperature could be decreased very considerably by using an excess of sulphur, but this was almost worse than working at high temperatures.

The chlorine of the chloride of sulphur, which, together with the sulphur, entered into combination with the india-rubber, was utterly immaterial for the state of vulcanisation. All the characteristics of india-rubber remained unimpaired after the removal of the chlorine from the vulcanisation product, provided only they had the sulphur bond intact. Now, if india-rubber were treated with chlorine or chlorinating agents, substances were obtained which physically very closely resembled vulcanised rubber. They were, however, easily distinguished from the former by their great and complete solubility in chloroform. At temperatures very little above the boiling point of water the chlorine derivatives of india rubber lost their chlorine completely, a hydrocarbon differing materially from india-rubber remaining behind. It was therefore evident that the chlorine derivatives of india rubber, although in some respects similar to vulcanised india-rubber, were utterly deficient just in the most important properties of the latter.

## Nottingham Section.

UNIVERSITY COLLEGE, NOTTINGHAM.

Chairman: J. M. C. Paton.

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L. Archbutt.	J. O'Sullivan.
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H. Forth.	

Treasurer: J. T. Wood.

Hon. Local Secretary:

R. L. Whiteley, University College, Nottingham.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

SESSION 1893-94.

February 14th, 1894.—Mr. J. T. Wood. "Fermentation in the Leather Industry."

\* Loc. cit.

† Lehrbuch d. allgem. Chem. 1885, I. 527.

‡ Ann. Phys. Chem. [2], 1888, 35, 552.



## Dorsetshire Section.

Chairman: A. Smithells.

Vice-Chairman: T. Fairley.

### Committee:

J. E. Bedford.	C. Rawson.
F. Branson.	P. W. Richardson.
J. B. Cohen.	Jas. Sharp.
J. J. Hummel.	G. W. Slatter.
J. Lewkowitsch.	G. Ward.
W. McD. Mackey.	T. B. Wilkinson.

### Hon. Local Secretary:

H. R. Procter, Yorkshire College, Leeds.

Notices of Papers and Communications should be addressed to the Hon. Local Secretary.

### SESSION 1893-94.

Feb. 5th.—

Messrs. Liechti and Hummel. "The Mordanting of Wool with Chromium." Part III.  
Dr. C. Otto Weber. "Substantive Dyes and Substantive Dyeing."

March 5th.—Dr. C. Otto Weber. "The Tinctorial Properties of some Indian Dyestuffs." Part I.

Meeting held Monday, November 6th, 1893.

### CHAIRMAN'S ADDRESS.

BY PROFESSOR ARTHUR SMITHELLS, B.Sc.

It is the duty of my position this evening to address you on some subject of general interest to those who are engaged in chemical industry. I trust you will pardon me if I choose a subject which though very stale, is one that concerns me closely, and one on which I am really anxious to have my say. I mean chemical education so far as it concerns chemical industries. You may ask me, can there possibly be anything new to say on this subject—is it not already worn out? Perhaps there is not anything new to say, but I do feel this, that the teacher has not had his share of the talk. If there is one subject more than another in which everyone claims a right to lay down the law, it is in education, and especially technical education, and we are becoming day by day more bewildered and entangled in a network of schemes and doctrines which defy all orderly adjustment. The race seems to have been educated to the point of seeing that the human body is a complicated mechanism, the care and discipline of which had best be relegated to a professional brotherhood who have made a special study of its working. But we do not seem to have realised that if the body is complicated the mind is more so, and that the means of making it an effective instrument and of giving it the sustenance it demands in the best possible way, is quite as much a matter for skilled opinion.

The importance of chemistry in relation to industry has been recognised in a vague sort of way ever since the chemical industries became at all considerable. It has been felt that all who have been engaged in such industries might become more intelligent in them by learning something of the general principles and chief facts of chemical science. It has been proved again and again that a deeper knowledge of this science has enabled some of those who have had the capacity, and means, and opportunity of acquiring such knowledge, to effect important changes and innovations in chemical industries. As time advanced, as these industries became more numerous and of greater national importance,

it seemed desirable to set in motion some machinery for providing this desirable dissemination of chemical knowledge. This was particularly illustrated by the great Exhibition of 1851, and in 1853 the Science Division of the Science and Art Department was established in order "to extend a system of encouragement to local institutions for practical science." Accordingly, experimental schools were started in various parts of the country. These were so far a failure, that in 1859 only four such schools survived in Great Britain, and the sum total of the science students was 400. It is difficult at this distance to ascertain the cause of this poor result. It may have been that the teaching was not good, or that it was not well paid, but I am inclined to think that it may have been due mainly to sheer apathy both of the potential teachers and potential students. It was then as it is to-day, to offer education without easily visible gains is to dangle a hare before the fish.

In 1859, someone, whose name had best remain in obscurity, invented the device of payment on results. From that time chemistry classes multiplied on the face of the earth and they have gone on multiplying ever since. In the seventies there were not many districts in which chemical industries existed that could not offer to the employes the opportunity of becoming more or less acquainted with chemistry. Schools likewise—grammar schools, rose from their torpor and shook themselves, throwing out "modern sides" which frequently embraced chemistry as one of the subjects of instruction.

So much then for the general dissemination of chemical knowledge which was to lighten the lump, to give the artisan an intelligent appreciation of the principles of the science which underlay his daily work. Now for the other part of the problem—how to train the experts. The means were in existence from the first. The College of Chemistry with the giant Hofmann (would that he had stayed with us!) came into existence in 1845. Owens College, and with it almost immediately Roscoe, were established in Manchester in 1851. After a considerable interval other great towns produced their university colleges, and in the seventies there was a good supply of institutions where chemists could be trained into investigators, and where many chemists were so trained.

Here then was the apparent solution of the problem so far as chemistry was concerned. What more was wanted? I ask the industrial chemists before me—what more do you want than this, an opportunity for all to learn the general principles of the science and an opportunity for the few to become chemical investigators? You will answer this question by asking me another—"was this a reality that you have pictured?" I answer unhesitatingly that it was not—it was not a working system, and if you ask me for proof I will simply say this, that for the last ten years the land has been ringing with the cry for what in chemistry was supposed to have been obtained, namely, chemical education adapted to the needs of industry. The cry is merged in a very babel of sound about the very bogey of a name—"technical instruction"—but it is there clearly distinguishable. Let us neglect everything but chemistry—for the rest is not our business. Surely it is worth pondering, that although ten years ago we had apparently realised the primary objects of those who wished to direct chemical instruction into industrial channels, we are talked at to-day as if nothing had been done.

I will ask you to bear with me whilst I state to you my own explanation of the anomaly. Superficially and summarily it is this—that somehow it has become implanted in the public mind that in chemistry there is an intermediate realm of knowledge which is free from the abstractions and futilities of pure science, yet in its nature scientific; which has for its apparatus something between the beaker and the boiler; for its teacher something between the professor and the unpolluted practical man; for its goal pure gold.

This, I believe, is a fair rendering of the vague feeling that prevails, and its prevalence would seem to indicate that our previous efforts to apply chemistry to industry have been unavailing. Let us look, therefore, at the means that have been tried and found wanting. We have to deal with the general instruction of the many and the extended

training of the few. The means have been for the first the Science and Art Department, for the second the universities and university colleges.

I do not propose to enter into a detailed criticism of the chemical teaching promoted by the Science and Art Department. It has often formed the theme of denunciation, less frequently of faint praise; never, so far as I know, of enthusiastic commendation. I confess that I am impatient of the tolerance with which it is regarded by so many. No doubt much good teaching has been done under the auspices of South Kensington, but a good teacher will do good work under any system. The question we have to ask is whether the system naturally conduces to good teaching, and to that question I fear we must return a negative answer. Until quite recently the chemistry teacher under the Science and Art Department was bound to teach chemistry the wrong way about. Instead of beginning with everyday things and phenomena, he had to start formally with the technicalities of the subject, as in a college course. Air did not lead to oxygen, but oxygen to air; water not to hydrogen, but hydrogen to water; a candle-flame not to chemical combination, but chemical combination with formulæ, equations, and all the paraphernalia of the chemist, eventually to the candle-flame. Latterly, an immense advance has been made by the framing of an alternative first stage of instruction, which is really a chemistry of common things, and an admirable introduction to the subject. But this relief is largely illusory, for if a teacher puts his class through the course and obtains grants on the results of the examination, he has to take the pupils right on to the advanced stage before he gets more grants. If he follows the natural sequence of taking the ordinary elementary syllabus after the "alternative" course, he has to teach for nothing. The consequence is that a great many teachers who (as they have assured me again and again) would greatly prefer to begin with the rational introduction furnished by the alternative first stage, omit it altogether, in order simply that they may live. The practical work which accompanies the first stages of South Kensington chemistry consists essentially in cultivating the art of detecting simple salts—a pursuit which I believe is generally regarded by the youth of this country in the light of an agreeable indoor recreation of the "puzzle" order. It is usually done with a zest easily mistaken for intellectual zeal and very impressive to the casual observer. What good intellectual or practical purpose this "test-tubing" serves, I have never been able to discover, unless that it provides the basis for a practical examination suitable to the exigencies of the Department. Do not think I undervalue analysis as a means of training. In the training of a chemist analysis is the backbone of his education. But for familiarising the schoolboy with the leading facts and principles of every day chemistry, it is, in my judgment, worse than useless, and its imposition not justified by the fact that a more profitable kind of work would be more difficult to examine.

I shall not speak about the later stages of the chemistry of the Science and Art Department. The beginning is everything. If that is not good, the later stages can only be so by accident. In my opinion, there is a tendency to teach far too much chemistry in schools—so far as the syllabus is concerned—and I prefer infinitely to receive as students those who have been intelligently taken through the alternative elementary stage rather than those who have attained to the doubtful glory of a "second-class advanced."

Perhaps the most conspicuous blot on the South Kensington system of teaching has been the absence of anything like real inspection. There have been four permanent inspectors to control the whole of the classes in all the science subjects patronised by the Department. In order to eke out an even nominal inspection it has been necessary to enlist as auxiliaries a large force of military officers stationed in various parts of the country, and the inspection has been very largely in their hands. This inspection has been, as a rule, merely statistical, the inspectors knowing nothing of the subjects taught, and still less of the proper method of teaching them. The inspection has, in fact, been a farce, and from what I have seen I think no one

has felt this more than the officers themselves.\* The consequence has been that the only control of the teaching has lain in the May examinations, when the teacher has been paid "on results." I need not spend time in preaching against the system of payment by results. It is a doomed system, but I fear it will die a lingering death at South Kensington. There is just one more point in reference to the Science and Art Department chemistry that I wish to mention. Chemistry figures as a subject of instruction under the Whitehall "Code,"—entirely apart from South Kensington. Recent changes in the Code have made it possible to teach chemistry in a really satisfactory manner in our elementary schools. But it is also the case that in the higher standards the teacher may, if he likes, desert the Code and go under the Science and Art Department. By doing so he would make an educational sacrifice, but obtain a very greatly increased grant. Can one wonder that, under these circumstances, the teacher, as the phrase goes, "runs the pupils for South Kensington"?

I think that I have now said enough to make it clear why the teaching of elementary chemistry under the influence of the Science and Art Department has not realised the end in view. I do not think it is the least surprising that by a system taught with so many deficiencies and anomalies we have failed to bring about a general dissemination of elementary chemical knowledge—useful in the sense of education or of instruction which should be the basis of specialised study. Nor do I think it surprising that we should have failed to impress the public with the educational or purely utilitarian value of science teaching.

Having described South Kensington as it was and is, I now pass to the institutions for training experts. It would be unbecoming on my part to attempt to state how far I and my brother professors of chemistry are cut out for discharging our duties to the community. I trust we have a proper measure of sublime discontent with ourselves. We have most of us got good laboratories, competent help, and enough to keep up a tidy personal appearance. If we are not able to teach chemistry up to the high level that is desirable, it is the fault of the public who appoint us. I will at any rate go so far as to say this, that, given enough ardent and intelligent students, there are not wanting in this country places which will turn out in a reasonable time a supply of chemists trained to the pitch of investigators adequate to the wants of the industrial world. I will ask you at any rate to assume the truth of that proposition. And now comes the important commentary. There are not enough ardent and intelligent students to make into good chemists, and, if there were, the industrial world would not want them.

I will carefully examine this statement. In the first place, it might be said it is a matter of supply and demand. No doubt this is true to a considerable extent, but there is something else. If there were in existence what Professor Huxley calls "a capacity-eating machinery" which would pick out the intelligent pupils from the schools and bring them to the colleges we should turn out more and better chemists whether they were wanted or not. Such as were appropriated would at any rate be good specimens of their kind, and by their practical achievements in the field of industry do more to create right views as to the relation of science to industry than any amount of argument. But, as I have said, they are not wanted. What, then, is wanted?

For a reply to this question I might refer you to innumerable utterances by public men, but I will keep to our own Society, and reproduce a short passage from an address printed in this Journal. It has special reference to dyeing. After a criticism of existing schemes and methods, the following indications are given of the kind of instruction that is deemed better by "practical" men:—

"If, therefore, we want to train in our schools day students destined to become foremen or managers or masters of such works, we must teach them the application of science to these industries, with the appliances and apparatus

\* Since this address was delivered, I have learned with intense satisfaction that a new and highly-qualified inspectorate is to be created for the Science and Art Department.

which they will actually meet with in practice. We must have school workshops equipped with the very latest and most modern machinery of this or any other country, which must be kept on a level with the progress of discovery and invention. We must have a model bleach, dye, print, and finishing works, of course on a small scale, but yet large enough to allow the yarns in our spinning department to be made up, bleached, prepared, dyed, &c., and the cloth woven in our weaving school to be bleached, printed, and finished, and the work turned must not be schoolboy's work, but equal to the best to be found in the trade. Only thus shall we be able to put our student in a position at once to confront and solve all the difficulties and problems which he may encounter in practice, and only thus will his scientific knowledge be of real advantage to himself and the trade."

Let us now examine these proposals. We have, first of all, to make a chemist who shall have a good general knowledge of the science, and a specially good knowledge of the chapters relating to his future pursuits. He must be able to keep abreast of the progress of the science and ready to turn new discoveries to practical account. For a student of intelligence and possessing a good preliminary education, I do not think it extravagant to fix four years as the time necessary for this chemical training. At the end of it he would be a chemist pure and simple—there would have been no time for anything else except the indissociable sciences like mathematics and physics. He is now to be made an engineer. How long this would take is difficult to say. A smattering of engineering may be picked up in a short time, but to make a man a resourceful mechanical engineer is a serious undertaking, and an allowance of one or two years for the purpose does not seem extravagant. Being now a chemist and engineer, our student is to move among the model plant of a model dyeworks. Manufacturers are supposed to have furnished the teacher with their treasured devices and receipts. The institution is to keep the machinery going, and to supply cloth to be dyed and mis-dyed. Shafts are to break, vats to leak, water to foul, and everything to be right and wrong by turns, so as to be as like the reality as possible. Perhaps, by a judicious concentration of misfortunes, six months might suffice to give the student some idea of the ups and downs of a dyehouse.

This is a long and exacting programme, but I do not think it comprises all that is aimed at. A dyeworks chemist has other things to prepare for. If the school is to equip him for the difficulties of his post, there should be in it some model British workmen tied hand and foot by the rules of their union; a model foreman dyer, who, armed with his grandfather's receipts, regards your chemist as a mischievous interloper; and, last of all, there should be a model British manufacturer, ignorant of science, distraught by foreign competition, distrustful of all that is new-fangled, dimly conscious that there is something in science that can help him, but fatally impatient of experiment or investigation.

I trust that the above will not be considered a travesty of the proposals that I have quoted. It is intended to be a serious filling in of the sketch there given. It may be said that the proposals were for the training of managers and foremen, not of experts. But the earlier part of the address which I have quoted is an explicit attack on the expert as at present trained, and the latter part an indication apparently of how he should be trained. In any case I believe the scheme to be impracticable and impolitic, and I do not believe that it is in this way that we shall make science available for the purposes of industry.

What then is to be done? I would reply let us do better what we are already doing badly. Let us by all means train the whole industrial community to an appreciation of science and its relation to the phenomena of nature and the practical arts of mankind. But our aim should be to cultivate their intelligence rather than to specialise their knowledge; and we have to look mainly at imparting a sound and extended general education. For the future masters we may do more. We cannot as a rule make them experts, but we may give them a sound general knowledge of chemistry. We can teach them to look abroad and see

the meaning of the scientific laboratory of a German colour-works. They can be made to see and understand how educated German manufacturers solve the problems of industrial chemistry.

Lastly there is the training of the expert. An expert in chemical industry must principally be a chemist pure and simple. There is only one science of chemistry, and only one set of chemical principles; only one straight course which a man may travel if he is to become a chemist, and that is the unbiased and unabated study of chemistry as a science. If it is true that we speak of the chemistry of this, that, and the other industry, it is just as true that it is chemistry—the one and only chemistry—applied to this, that, or the other set of phenomena. It is, in short, the chemistry of the university. In Germany you find it also (little as this seems to be realised here) in the higher polytechnics. It means in time a bare minimum of four years continuous hard study on the basis of a sound and liberal general education.

If I am asked whether such a chemist is really a productive man, I would reply that it is precisely these men that by their labours in other countries have given such an impulse to foreign industries and such alarm to British manufacturers. It is true that we have in this country and in this Society some splendid examples of the class; they multiply, however, but slowly. It may be that the supply is not great, but the demand is less than the supply. The British manufacturer as a rule wants something more than a mere chemist, or if he does take a chemist from the university, he forms unreasonable expectations, and is soon disappointed. I ask what is it reasonable to expect of a man going to a works as chemist? Is it reasonable to expect him to know the working conditions of a large industrial concern? Is he to have all the resources of chemist, mechanic, mason, and manager? Is he to translate instantly the experiments of beakers and crucibles into boilers and furnaces? Is he to be forbidden the one sure method of progress—the method of learning from mistakes? To these questions a great part of the public opinion of to-day virtually says yes, for it is surely this that is at the bottom of all these schemes for producing chemical engineers.

Fortunately there are some business men who can be business-like, even on the question of availing themselves of scientific help. They will invest their money in a chemist's brains as they will in a new departure in their manufacturing apparatus. They do not simply turn on the steam and excrete if the machine will not work instantly to the advertised standard of efficiency. They are content at first with a small or imperfect output and a dead working loss. But soon, by a reasonable regard to functions and adaptabilities, by giving the thing fair play, it rises to its reputation and more than repays the costliness of its early days. It is precisely thus that a reasonable manufacturer treats his chemist and secures the return on money invested in the machinery of a trained chemical intelligence.

Is there then to be no specialisation in the training of technical chemists? I do not say so. What I maintain is that an unspecialised chemist is in himself a profitable servant, who, if reasonably treated in the works, will soon become a specialist.

I believe in specialisation on the basis of sound chemical education, where it can be reasonably taught. I recognise the existence of a "borderland" in chemistry. I find in the first-rate works chemist a perfect treasury of technical knowledge, much of which might be imparted to others within college walls. Let us have such teaching by all means, but on two conditions—first that it is not at the expense of those foundations of purely scientific chemistry on which alone it can properly rest; secondly, that it is first hand. Borderland knowledge, if it is to be valuable, must come direct from the man of practical experience in the industry concerned. It is one man's work to impart it, for he must be alive to every forward move of the industry and every advance in chemistry. If not, either his chemistry or his technology will be immediately out of date. If specialists can be procured; if you can make it worth their while to teach; if you can give them properly trained students, you will have something in the nature of techno-

logical teaching, which would be an invaluable addition to a purely chemical training.

But such teachers will be neither abundant nor inexpensive, and I do not think they are indispensable. Under the most favourable circumstances there will remain much that the chemist can only learn within the precincts of the works. I believe there is no royal road to chemical engineering, and it is greatly to be feared that those who attempt to find one will emerge neither chemists nor engineers, but with that smattering of both sciences which is such a deceptive and dangerous thing.

In conclusion, let me say that whilst I maintain above all else the necessity of the scientific chemist for the maintenance of chemical industries, I am not insensible to the possibility of doing something for the manager and foreman. They may have their half-loaf of chemical instruction, and it may possibly be best given at technical schools. I do not feel specially interested in the subject, for it seems of such secondary importance, and I am not aware that in their own proper sphere our managers, foremen, or workmen are inferior in aptitude to their foreign rivals. I have asked again and again what is the foreign industry that beats us in consequence of the superior chemical knowledge of the workman. I can get no answer. But no one who has looked into the matter with any desire to learn can ignore the fact that our great and pressing need is the affiliation to our chemical industries of adequately trained chemists. When I say adequately trained I do not mean technically trained. I mean rather scientifically trained, for that is where we break down; we are slow to recognise that a works chemist should be trained to the pitch of a scientific investigator. It is the want of such men that has given ground for this wearying talk about the raw coal-tar products we export and the refined products we import; and we may double the National Debt in paying South Kensington grants for chemical teaching, we may erect technical schools with the model plant in every parish in the land, but until the British manufacturers realise the true value of the scientific chemist, we shall continue to import refined coal-tar products, and much besides, and shall steadily fall behind in the race for industrial prosperity.

## Journal and Patent\* Literature.

Class.	Page
I.—General Plant, Apparatus, and Machinery .....	21
II.—Fuel, Gas, and Light .....	25
III.—Destructive Distillation, Tar Products, &c. ....	27
IV.—Colouring Matters and Dyes .....	28
V.—Textiles: Cotton, Wool, Silk, &c. ....	32
VI.—Dyeing, Calico Printing, Paper Staining, and Bleaching .....	34
VII.—Acids, Alkalis, and Salts. ....	34
VIII.—Glass, Pottery, and Enamels. ....	39
IX.—Building Materials, Clays, Mortars and Cements. .	39
X.—Metallurgy .....	40
XI.—Electro-Chemistry and Electro-Metallurgy .....	42
XII.—Fats, Oils, and Soap Manufacture. ....	45
XIII.—Pigments: Paints, Resins, Varnishes: India-Rubber, &c. ....	46
XIV.—Tanning, Leather, Glue, and Size .....	49
XV.—Manures, &c. ....	—
XVI.—Sugar, Starch, Gum, &c. ....	52
XVII.—Brewing, Wines, Spirits, &c. ....	52
XVIII.—Chemistry of Foods, Sanitary Chemistry, and Water Purification; Disinfectants .....	54
XIX.—Paper, Pasteboard, &c. ....	57
XX.—Fine Chemicals, Alkaloids, Essences, and Extracts ..	—
XXI.—Photographic Materials and Processes .....	—
XXII.—Explosives, Matches, &c. ....	61
XXIII.—Analytical Chemistry .....	61

\* Any of these specifications may be obtained by post by remitting 8d.—the price now fixed for all specifications, postage included—to Sir Henry Reader Lack, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

## I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

*New Process and Apparatus for Concentrating Sulphuric Acid.* M. M. Gerber. *Monit. Scient.* 41, 366.

See under VII., page 36.

*The F. L. Bartlett Zinc-Lead Process.* Abstract of Report, by E. W. Hawker. *Eng. and Mining J.* 56, 1594.

See under X., page 41.

*A Crucible Furnace for Burning Petroleum.* W. E. Crane. *Eng. and Mining J.*, December 23, 1893, 644.

This furnace is built round as high as the top of the crucible, and 4 or 5 in. larger in diameter. From thence upwards it is built square, and the corners used as described further on. The pot stands on a support about 6 in. high. If it is a furnace running continually, it is immaterial of what substance it is made, or how solid, provided it will withstand the high temperature. If a furnace runs to 12 hours only per day it should be made with as little substance as will withstand the temperature and weight. If made solid, the first heat will take longer, as this support must be thoroughly heated through before the bottom of the pot will heat. After the first heat any support will be all right. Fig. 1 shows an elevation of the furnace, which has been rebuilt from one using grates for coal. A brick bottom is put over the grates to preserve them, but one grate should be taken out and a hole left through the bottom, so that the metal can run through if the pot breaks. Fig. 2 shows a part plan of furnace at the top of the pot. Four pieces should be provided of the shape shown at A in one of these corners, which will rest on top of the pot and in the corner of the furnace, shutting in the products of combustion around the pot below the top. By this means fully 15 per cent. of the fuel will be saved, a much hotter fire secured, and faster work done. A small flue should lead off from the top of this chamber, not over 2½ in. square, either to the chimney or to a heater for heating the air, or to other system of economising the heat from waste gases. When the waste heat is utilised, this construction is essential in melting alloys, especially where spelter or zinc is used, as the fumes would coat over or fill up any economiser. By closing the space at the top of the pot the fumes from the alloys could be carried off through a separate flue. This flue should have from 30 in. to 40 in. area. The burner would probably be an air burner, with as low a pressure as possible. Some burners work nicely with as low as 6-oz. pressure. All of the air for combustion should be supplied by the blower. With an air burner at high temperatures there is usually a deposit of carbon. This can be prevented by introducing a small jet of steam, preferably superheated. As a general thing this will be

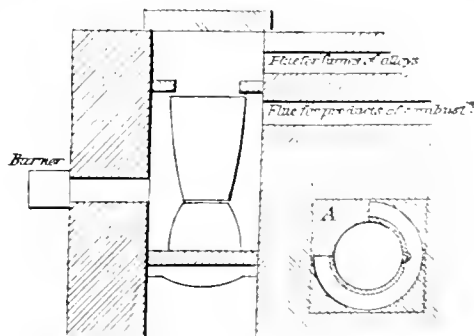


Fig. 1.

Fig. 2.

CRUCIBLE FURNACE FOR BURNING PETROLEUM.

found to cause more annoyance than that of removing the deposited carbon. The steam will have a tendency to lengthen the flame, and as a consequence the heat will be less intense, more of the heat passing off into the flame.

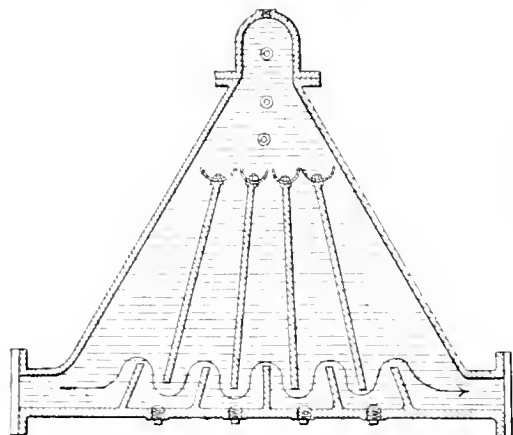
There should be a good draught to take off fumes when melting alloys, which smoke a good deal; but the draught from the products of combustion should be light. For the support for the crucible in furnaces running 10 hours per day, I have found an old pot cut off at the right length and turned bottom up to be as good as anything tried. The pieces to shut off at the top of the pot should be made of crucible stock and well seasoned.

### PATENTS.

*An Improved Oil Separator for Use with Steam Boiler Feed-water Apparatus.* J. S. McDougall, Didsbury. Eng. Pat. 21,867, November 30, 1892.

ONE shape of this apparatus, which is more particularly intended for the extraction of oil from the water passing through condensers for repeated use in boilers, is represented in the illustration.

It consists of a flat casing of pyramidal shape, provided with a number of inclined surfaces, as shown below in



IMPROVED OIL SEPARATOR.

section, which reach with their lower ends between other short inclined partitions attached to the bottom of the casing. The impure water enters near the bottom at one side of the chamber, and on passing over the several partitions in an undulatory fashion, parts with the admixed oil and leaves at the other end of the chamber in a purified state. The oil rises to the top of the chamber, where it can be withdrawn at intervals. Other impurities left on the bottom of the chamber can be taken out through special cleaning holes.—B.

*Process of Enriching Milk and Producing Cream and Butter.* M. Julien, sen., and A. Brin, London. Eng. Pat. 22,115, December 2, 1892.

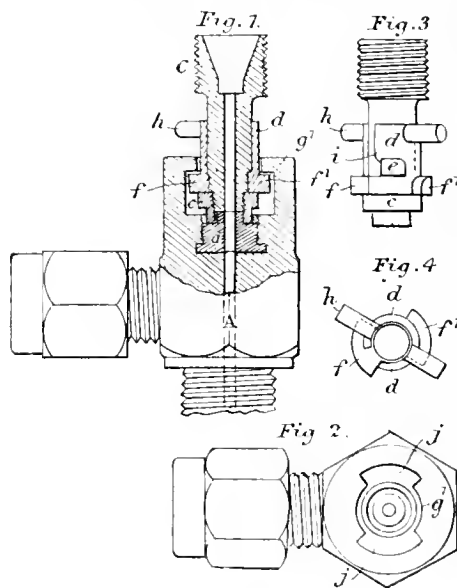
See under XV111., page 55.

*Improved Application of Aluminium and its Alloys.* W. R. Taylor, Rochester. Eng. Pat. 22,843, December 12, 1892.

See under X., page 42.

*An Improved Connection or Coupling for Use in Conjunction with Cylinders, Flasks, and Vessels containing Gases and Fluids under High Pressure.* The Manchester Oxygen (Brin's Patent) Co., Limited, and W. M. Jackson, Manchester. Eng. Pat. 23,837, December 24, 1892.

The coupling is shown in sectional elevation and plan by Figs. 1 and 2 respectively, while Figs. 3 and 4 are an elevation and plan of the detachable connection C, fitted with the locking sleeve d. This sleeve has a longitudinal slot i, so that it can be slipped over the connection C and the lug e thereon, the thimble e being afterwards screwed on. The wings f of the sleeve d are now inserted in the recessed portions j of the valve fitting A, and then turned round by the capstan heads h, whereby they assume the position shown in Fig. 1, the raised portions f<sup>1</sup> of the sleeve d bearing upon similar raised portions at g<sup>1</sup> and



GAS CYLINDER CONNECTION.

pressing the thimble e into the rubber ring a, which latter ring is inserted in a suitable undercut recess in the fitting A. With this arrangement the pressure in the bore of the valve fitting presses the rubber tightly against the thimble e, and the thimble e is pressed very tightly into the rubber by the arrangements above described; thus an extremely good joint is secured. Various modifications are described and illustrated.—E. G. C.

*Improvements in the Manufacture of Linoleum, Kamptulicon, and other similar Floorcloths, and in the Means employed in such Manufacture.* J. S. Farmer, Salford, and H. L. Storey and I. H. Storey, Lancaster. Eng. Pat. 23,983, December 29, 1892.

See under V., page 33.

*An Improvement in Apparatus for Heating, Evaporating, Condensing, and the like.* A. Normandy and F. Normandy, London. Eng. Pat. 125, January 3, 1893.

THIS is an arrangement for facilitating the cleaning of the tubes of heating coils, by fitting the coil on hinges in a casing provided with a lateral opening, through which the coil may be swung on the removal of the cover. Or the coil may be made to slide along a hinged arm, on which it may be swung through the said opening clear of the casing.—B.

*Improvements in the Method of and Apparatus for Washing Wool and other Fibres.* P. Kelly, Cross Hills, Works. Eng. Pat. 1897, January 28, 1893.

See under V., page 34.

*Improvements in Centrifugal Separators for Cream and Butter.* A. Wahlén, Rainbridge, U.S.A. Eng. Pat. 11,026, June 6, 1893.

See under XVIII. A., page 56.

*Improvements in the Production of Vacuum and Means for that Purpose.* J. Dewar, Cambridge. Eng. Pat. 439, January 9, 1893.

THE aim of this invention is to provide a substitute for the mercurial air pump hitherto used for attaining a high degree of vacuum in closed vessels. For his purpose the inventor fills the vessel to be exhausted with a condensable vapour in such a manner as to entirely exclude the atmospheric air. He then subjects a portion of the vessel, which can be hermetically sealed off from the main body, to a lower degree of temperature than the main vessel, whereby the vapours are condensed, producing a highly vacuous space in the main vessel, the degree of vacuity being in proportion to the lowering of the temperature in the subsidiary vessel. The latter is then hermetically sealed off the main vessel.

The vapours proposed to be used are either those of mercury, iodine, sulphur, or selenium in the case of elementary bodies, or else those from compound volatile inorganic or organic substances.—R.

*Improvements in Apparatus for the Making or Manufacture of Caustic Soda or Caustic Potash, being a Method of Passing the Chlorine and Sodium Gases to the Receiver.* F. W. Petrie, Rochdale. Eng. Pat. 472, January 10, 1893.

See under VII., page 38.

*Improvements in Apparatus for Removing Impurities from Water.* W. Shedlock, Stockwell. Eng. Pat. 1052, January 17, 1893.

THE invention relates particularly to water-purifying apparatus for boilers. A number of sections are arranged inside the boiler, each consisting of two tubes connected at each end by a narrow neck joined by suitable sockets to form the depositing chambers. The feed-water is admitted by a cock and passes through the apparatus, depositing salts and other impurities in the tubes, finally passing out by the orifice into the boiler. The impurities deposited in the apparatus are removed by turning round the plug of the cock, thereby arresting the entrance of the water and allowing steam to enter to blow through the apparatus, whereby the deposited matter is swept out. By giving the plug of the cock a further turn, hot water from the boiler is admitted to the chambers to obviate the formation of a vacuum in the apparatus, which might be caused by feeding in water at a low temperature after steam has been blown through them. Feed-water is now admitted by turning the cock round till it assumes the first position, when the same series of operations is performed. The cock is of special design with numerous parts; it is rotated by a worm and worm-wheel arrangement; suitable screwed connections for the different pipes are formed on the valve casting.—E. G. C.

*Improvements in and Apparatus for the Manufacture of Bleaching Powder.* E. K. Muspratt, Liverpool, A. Carey, and V. C. Driffield, Lancaster. Eng. Pat. 1214, January 19, 1893.

See under VII., page 38.

*Improvements in Apparatus or Vessels for the Continuous Evaporation or Concentration of Liquids.* E. B. Harlock. Eng. Pat. 1708, January 25, 1893.

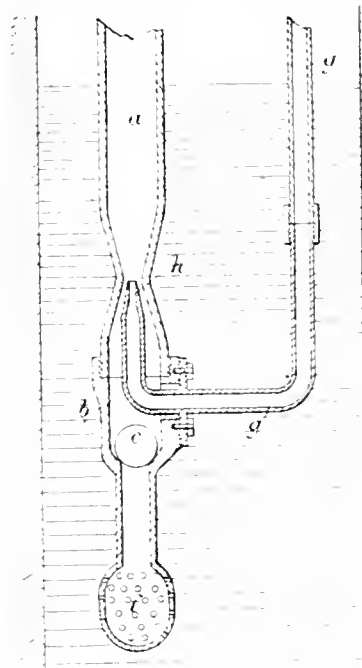
THIS invention refers to the shape of vessels employed in a series, each constructed with an internal diaphragm or open-mouthed tube along one side, which causes the incoming liquor to descend to the bottom of the vessel, whilst the outflowing liquor is drawn off near the surface.—R.

*Improvements in the Treatment of Cotton Cloths for Filtering Purposes.* H. E. Newton, London. From E. de Haen, List vor Hanover, Germany. Eng. Pat. 1770, January 26, 1893.

See under V., page 34.

*Improvements in Apparatus for Raising Liquids.* M. Macer, Enfield Highway, in the county of Middlesex. Eng. Pat. 1733, January 26, 1893.

THE apparatus is shown in sectional elevation in the accompanying figure. The rising main *a* has a strainer *f* at its lower extremity, and is also provided with a valve box *b*, containing a spherical non-return valve *c*. A jet of steam



APPARATUS FOR RAISING LIQUIDS.

is introduced into the contracted part *h* of the rising main by means of a steam nozzle *g*, thereby forcing the water contained in the tank up the pipe *a*, as in the ordinary form of injector.—E. G. C.

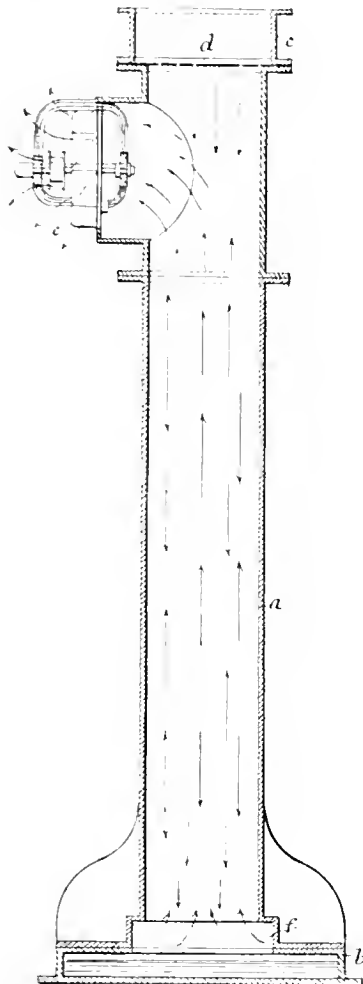
*Improvements in the Extraction of Carbonic Acid from Mineral Carbonates, and in Apparatus therefor.* A. van Berkel, Berlin. Eng. Pat. 2274, February 1, 1893.

See under VII., page 32.

*Improvements in Apparatus for Evaporating and Cooling Liquids.* E. G. Scott, Liverpool, and F. W. Scott, London. Eng. Pat. 2340, February 2, 1893.

THE invention has reference to apparatus for evaporating hot liquids by means of currents of air. The apparatus is shown in sectional elevation by the accompanying figure,

in which *a* is a long vertical column carrying at its upper extremity a cistern *c* with a perforated bottom *d*, and below



EVAPORATING APPARATUS.

resting upon a cistern *b*. The hot liquid is introduced into the cistern *d* and thence falls in fine streams down the pipe *a*, being met by a current of air which enters the column at its base *f* and is discharged at the point *e* by an exhausting fan shown at the side. A pump attached to the lower cistern serves to remove the liquid which collects in *b*. With this arrangement the coolest air traverses first the coolest liquid and *vice versa*.—E. G. C.

*Improvements in Effecting the Protection of Iron and Steel Surfaces, and in the Furnaces employed therein.* F. S. Barff, Kilburn, and G. Bower and A. S. Bower, S. Neots. Eng. Pat. 3304, July 28, 1881. (Reprint.)

See under X., page 41.

*Improvements in the Manufacture of Dough from Cereals and Apparatus therefor.* F. J. Gelinck, Riga. Eng. Pat. 15,067, August 5, 1893.

See under XVIII. A., page 56.

*Improvements in or relating to Apparatus for Drying "Slip" and Evaporating Liquids.* S. Peake, Gillow Heath, near Congleton. Eng. Pat. 15,616, August 17, 1893.

See under VIII., page 39.

*Improvements in Centrifugal Liquid Separators.* C. J. Lundström, D. H. Burrell, and E. J. Burrell, Little Falls, Herkimer, U.S. America. Eng. Pat. 15,843, August 21, 1893.

THE essential points of the patented improvement as applied to cream separators are represented in the accompanying illustrations, in which *A* is the casing, *B*

Fig. 1.

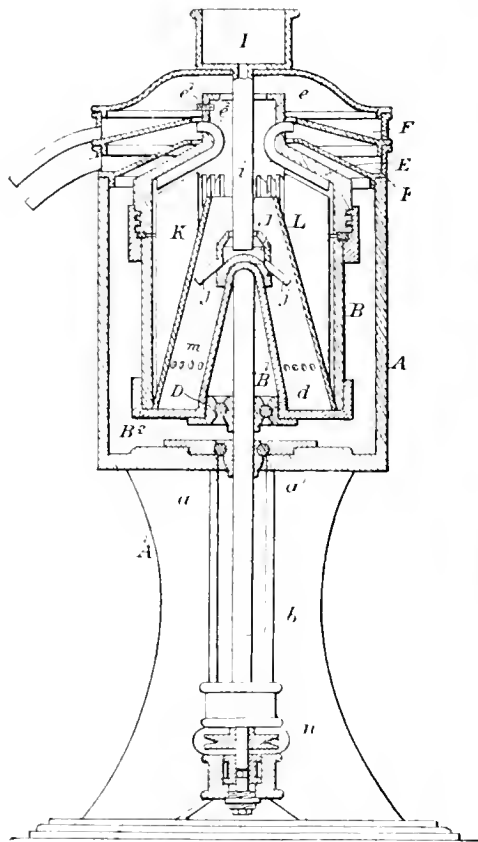
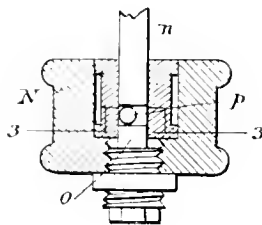


Fig. 2.



IMPROVED CENTRIFUGAL LIQUID SEPARATOR.

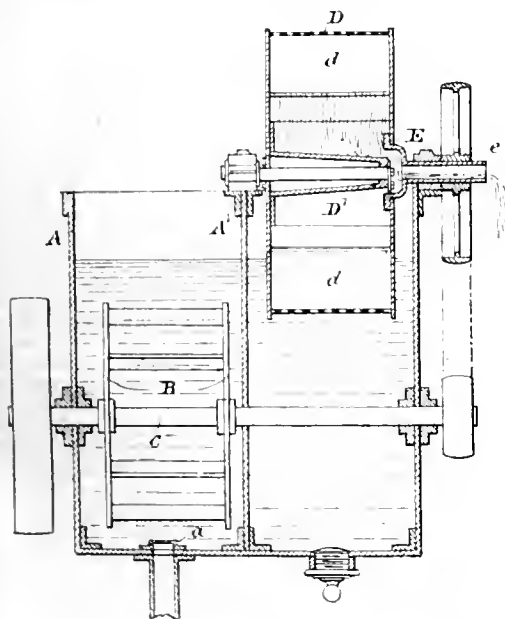
the revolving drum supported on the spindle *b*, which passes, near its upper extremity, through bushes *a* and *D*, held to their respective places by means of elastic rings *a'* and *d'* sprung into grooves. The lower end *n* of the spindle is supported on a steel ball resting on a pin screwed into a bush from below. The ball is somewhat



smaller in diameter than the space left in the bush for its reception. When at work it does not thus remain directly under the centre of the spindle, but at a distance therefrom, which causes it to be carried round and kept in constant motion. The rotary drum is fitted with a conical vessel *m*, the lower part of which is intended to retain the coarser impurities contained in the liquid, whilst the radial arms *l* tend to assist the action of the drum. The full milk enters through the centre at *i*, whilst the cream and skim milk leave the vessel separately as indicated.—B.

**Improved Apparatus for Washing Filtering Media.** W. P. Thompson, Liverpool, Manchester, and London. From W. Arnold, Gablenberg, near Stuttgart, Germany. Eng. Pat. 18,122, October 2, 1893.

THE apparatus for washing filtering media, shown in sectional elevation by the accompanying figure, consists of a vessel A divided into two compartments by a vertical partition A', which does not extend right through longitudinally. In the left-hand compartment is an agitating or stirring drum B, driven by the shaft and pulley C, and in the right-hand compartment is a lading apparatus or drum D, provided with a number of scoops *d*, and driven from the lower shaft C by a belt and pulleys. The material to be



APPARATUS FOR WASHING FILTERING MEDIA.

cleansed is placed in the vessel A, which latter is supplied with fresh water by the pipe *a*. The material is thoroughly agitated and washed by the drum B, and the dirty water is caught up by the revolving scoop *d* and emptied between the radial vanes D' of the drum D, escaping by the outlet E on the axle into the hollow trunnion shaft *e* and away. When the filter material has been thoroughly cleansed, the water may be run off and the mass of material removed.

—E. G. C.

## II.—FUEL, GAS, AND LIGHT.

*Gases enclosed in Coal and Coal-Dust.* W. McConnell. Report of Investigation carried out in the Durham Coll. of Science, 1893.

THE author, pursuing the work of Bedson, von Meyer, and others, has examined the gases given off by coal and coal-dust when heated in a vacuum. The samples used in the investigation included the Ryhope coal, which formed the subject of Bedson's inquiry. Particular attention was paid to the nature of the paraffin hydrocarbons obtained in the course of the analysis of the gases extracted from the coal, the results of their explosion, and the observation of the contraction and absorption of carbon dioxide in the usual way, sufficing to arrive at an approximate estimate of the mean number of carbon atoms per molecule, and thus to determine which members of the paraffin series were probably present.

**Ryhope Coal (a gas-coal).**—100 grms. of coal gave off 818 cc. of gas when heated in a vacuum to 100° C., the average composition of the gas being:—Carbon dioxide, 0.72; oxygen, 9.41; olefines, nil; paraffins, 16.91; nitrogen, 72.95 vols. per cent.

The combustible constituents of the first fraction drawn off appeared to contain methane and hydrogen, while those of the last fraction consisted of a mixture of methane and ethane. A further quantity of gas was obtained on powdering the extracted coal and again exhausting. It was distinguished from the former sample by the presence in it of some paraffin higher than ethane, probably propane. When the temperature of extraction was raised to 184° C., indication of the presence of yet higher hydrocarbons was obtained.

**Seaton Delaval Coal.**—This, which is a steam- as distinguished from a gas-coal, differs from the Ryhope coal in the fact that the gases obtained from it consist solely of carbon dioxide, oxygen, and nitrogen, being quite free from hydrocarbons.

**Hebburn Coal.**—This coal liberates so much gas while being worked in the colliery, that the gas is in some cases conveyed to the surface and burnt under the boilers. When examined by the author it was found to evolve 3.56 times its volume of gas, the average composition of which is given below:—Carbon dioxide, 1.5; oxygen, 1.1; paraffins, 78.0; nitrogen, 19.3 vols. per cent.

The paraffins from the coal itself consisted of a mixture of methane and ethane, while the powdered coal and the coal-dust yielded gases containing higher hydrocarbons. The combustible constituent of the gas given off naturally as "blowers," is on the other hand, mainly methane.

**Coal from another Durham Colliery.**—This colliery, which works the Hutton seam, and was some time ago the scene of a disastrous explosion, is not definitely named in the paper. 100 grms. of coal gave 160.9 cc. of gas, which had the following composition:—Carbon dioxide, 1.7; oxygen, 1.1; carbon monoxide, 0.0; olefines, 0.0; paraffins, 88.5; nitrogen, 8.7 vols. per cent.

The portion of the gas recorded above as "paraffins" consisted almost entirely of methane.

The crushed coal and the coal-dust yielded gas which was poorer in combustible constituents than that obtained from the coal itself, but contained higher hydrocarbons than methane.

From the comparison of these results the author arrives at the conclusion that the deeper seams of coal yield coals containing combustible gases and carbon dioxide, and that in passing to the younger seams the proportion of carbon dioxide increases even to the total exclusion of combustible constituents.

Certain subsidiary experiments were made to determine the amount of ethane absorbed by charcoal and coal. It was found that 100 grms. of boxwood charcoal, when saturated with ethane and placed in a vacuum at the ordinary temperature, retained 109.3 cc. of ethane, which it yielded on heating to 100° C. Similarly, 100 grms. of Hebburn coal retained 96.5 cc. of ethane.



The ease with which occluded gases are expelled from coal by diffusion was shown by experiments on Ryhope coal in an atmosphere of hydrogen. From these experiments it was evident that an exchange of gases takes place between the coal and the hydrogen atmosphere, the coal first taking up the hydrogen, which in turn expels nitrogen and a little oxygen from the coal. Then, as the heating is continued, the hydrogen is expelled, and along with it some methane, the proportion of the latter and of the carbon dioxide, increasing as the heating is continued and the pressure reduced.

Experiments on the limiting proportions of an explosive mixture of ethane and oxygen and ethane and air, led to the following conclusions:—

(1.) When mixed with oxygen, a mixture containing 3·3 per cent. of ethane will not explode even under a pressure of nearly 1,300 mm. of mercury; a mixture containing 4·6 per cent. of ethane explodes very slightly under a pressure of 250 mm.; while one of 4·5 explodes at once under a pressure of 350 mm. Mixtures containing more than 8 per cent. of ethane explode too violently to be manageable.

(2.) When mixed with air, a mixture containing 3·8 per cent. of ethane will only explode when the pressure is as high as 1,530 mm., while a 4 per cent. mixture explodes under a pressure of 596 mm.—B. B.

*Heat of Combustion of Coal-Gas, and its Relations to Lighting Power.* M. Agnition. *Comptes rend.* 117, 56—58.

WHEN the calorific power of a given sample of coal-gas (determined by means of Berthelot's bomb, modified for industrial use by Mahler) is compared with its illuminating power, a close connection is found to subsist between the two. As the mean result of experiments with more than 100 specimens of gas of varying degrees of richness, the relationship is found to be given by the rectilinear equation,  $C = 1 \times 352 \cdot 6 + 2280$ , where  $C$  is the heat of combustion, and  $I$  the lighting power in decimal candles, this formula being verified between the limits of 5 and 15 candles. The following table exhibits in more detailed fashion this relationship, the heat of combustion being that of 1 cubic metre of gas at 15° and 760 mm., measured dry, the water vapour produced by combustion being condensed, and the volume supposed constant; and the illuminating power being the light produced (measured in decimal candles) when 100 litres of gas are burnt per hour (1 carcel = 9·6 decimal candles):—

Illumination in Decimal Candles.	Photometric Titre according to the Regulation Photometer.	Heat of Combustion of 1 Cubic Metre of Gas at 15° and 760 mm.  Calories.
15	64	7,569
14·5	66·2	7,392
14	68·5	7,216
13·5	71·1	7,049
13	73·8	6,884
12·5	76·8	6,717
12	80	6,551
11·5	83·1	6,385
11	87·2	6,218
10·5	91·4	6,052
10	96	5,886
9·5	100	5,720
9·14	105	5,563
9	106·6	5,453
8·5	112·9	5,277

Illumination in Decimal Candles.	Photometric Titre according to the Regulation Photometer.	Heat of Combustion of 1 Cubic Metre of Gas at 15° and 760 mm.  Calories.
8	120	5,101
7·5	128	4,924
7	137	4,748
6·5	147·6	4,572
6	160	4,396
5·5	174·5	4,219
5	192	4,043

Since the photometric value of lighting gas varies directly with its calorific power, it would be preferable to use the latter as the means of valuation, because the former varies with the nature of the burner used, and its mode of use. Moreover, valuations on this principle of the quality of coal-gas, are directly applicable when the gas is used as a heating agent or as a source of motive power; accordingly the replacement of the photometer by the calorimetric bomb is recommended.—C. R. A. W.

*The Causes and Prevention of Spontaneous Combustion.* L. Häpke. *Chem. Zeit.* 1893, 17, 916.

THE cause of spontaneous combustion is to be found in the physical and chemical capacities of certain carbonaceous and organic matters, to condense on their surfaces gases and moisture. The amount of gases thus condensed, varies with the chemical nature of the gas, and is also dependent upon the degree of subdivision of the absorbing substances. If the atmospheric oxygen is the gas thus condensed, evolution of heat accompanies the condensation, and if this occurs within a bad conductor of heat, the latter accumulates and eventually gives rise to spontaneous combustion. In the case of fermentable matter, such as hay and tobacco, bacteria frequently initiate the process of spontaneous combustion. In all cases the molecular structure of the substances, the temperature, moisture and pressure of the air, present determinative conditions. The first record of spontaneous combustion observed on an artificially prepared substance was given by Boyle in 1680. Shortly afterwards Homburg discovered another pyrophorus consisting of potassium sulphide, carbon, and alumina. Coal fires due to spontaneous combustion were first observed at Brest (1757) and on board the "Maria" at Kroustadt (1781). In the latter case the fire was found to have originated in a mixture of linseed oil and soot. The explosion of four gunpowder factories within a short space of time in the beginning of this century was caused by spontaneous combustion. Aubert found that finely-ground charcoal placed in a barrel attained a temperature of 75° C. after 15 hours; after 20 hours ignition took place. The charcoal was found to have absorbed oxygen and also a not inconsiderable quantity of water. Liebig (1866) showed the liability to spontaneous combustion of coal containing pyrites. Abel and Percy (1876) confirmed this observation and recommended frequent observations of the temperature. Air shafts through the masses of coal on board ship, were found to be actually favourable to spontaneous ignition, and of four vessels carrying coal to India, three were thus well ventilated, but they were all lost through spontaneous combustion taking place; the fourth, having no such ventilation whatever, reached its destination safely. Experiments on the extinction of fires on board ship with liquid carbonic acid proved unsuccessful. Coals for export by water, ought to be carefully selected as to quality, and means should be adopted for preventing the coal from being moved about and crushed through the rolling of the vessel. Spontaneous combustion of coal stacks not seldom takes place, where great masses of coal are stacked. The height of a coal stack should never exceed 2·5 m. The

often asserted spontaneous combustion of wet cotton is very improbable, but a spark getting into a bale will smoulder for a long time, and the fire will then doubtless be put down to spontaneous combustion. Greasy cotton is, however, exceedingly dangerous, most fatty oils absorbing large quantities of oxygen with evolution of much heat. In cotton mills the time elapsing between Saturday afternoon and Monday morning particularly calls for watchfulness, as during this time all those materials inclining to spontaneous combustion lie uncontrolled. The few cases of spontaneous combustion of wool, flax, jute, or oakum, were also due to these materials being impregnated with oil, varnish, or certain dyes. Heavily weighted silk is also liable to spontaneous combustion, one case being recorded by Perroz, and another having occurred on board ship. Spontaneous ignition of hay stacks, although frequently disputed, is an undoubted fact, which was experimentally established by Ranko at the instigation of Liebig. The heat in the stack was found to rise to 300° C., the hay being converted into a voluminous mass of carbon. Cohn showed that fermentation caused by bacteria is responsible for such occurrences, which in Holland are prevented by throwing common salt between strata of the hay in erecting the stacks. For the same reason the stacks are built with an air shaft in the centre. Fermentation is also frequently the cause of the spontaneous combustion of tobacco. Also a case of spontaneous combustion of lupulin has been lately observed. The attempt to indicate the danger of spontaneous combustion by means of electric signals has not been successful so far. A lately invented tire indicator, which utilises the expansion of air by heat, and which can be connected with any electric or telephonic circuit, may be found very useful.—C. O. W.

*An Apparatus and Method for Determining the Density of Gases suitable for Industrial Purposes.* M. Meslans. *Comptes rend.* 117, 386.

See under XXIII., page 64.

*Manufacture of Oxygen by means of the Dissociation of Plumbate of Lime.* H. Le Chatelier. *Comptes rend.* 117, 109—110.

PLUMBATE of lime,  $\text{PbO}_2 \cdot 2\text{CaO}$ , breaks up on heating, forming lead oxide, and quicklime, and evolving oxygen, whilst the residual solid on exposure to air at a suitable temperature takes up oxygen again, reproducing the original compound. The manufacture of oxygen in this way (Kastner's process) has the advantage that the reabsorption of oxygen is more rapid than in the barium dioxide processes, whilst it is not necessary to dry the air employed, nor to remove carbon dioxide therefrom. On the other hand, a perceptibly higher temperature is requisite for the decomposition, viz., about 900° instead of 700° under a pressure of  $\frac{1}{10}$  atmosphere, which causes more rapid destruction of the iron retorts employed, as well as greater fuel consumption. The following values represent the oxygen tensions of plumbate of lime for various temperatures:—

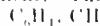
Temperature.	Pressure in Mm. of Mercury.	Temperature.	Pressure in Mm. of Mercury.
880	47	1060	557
940	112	1070	570
950	117	1100	940
1020	350	1110	1040

—C. R. A. W.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Chrysene.* E. Bamberger and F. Chattaway. *Ber.* 26, 1893, 1745—1751.

THE authors have found that chryseneic acid obtained from chrysoquinone or chrysoketone may be decarboxylated by subjecting it to distillation with lime under greatly diminished pressure and at as low a temperature as possible. The greater part is converted into chrysoketone; besides this, however, a hydrocarbon,  $\text{C}_{16}\text{H}_{12}$ , is obtained. It is isolated from the ketone by distillation with steam, and forms white, lustrous laminae, melting at 102°—102.5°. It is identical with  $\beta$ -phenyl-naphthalene obtained from phenyl-glycol and from naphthalene. Thus the formula—



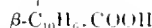
assigned to chrysene has been confirmed both by synthesis and analysis, chryseneic acid being the carboxylic acid of  $\beta$ -phenyl-naphthalene, probably  $\beta$ -phenyl- $\alpha$ -naphthoic acid—



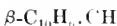
It is also shown that the product obtained by Graebe and Schmidt from chrysoquinone by distillation with soda lime is identical with the  $\beta$ -phenyl-naphthalene obtained from chryseneic acid.—D. B.

*Picene.* E. Bamberger and F. Chattaway. *Ber.* 26, 1893, 1751—1754.

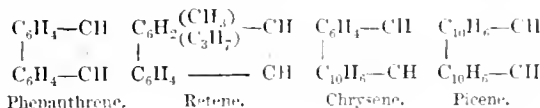
PICENE, which is the only well-defined, high molecular coal-tar hydrocarbon, melts at 350° (uncorr.) and at 364° (corr.), having a higher melting point than any other known hydrocarbon. It yields a quinone,  $\text{C}_{20}\text{H}_{10}(\text{CO})_2$ , and possesses all the properties characteristic of an ortho-diketone. It shows the carbidine, azine, and Bamberger reactions, dissolves in sodium bisulphite, and yields picene ketone when distilled with lead oxide. The latter corresponds with diphenylene ketone, retene ketone, and chrysoketone, has the formula  $\text{C}_{20}\text{H}_{14}\text{CO}$ , and on reduction gives rise to the formation of picene fluorene alcohol,  $\text{CH}(\text{C}_{10}\text{H}_7)_2 \cdot \text{OH}$ , and picene fluorene,  $\text{CH}_2(\text{C}_{10}\text{H}_7)_2$ . When fused with potash, a monobasic acid,  $\text{C}_{20}\text{H}_{15} \cdot \text{COOH}$  is obtained, which closely resembles chryseneic acid. As this substance on heating with lime yields carbonic anhydride and a hydrocarbon  $\text{C}_{20}\text{H}_{14}$ , which is identical with  $\beta$ - $\beta$ -dinaphthyl, picenic acid must be  $\beta$ -dinaphthyl-naphthoic acid—



and picene itself  $\beta$ - $\beta$ -dinaphthylethylene—



Picene is therefore the phenanthrene of the naphthalene series, being related to phenanthrene, retene, and chrysene, as illustrated by the formulae—



—D. B.

*The Colour of Acenaphthylene.* C. Graebe. *Ber.* 26, 1893, 2354—2355.

FROM the author's investigations it appears that acenaphthylene is a coloured hydrocarbon, all attempts to decolorise it having given negative results. Like diphenylene ethane

a red hydro-carbon (this Journal, 1893, 187), acenaphthylene yields a colourless bromide, and on dissolving the latter in toluen and heating with sodium, the colourless solution rapidly changes to yellow. As both acenaphthene and acenaphthylene bromide are colourless, the author concludes that for the coloration of acenaphthylene the group  $>C=C<$  is essential, in addition to which the groups replacing hydrogen in ethylene are of influence, seeing that both diphenylethane and tetraphenylethane are colourless.

Armstrong has extended his "quinonoid" theory (Proc. Chem. Soc. 1892, 192) to bidiphenylene ethane; the author, however, considers that his formula—



is more probable than that advocated by Armstrong.—D. B.

#### IV.—COLOURING MATTERS AND DYES.

*Saffron and its Adulterants.* E. Vinassa. Archiv. der Pharm. 1892. 231, 353; Dingl. Polyt. J. 290, 47.

See under XXIII., page 71.

*Azo Derivatives of Hydroquinone.* O. N. Witt and E. S. Johnson. Ber. 1893, 26, 1908—1912.

On mixing solutions of a diazo-compound and hydroquinone, reaction occurs but no azo-colouring matter is deposited owing to the great reducing power of hydroquinone; the latter thus behaves similarly to pyrocatechol in this respect. Azo-compounds are, however, readily obtained by combining the diazo-compound with benzoylhydroquinone and subsequently eliminating the benzoyl-group. *Benzoylhydroquinone*,  $\text{C}_6\text{H}_4(\text{OH})\text{OCOC}_6\text{H}_5$ , is prepared by stirring benzoyl chloride into a caustic soda solution of hydroquinone; 100 grms. of the latter give 120—130 grms. of the benzoyl derivative if a mechanical stirrer be used, otherwise a much smaller yield is obtained. The deposited product is extracted with caustic soda and crystallised from alcohol; it finally melts at 162—163°. The concentrated diazo-solution is added to the calculated quantity of benzoylhydroquinone dissolved in 20—25 parts of alcohol; 25 per cent. caustic soda is then added until the colouring matter begins to separate. The weight of the latter obtained is usually about equal to that of the benzoyl-derivative employed. The benzoyl group is removed from the azo-compound by adding concentrated potash to its alcoholic solution, diluting with water, and precipitating with hydrochloric acid. In this way the aniline, *p*-toluidine, *p*-nitraniline, tolidine, and sulphanilic acid derivatives of azohydroquinone and their benzoyl derivatives, were prepared. The benzoyl derivatives are mostly yellow, whilst the unsubstituted compounds are mostly red or blue; nearly all crystallise well.—W. J. P.

#### *Nitrohydroxy- and Nitro-amido-azo Compounds.*

E. Tauber. Ber. 1893, 26, 1872—1876.

NITROHYDROXYAZOSULPHONIC acids are, as a rule, completely converted into the corresponding nitro-amido-azosulphonic acids by heating for 16 hours at 150°—160° C. with 20—25 per cent. ammonia. The use of so costly and undesirable a material as orthonitrophenol in the manufacture of nitrohydroxyazosulphonic acids may be avoided by nitrating the corresponding hydroxyazosulphonic acid; the latter is dissolved in concentrated sulphuric acid and the calculated quantity of potassium nitrate added at 10°—20°. On pouring the product into water the nitro-derivative separates, and may be crystallised from 10 per cent. hydrochloric acid. The above processes give excellent results in a number of cases.

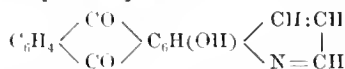
*m*-Nitro-*p*-hydroxyazobenzene para- and meta-sulphonic acids were prepared by nitrating the corresponding hydroxyazobenzenesulphonic acids and were then converted

into the amido-derivatives by heating with ammonia. *m*-Nitro-*p*-amidoazobenzenesulphonic acid yields very beautiful azo-colouring matters by diazotisation. The homologous compounds derived from the toluidinesulphonic acids are very similar to those obtained from the aniline acids.

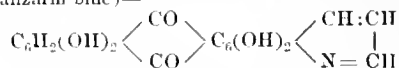
—W. J. P.

*Hydroxy-Derivatives of Anthraquinolinequinone.* C. Graebe and A. Philips. Annalen, 276, 21—35.

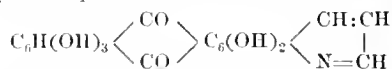
*Hydroxyanthraquinolinequinone*—



is prepared from the amide of Alizarin blue (amidohydroxyanthraquinolinequinone); this (2 grms.) is dissolved in concentrated sulphuric acid (16 cc.), water (4 cc.) is added, and the whole cooled to 0°; sodium nitrite (2.5 to 3 grms.) is gradually introduced and the product poured into boiling alcohol (200 cc.) and kept boiling for 15 minutes. The product is almost insoluble in water; it forms brick-red prisms (melting point 208°), is soluble in alcohol, forms a violet solution in alkali and a yellowish-brown solution in strong sulphuric acid. *Trihydroxyanthraquinolinequinone* was obtained from the portion of crude Alizarin green which is insoluble in nitrobenzene (compare also Schmidt and Gattermann, this Journal, 1891, 1003). It crystallises in scales having a coppery lustre, dissolves in sulphuric acid with a bluish-violet coloration, and in caustic alkalis to a blue solution. *Tetrahydroxyanthraquinolinequinone* (Dihydroxyalizarin blue)—



occurs in small quantity in commercial "Alizarin green" and in "Alizarin indigo blue," and is identical with the substance which Schmidt and Gattermann obtained by nitrating quinalizarin and conversion of the nitro product into the corresponding quinoline derivative. It forms blue solutions in alkalis and in sulphuric acid. *Pentahydroxyanthraquinolinequinone*—



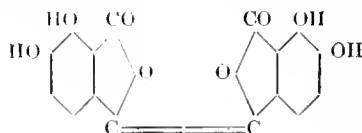
forms the constituent of "Alizarin indigo blue" which is soluble in nitrobenzene; it forms an indigo blue crystalline mass, and is soluble in alkalis and in sulphuric acid, forming blue solutions.

"Alizarin blue-green" contains a monosulphonic acid of Hydroxyalizarin blue, as stated by Schmidt and Gattermann, whilst an isomeric acid is contained in "Alizarin green." "Alizarin indigo blue" is a mixture of pentahydroxyanthraquinolinequinone with some tetrahydroxyanthraquinolinequinone and a sulphonic acid derived from the latter.

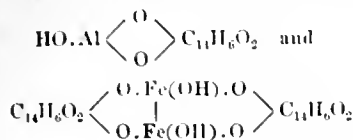
The higher hydroxy-derivatives of "Alizarin blue" yield *quinolinic acid* on oxidation with nitric acid.—A. K. M.

*The Theory of Mordant Dyes.* C. Liebermann. Ber. 1893, 26, 1574—1578.

It has been recently pointed out by the author, in conjunction with Bistrzycki, that not only are numerous derivatives of opianic acid coloured, but that some of them, such as tetramethoxydiphthalyl and bisdimethoxymetindolone, when heated with concentrated sulphuric acid, yield yellow colouring matters which unite with mordants. The view then expressed that the action of the sulphuric acid consisted in the elimination of the methyl groups is confirmed by the formation of the same products by the action of hydriodic acid. Thus the product obtained from tetramethoxydiphthalyl and hydriodic acid is tetrahydroxydiphthalyl—



previously described by Goldschmidt and Egger (Monatsh. 12, 61). This substance affords confirmation of the view advocated by the author and Kostanecki that the property of forming coloured derivatives with mordants depends upon the presence of two hydroxyl groups in the ortho-position to one another. Following up this view, he suggests that combination with the polyval metal (e.g., Al or  $\text{Fe.Fe}$ ) will only be possible when the hydroxyl groups of the colouring matter are in the ortho-position, and the formulae of alizarin red and alizarin violet may be—



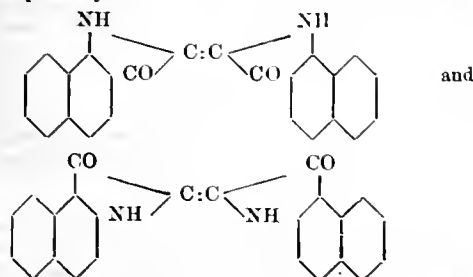
According to this view, the metal of the mordant forms a link of a pentatomic (Al) or hexatomic ( $\text{Fe.Fe}$ ) ring, and this would account for some hitherto unexplained properties of certain mordant dyes, such as their great stability and the fact that the ordinary properties of the metal are frequently masked.—A. K. M.

*$\alpha$ - and  $\beta$ -Naphthalene Indigo.* H. Wiechelhans. Ber. 1893, 26, 2547—2550.

NAPHTHALENE indigo may be obtained as follows:—Anhydrous sodium acetate (50 parts), chloroacetic acid (16 parts), and  $\alpha$ - or  $\beta$ -naphthylamine (24 parts) are fused together. At about  $120^\circ$  the mixture froths strongly, and at about  $180^\circ$  it settles down again and becomes nearly dry. Caustic potash (50 parts) is then added, the temperature is raised to about  $290^\circ$ , and the operation stopped when the vapours become greenish and when a sample introduced into hot water and agitated with air, yields a green precipitate. The product, after purification, has a composition agreeing with the formula,  $\text{C}_{24}\text{H}_{14}\text{N}_2\text{O}_2$ . Both the  $\alpha$ - and  $\beta$ -compounds are obtained as green powders which exhibit a copper lustre; they are soluble in aniline, from which they can be crystallised,  $\alpha$ -naphthalene indigo forming blackish-violet needles, and  $\beta$ -naphthalene indigo bluish crystals. The following table shows some of their properties compared with those of benzene indigo:—

	Benzene Indigo.	$\alpha$ -Naphthalene Indigo.	$\beta$ -Naphthalene Indigo.
Reduction product in solution.	Yellow	Orange	Red
Vapour.....	Red	Violet	Blue
Solution in aniline.....	Red	..	Blue-green to blue.
Solution in chloroform, benzene, glacial acetic acid, &c.	Violet to blue.	Blue	
Sulphonic acid in aqueous solution.	Blue	Blue on boiling; blue-green cold.	Blue-green
Sulphonic acid on wool and silk.	Blue	Blue to green	Green
Cotton, dyed.....	Blue	Blue to green	Green

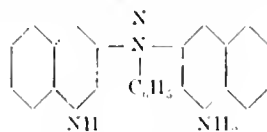
The constitutional formulae of  $\alpha$ - and  $\beta$ -naphthalene indigo are probably—



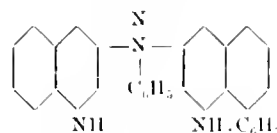
*Naphthyl Red and Magdala Red.* O. Fischer and E. Hepp. Ber. 1893, 26, 2235—2236.

THE authors have recently shown (this Journal, 1893, 671) that the Mauveines and Indazines must be regarded as symmetrical Indulines, and they have also proved Naphthyl violet and Naphthyl blue to have a like symmetrical constitution. This was shown by the formation of Naphthyl blue by an "inner" condensation of benzene-azophenyl- $\alpha$ -naphthylamine. Applying this reaction to other azo-compounds, the authors find that a new dyestuff

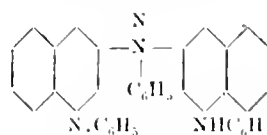
*Naphthyl red*—is formed when benzene-azo- $\alpha$ -naphthylamine is heated with phenol, whilst *Magdala red* is produced when amido-azonaphthalene is heated with phenol. Naphthyl red bears a striking resemblance to Magdala red; its relationship to Naphthyl violet, Naphthyl blue, and Magdala red is shown in the following formulae:—



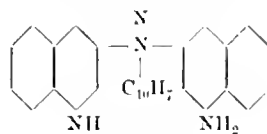
Naphthyl red.



Naphthyl violet.



Naphthyl blue.

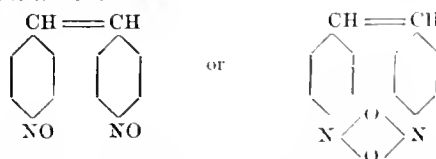


Magdala red.

—A. K. M.

*Action of Alkalis on Paranitrotoluene and its Sulphonic Acid.* O. Fischer and E. Hepp. Ber. 26, 2231—2234.

KLINGEN obtained a red amorphous substance (Ber. 16, 941) on heating *p*-nitrotoluene with alkalis, and this was further investigated by Bender and Schultz (Ber. 19, 3234); this Journal, 1887, 817), who showed it to have a composition agreeing with that of azoxystilbene. By the action of alkalis on *p*-nitrotoluene-*o*-sulphonic acid, Walter obtained the dyestuff *Sun yellow*, whilst Bender obtained *Mikado brown* and *Mikado orange*, but the constitution of these dyes is not yet established. By modifying the conditions of the experiment, the authors have obtained certain intermediate products from paranitrotoluene, by heating it with a solution of caustic soda in methylalcohol, namely, *paradinitro-dibenzyl*, *paradinitrostilbene*, and *paradinitrosostilbene*. The last substance is the main product, and the authors assign to it the formula—



Similarly from paranitrotoluene-orthosulphonic acid, the disulphonic acid of *p*-dinitrosostilbene is obtained, but is

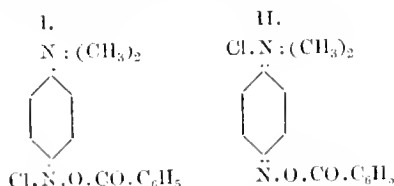
only formed when a concentrated soda solution is employed. Its sodium salt forms a dyestuff and is readily soluble in 10 parts of hot water. When a more dilute solution of soda is used, the commercial product known as *Sun yellow* is produced.—A. K. M.

*Oxidation of Chloroform by Chromic Acid, and the Preparation of Carbonyl Chloride (Phosgene) from Carbon Tetrachloride.* H. Erdmann. Ber. 26, 1990.

See under XX., page 65.

*The Action of Acid Chlorides on Nitrosodimethylaniline.* P. Ehrlich and G. Cohn. Ber. 26, 1756—1757.

Acid chlorides, such as acetyl chloride, benzoyl chloride, benzene sulphochloride, *m*-nitrobenzene sulphochloride, carbonyl chloride, and chlorocarbonic ether react very readily with nitrosodimethylaniline, nitrosotetramethyl-*m*-phenylene diamine, and nitrosodiethyl-*m*-amidophenol. The combination is best performed by mixing benzene solutions of the components. The compound from benzoyl chloride and nitrosodimethylaniline is a slightly yellowish precipitate, which, after well washing with benzene and drying in a vacuum desiccator, is tolerably stable. It dissolves easily in water, alcohol, and acetic acid, slightly in benzene and ether, and is insoluble in petroleum ether. It melts at 92° C. with decomposition, has a bitter taste, and does not give Liebermann's reaction for nitroso compounds. Zinc dust, even in the cold, gives dimethyl-*p*-phenylene diamine, and picric acid or mercury, platinum or gold chloride, all precipitate yellowish double salts. According to analysis the substance has the composition  $C_{15}H_{15}N_3O_2Cl$ , from which it would appear to be an addition product of equal molecules of nitrosodimethylaniline and benzoyl chloride. Its properties, however, do not accord with this view, as it reacts much more energetically than nitrosodimethylaniline or even chloroquinone-imide with amines and phenols. The formation of Toluylene blue takes place instantaneously by mixing alcoholic solutions of toluylene diamine and of the benzoyl compound and other amines, such as dimethylaniline,  $\alpha$ - and  $\beta$ -naphthylamine and tetrahydro- $\alpha$ -naphthylamine, or phenols, such as phenol, resorcinol, pyrogallol, phloroglucinol, dimethyl-*m*-amidophenol,  $\alpha$ - and  $\beta$ -naphthol and *o*-hydroxyquinoline react equally readily, forming the corresponding indanines and indophenols. The constitution is represented by one of the following formulae, of which II. appears to be the more probable:—



Referring to the great readiness with which the substance reacts, the authors call attention to a compound first obtained by Krause (Ber. 12, 47) by treating dichloroquinone di-imide with dry hydrochloric acid. They have obtained it as a white powder having the formula—



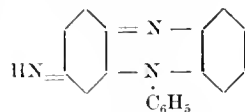
and state that it reacts much more readily than the mother substance with amines and phenols.—T. A. L.

*Relation between the Safranines and Indulines.*

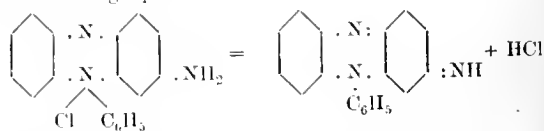
O. Fischer and E. Hepp. Ber. 1893, 26, 1655—1657.

In their previous paper (Ber. 26, 1194; this Journal, 1892, 671) the authors showed that the mauveines are to be regarded as indulines, whereas they were hitherto classed

as safranines, and as the mauveines may be considered to be formed by a phenylation of the safranines, the action of aniline on the simplest safranine was investigated. It is well known that safranine salts when heated with aniline yield colouring matters of the induline type, but up to the present no product has been obtained identical with any induline of the amido-azobenzene melt. The authors have succeeded in obtaining and identifying a known induline by this process. Starting with the simplest safranine (Nietzki and Otto, Ber. 21, 1590; this Journal, 1888, 497), which was obtained by removing the amido group from phenosafranine by diazotisation in alcohol in presence of hydrochloric acid, a well-crystallised hydrochloride was obtained of the formula  $C_{18}H_{14}N_3Cl$ , which the authors call *aposafranine*. When this salt is heated with 2½ times its weight of aniline for about three hours on the water-bath, a test dissolved in concentrated sulphuric acid no longer gives a greenish-brown colour, but a reddish-violet. The melt is extracted with benzene, which removes a small quantity of phenylinduline melting at 231° C. The residue consists principally of the hydrochloride of an induline,  $C_{18}H_{13}N_3$ , which can be purified by repeated crystallisation from alcohol containing some hydrochloric acid. The salt forms shining greenish prisms, which dissolve in concentrated or dilute mineral acids with a reddish-violet, and in acetic acid or in water with a yellowish shade. When heated with dilute sulphuric acid under pressure, the substance yields benzene-indone hydrate, and its constitution is expressed by the following formula:—



This shows it to be the simplest induline. When heated with aniline to 140°—150° C. it is converted into phenylinduline, and as this latter compound has also been obtained from azophenine and from the amidophenylinduline (Annalen, 262, 247 and 256; this Journal, 1891, 457) of the amido-azobenzene melt, it is thus shown that the safranines are converted into indulines by treatment with aniline. This is in accordance with the observation that has been made in the amido-azobenzene melt, that safranines are always formed at the commencement of the operation and disappear in the later stages. Assuming that the safranines are ammonium bases, the conversion of aposafranine into benzene-induline takes place according to the following equation:—



The elimination of hydrochloric acid from the safranine molecule is simultaneously accompanied by a change from the ortho to the para quinonoid form.—T. A. L.

PATENTS.

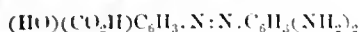
*Improvements in the Recovery of Iron from the Ferruginous Residue left in the Production of Aniline Bases.* S. S. Bromhead. From Lundschoff and Meyers, Grönan, Germany. Eng. Pat. 23,639, December 22, 1892.

See under X., page 42.

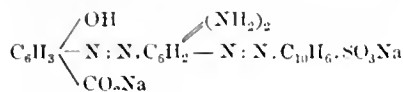
*The Manufacture or Production of New Colouring Matters.* H. E. Newton, London. From the "Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 558, January 10, 1893.

In Ger. Pat. 22,714 a process is described for obtaining colouring matters by combining diazo-compounds with chrysoidines, and the present invention is an extension of the method there mentioned. The patentees have discovered

that valuable colouring matters, as regards fastness to light and fulling, are produced by combining the diazo-compounds of aromatic amidohydroxy-carboxylic acids with *m*-phenylene or *m*-tolylene diamine and acting on the resulting chrysoidine compounds with a diazotised amine or amidazo-compound, &c. The following amidohydroxy-carboxylic acids are suitable for this invention:—*o*-amidosalicylic acid ( $\text{C}_6\text{H}_3(\text{OH})\text{NH}_2 = 1.2.6$ ), *p*-amidosalicylic acid ( $\text{C}_6\text{H}_3(\text{OH})\text{NH}_2 = 1.2.4$ ), amido-*p*-hydroxybenzoic acid (obtained from *p*-hydroxybenzoic acid by nitration and reduction), amido-*o*-hydroxy-*m*-toluic acid, amido-*o*-hydroxy-*p*-toluic acid, amido-*m*-hydroxy-*o*-toluic acid, or the sulphonic derivatives of these acids. The following is an instance of several examples given in the specification. The diazo-compound from *p*-amidosalicylic acid, corresponding to 10 kilos. of sodium nitrite, is added to a solution containing the corresponding quantity of *m*-phenylene diamine. After standing some hours or adding sodium acetate and heating, the intermediate compound separates out as a granular mass having the formula—



It is dissolved in water containing 32 kilos. of sodium carbonate, and an amount of diazotised naphthionie acid, corresponding to 10 kilos. of sodium nitrite together with a little water, is slowly added. The formation of the dyestuff is completed by heating, and after salting out and drying, it forms a black powder easily soluble in hot water. It has the following formula:—



A large number of possible combinations are mentioned and the colours they produce on un mordanted wool. These are chiefly various shades of brown.—T. A. L.

#### *Production of Orange Colouring Matters or Dyes.* T. R. Shillito, London. From J. R. Geigy and Co., Basle, Switzerland. Eng. Pat. 788, January 13, 1893.

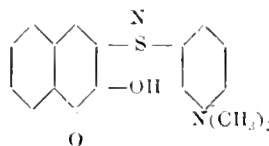
These colouring matters are produced by heating together benzidine or its homologues and derivatives with *p*-nitrotoluene sulphonic acid in presence of caustic alkali. The following substances may be employed:—Benzidine, diamidophenyltolyl, tolidine, benzidine sulphone, diamidophenyltolyl sulphone, tolidine sulphone, diamidodiphenylene oxide, diamidodiphenylene oxide, diamidocarbazol, diamidostilbene, the ethers of diamidodiphenol, hydroxy-diamidodiphenyl, hydroxydiamidophenyltolyl, and the mono- and disulphonic acids of these compounds. A colouring matter which dyes un mordanted cotton orange is obtained by dissolving 14 kilos. of sodium benzidine sulphone disulphonate and 17 kilos. of *p*-nitrotoluene sulphonic acid in 100 litres of boiling water, and adding 15 kilos. of caustic soda lye of 40° B. The whole is then boiled for about half an hour, and the dyestuff is extracted by boiling with water, and is salted out from this solution. It dissolves readily in water with an orange colour.

—T. A. L.

#### *The Production or Manufacture of Derivatives of Betanaphthohydroquinone or Betanaphthoquinone, and of Colouring Matters therefrom.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 825, January 13, 1893.

By acting with an aqueous solution of sulphurous acid on  $\beta$ -naphthoquinone it is converted into 1,2-dihydroxy-naphthalene ( $\beta$ -naphthohydroquinone). If instead of using sulphurous acid, soluble acid sulphites such as sodium bisulphite be employed, the product of the reaction is 1,2,4-dihydroxy-naphthalene sulphonic acid. The following method is given for the production of this acid:—10 kilos. of  $\beta$ -naphthoquinone "in the form of a paste

containing 30 per cent. of pure quinone" are gradually added to 30 kilos. of a sodium bisulphite solution of sp. gr. 1.1. When the quinone is completely dissolved, the mixture is allowed to stand for several hours, and after filtration is mixed with a concentrated solution of 7.5 kilos. of potassium chloride. The potassium salt separates out, and can be purified by recrystallisation from a solution of sodium bisulphite. The acid so formed can be employed as a photographic developer, and is said to be particularly useful for instantaneous photography. By means of suitable oxidising agents, such as nitrous acid, it can be converted into the corresponding  $\beta$ -naphthoquinone sulphonic acid. By substituting sodium thiosulphate for the bisulphite, a well characterised derivative of 1,2-dihydroxy-naphthalene containing sulphur is produced as follows:—1.6 kilos. of  $\beta$ -naphthoquinone are mixed with 10 litres of water, a solution of 2.5 kilos. of sodium thiosulphate in 10 litres of water is then added, and subsequently 3 kilos. of 20 per cent. acetic acid. After filtration, the addition of potassium chloride precipitates the potassium salt of an acid having the formula  $\text{C}_{10}\text{H}_6(\text{OH})_2(\text{S} \cdot \text{SO}_3\text{K})$ . When the aqueous solution is treated with alkalis it turns reddish-violet. On reduction with zinc dust in presence of acids it forms the mercaptan,  $\text{C}_{10}\text{H}_6(\text{OH})_2\text{SH}$ , which crystallises from hot water in yellowish needles. The sulphonic acid group in the 1,2,4-dihydroxy-naphthalene sulphonic acid is easily displaced, and by reacting on it with nitrosodimethylaniline in presence of thiosulphates, a product having the following formula results:—



The quantities given for the preparation of a dyestuff of this class are:—27.5 kilos. of potassium (1.2.4) dihydroxy-naphthalene sulphate are dissolved together with 50 kilos. of sodium thiosulphate in 1,500 litres of water. A concentrated solution of 32 kilos. of nitro-*o*-methylbenzylaniline sodium sulphate acidified with 36 kilos. of a 33 per cent. acetic acid is then added and the mixture is boiled until there is no longer any increase in the separation of the dyestuff. This is then filtered off and dried. It gives pure and bright blue shades on wool mordanted with chromium salts. The shades are fast to fulling and light, and are said to be even superior to alizarin dyestuffs. The colouring matters can also be used for printing on cotton or wool.—T. A. L.

#### *Manufacture of Blue Colouring Matters.* O. Imray, London. From "The Society of Chemical Industry in Basle," Basle, Switzerland. Eng. Pat. 957, January 16, 1893.

THESE dyestuffs are triphenylmethane derivatives and are obtained by condensing mono-methyl- and mono-ethyl-*o*-toluidine with dichlorobenzaldehyde and oxidising the leuco bases so obtained. About 60 kilos. of methyl-*o*-toluidine, 44 kilos. of dichlorobenzaldehyde, 60 kilos. of alcohol, and 25 kilos. of sulphuric acid are heated for 24 hours on the water-bath in an enamelled vessel. The mixture is then saturated with soda lye, distilled with steam, and the leuco base remaining behind is washed and dried. In order to convert it into a colouring matter 30 kilos. are dissolved in 400 kilos. of hydrochloric acid and 1,500 litres of water and oxidised by means of lead peroxide paste. The colouring matter is then precipitated as zinc chloride double salt, and can be purified by re-dissolving in water. The shades produced on wool and silk, or on cotton mordanted with tannin and tartar emetic, are greenish-blue of great intensity.—T. A. L.

*Manufacture of Monosulphonated Anthracene Acid by the Direct Sulphonation of Anthracene.* J. Murray, London. From "La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis" and Dr. Chapuis, Paris, France. Eng. Pat. 1280, January 20, 1893.

THE only method hitherto known for the production of a monosulphonic acid of anthracene was by reducing the sodium salt of anthraquinone sulphonie acid. By sulphonating anthracene with sulphuric acid of 66° B. at 100° C. only disulphonic acids are obtained, whereas by using an acid of 53° or 54° B. up to 60 per cent. of the anthracene employed is converted into monosulphonic acid. At the same time a small quantity of anthracene is unattacked, and the  $\alpha$ -,  $\beta$ -, and  $\gamma$ -disulphonic acids are also produced. The method of preparation is as follows:—100 kilos. of pure anthracene and 200 kilos. of sulphuric acid of 53° B. are heated for several hours to 120°—135° C. until the anthracene has disappeared. The melt is then poured into 1,000 litres of boiling water, neutralised with sodium carbonate, and left to crystallise. The sodium salt is very sparingly soluble in cold water and separates out almost completely. The properties of the salts of the acid thus produced are identical with those of the acid obtained by reducing anthraquinone sodium sulphonate. By oxidising the acid with chromic or nitric acid it is converted into anthraquinone monosulphonic acid, which, on fusion with soda, gives an almost quantitative yield of alizarin. The mother-liquor obtained as above, after separating the monosulphonic acid, contains disulphonic acids. Certain of these, when heated under pressure with hydrochloric acid, are hydrolysed, yielding anthracene monosulphonic acid or anthracene, whilst the  $\alpha$ - and  $\beta$ -anthracene disulphonic acids are not acted on. A small quantity of  $\gamma$ -disulphonic acid is also produced. These acids are converted into their barium salts, and the salt of the  $\beta$ -acid can be extracted by means of hot water. This acid, which hitherto has not been described, gives a barium salt having the composition  $C_{14}H_8(SO_3)_2Ba \cdot 4H_2O$ , soluble in water at 100° C. A litre of cold water dissolves 9 grms., and when dried in a desiccator it loses  $2H_2O$ . When heated to 110°—120° C. it loses  $4H_2O$ , but absorbs  $2H_2O$  on exposure to the air. The sodium, calcium, and magnesium salts are easily soluble, the lead salt is sparingly so. The acid, when oxidised by nitric or chromic acid, gives anthraquinone- $\beta$ -disulphonic acid, which, on fusion, yields pure anthrapurpurin, giving a tri-acetyl derivative melting at 222° C.—T. A. L.

*The Manufacture or Production of Azo Colouring Matters.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 1851, January 27, 1893.

COLORING matters which produce dark or black shades on unmordanted wool are chiefly obtained by combining the sulphonic acids of a diazotised amido-azo compound with naphthols, dihydroxynaphthalenes or their sulphonic acids, *m*-phenylene diamine, its alkyl or phenyl derivatives,  $\alpha$ - or  $\beta$ -naphthylamine or with their alkyl derivatives. In place of these it is proposed to use, according to the present specification, the  $\alpha$ -naphthylamine sulphonic acid ( $1.8$ )  $C_{10}H_6 \cdot NH_2 \cdot SO_3H$ . The colouring matters are said to produce deep shades on unmordanted wool from a neutral or weak acetic acid bath, and, in the case of those produced from amido-azo monosulphonic acids, to absolutely withstand fulling.—T. A. L.

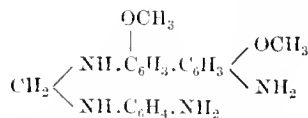
*Manufacture of a New Amidonaphtholdisulphonic Acid.* C. D. Abel, London. From "The Actien Gesellschaft für Anilin Fabrikation," Berlin, Germany. Eng. Pat. 2984, February 10, 1893.

THIS acid, which is described as a disulphonic acid of 1.8-amidonaphthol, is produced from the  $\alpha$ -naphthylamine disulphonic acid of Ger. Pat. 40,571 by conversion into  $\alpha$ -naphthylamine trisulphonic acid and subsequent removal of one sulphonic acid group by fusion with caustic alkalis. One kilo. of the sodium salt of  $\alpha$ -naphthylamine disulphonic

acid is heated on the water-bath with 3 kilos. of fuming sulphuric acid (containing 23 per cent. of  $SO_3$ ) until a sample diluted with water and treated with nitrite no longer gives a colour with phenols. The melt is then poured into water and converted into the lime or sodium salt. The neutral and acid sodium salts of the  $\alpha$ -naphthylamine trisulphonic acid, as well as the free acid, are very easily soluble in water, and the alkaline solutions have a fine yellowish-green fluorescence. In order to carry out the fusion, 1 kilo. of the sodium salt is fused with about 3 kilos. of caustic potash and some water in an open or closed vessel at 170°—220° C. until a sample diluted with water no longer shows the characteristic fluorescence of the  $\alpha$ -naphthylamine trisulphonic acid. The melt is then dissolved in water and hydrochloric acid, and the acid sodium salt of the new amidonaphthol disulphonic acid precipitates from the solution on cooling. The new acid is readily soluble in hot or cold water, yields no diazo compound, and gives a dark green solution with ferric chloride. It combines with diazo or tetrazo compounds to form valuable dyestuffs, and differs from the isomeric acid of Eng. Pat. 13,443 of 1890 (this Journal, 1891, 760) in only combining with one molecule of a diazo compound.—T. A. L.

*Improvements in the Manufacture of Unsymmetrical Diamido Bases, and of Colouring Matters therefrom.* J. C. L. Durand, E. Huguenin, and A. J. J. d'Andiran, Basle, Switzerland. Eng. Pat. 20,301, October 27, 1893.

THIS is an extension of Eng. Pat. 12,704 of 1893 (this Journal, 1893, 824), and describes the formation of a condensation product by heating together formaldehyde, tolidine, or dianisidine, and the basic hydrochloride of *m*- or *p*-phenylene diamine. If dianisidine and *m*-phenylene diamine be employed, the resulting compound has the formula—



The process is carried out in a similar manner to that already described, and the base when separated is slightly soluble in hot alcohol and benzene, but insoluble in ether. The hydrochloride and sulphate are readily soluble in water. The nitrite required for diazotising it corresponds to two amido groups, and the method for employing it in the production of colouring matters is similar to that described in the first-named patent. The condensation product now described gives a greenish-blue cotton colouring matter with  $\alpha$ -naphthol-*p*-sulphonic acid, whereas the condensation products described in the earlier specifications gave reddish-blue dyes with this component.—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Action of Nitric and Nitrous Acids on Silk.* L. Vignon and P. Lislely. Romen's Journal, 1893, 5.

WHEN silk is immersed in nitric acid of sp. gr. 1.133 at 45° for one minute it becomes coloured yellow, and the colour is fast to light and air. Nitric acid when pure does not produce this effect, but only after the addition of some nitrite. The coloured silk takes up base from alkaline solutions and the colour becomes darker. A fugitive yellow is produced by an acidified solution of nitrite, but is rendered fast by the addition of nitric acid. The yellow colour is bleached by a strong acid solution of "tin salt."—A. K. M.



*The Manufacture of India-rubber Waterproof Cloth.*  
C. O. Weber. *Zeits. ang. Chem.* 1893 [21], 22.

Very little reliable information on this important branch of the india-rubber industry is to be obtained from the various books published on this subject. This is largely due to the fact that india-rubber manufacturers maintain the strictest secrecy with regard to the materials, machines, and working methods employed. It is very doubtful whether all the waterproof manufacturers combined could point out a single secret worthy of the name. On questions regarding the nature and chemical capabilities of india-rubber, which certainly will play an important part in the future development of this industry, the manufacturers are woefully ignorant.

Of the raw materials employed in this industry, india-rubber is of course the most important, Pará rubber being used almost exclusively. Of late years "recovered rubber" is used in considerable quantities along with fresh rubber. This recovered rubber is obtained by boiling cuttings from waterproof cloth with dilute hydrochloric acid, whereby the cloth is destroyed, thin films of "proofing" remaining behind, which are then washed and subsequently dried. India-rubber substitutes, obtained by treating colza oil with sulphur monochloride, or by boiling castor oil with sulphur, are also largely employed. Free oil and free fatty acids, which these substitutes may contain if not carefully prepared, are apt to act upon the india-rubber most injuriously. The mineral substances added for weighting or colouring the proofing are safe if they are insoluble in water and do not contain any oxides capable of acting as oxygen carriers. As solvents for the india-rubber, petroleum benzine and a mixture of toluene, xylene, and cumene are almost exclusively used. Neither of these solvents should contain any fractions boiling above 140° C. The vulcanising agents are sulphur and sulphur monochloride. Fawcitt's antimony iodide has not been much used so far; its practical advantages seem as doubtful yet, as its vulcanising action appears mysterious. Adulteration of sulphur is practically an unknown thing; if suspected, its solubility in carbon bisulphide readily affords the desired information. Sulphur monochloride may contain large quantities of free sulphur and also sulphur bichloride. Both these impurities are liable to cause trouble in the vulcanising process. Sulphur monochloride should therefore always be analysed before use. This is done by determining its percentage of chlorine, of which the monochloride contains 52.6, the bichloride 68.9 per cent., so that the presence of 3 per cent. of bichloride in a sample of monochloride can be easily detected, and this quantity is considered unobjectionable. The analysis is carried out by filling a small glass stoppered bottle with the monochloride and weighing it. From 20 to 30 grms. of this monochloride are then poured off into a 1,000 cc. flask containing about 500 cc. of water, and decomposition is effected by shaking the bottle vigorously for some minutes. The weight of the decomposed monochloride is found by subsequently weighing the small bottle. The monochloride being decomposed, the flask is warmed on the water-bath, and for every gram. of decomposed monochloride 1 cc. of nitric acid (1.42) is added, the flask well shaken, cooled, and then filled up to the mark with water. In the solution so obtained the chlorine is then determined gravimetrically or volumetrically. The following analyses were made in this manner:—

I. Used 22.88 grms. of  $S_2Cl_2$ : carefully purified specimen.

After decomposition oxidised with 23 cc. of  $HNO_3$ .  
50 cc. of filtrate yield 2.428 grms.  $AgCl = 0.601$  grms.  $Cl$ .

Total  $Cl = 0.601 \times 20 = 12.02$  grms. = 52.53 per cent. (calcn. 52.6).

The sample contains 99.86 per cent. of  $S_2Cl_2$ .

II. Used 24.39 grms. of  $S_2Cl_2$ . Reddish amber-coloured liquid.

After decomposition oxidised with 24 cc. of  $HNO_3$ .  
50 cc. of filtrate yield 2.224 grms.  $AgCl = 0.550$  grms.  $Cl$ .

Total  $Cl = 11.00$  grms. = 45.10 per cent.

The sample therefore contains  $\begin{cases} 85.9 \text{ per cent. } S_2Cl_2 \\ 11.1 \text{ per cent. } S. \end{cases}$

III. Used 20.74 grms. of  $S_2Cl_2$ . Colour dark red-orange.

After decomposition oxidised with 21 cc. of  $HNO_3$ .  
50 cc. of filtrate yield 2.27 grms.  $AgCl = 0.502$  grms.  $Cl$ .

Total  $Cl = 11.24$  grms. = 54.1 per cent.

The sample therefore contains  $\begin{cases} 89.57 \text{ per cent. } S_2Cl_2 \\ 10.41 \text{ per cent. } SCl \end{cases}$

In connection with chloride of sulphur, carbon bisulphide must be mentioned, which should be as pure as possible. It contains generally some free sulphur, but seldom in objectionable quantities. The more or less nauseous smell of the commercial bisulphide seems to be due to two different substances, the composition of which is not known. The iron drums in which the bisulphide is stored frequently contain water, which must be carefully kept out of the vessels in which the bisulphide is mixed with the chloride of sulphur. The safest plan is to keep the bisulphide in storage tanks containing a quantity of burnt lime, as in this manner the bisulphide is always perfectly dry, and at the same time most of the objectionable impurities are retained by the lime. Amongst the raw materials for the waterproofing industry, also the textile fabrics must be classed, as they occasionally contain impurities which cause the deterioration of the india-rubber coating upon these fabrics. Best known in this respect are oils and free fatty acids in cloth, and as a rule cloth containing more than 2 per cent. of either of them is considered dangerous. Much more objectionable, however, is the presence of copper and manganese compounds in a cloth, exceedingly small quantities of which show a most destructive effect upon the india-rubber coatings. It is remarkable that of the numerous colouring matters, Bismarck brown is the only one which is detrimental to india-rubber.

The compounds mixed with the india-rubber in the preparation of the india-rubber "dough" are exceedingly varied, but no chemical considerations of any kind enter into their selection. The textile fabrics, after having received a sufficient number of coatings, are vulcanised either by the cold or by the hot process. In the first case the vulcanising agent employed is a solution of sulphur monochloride in carbon bisulphide. The concentration of this solution is generally stated much too high; it is used containing from 0.5 to 3 per cent. of sulphur monochloride. Equally unreliable are the statements regarding the amount of sulphur taken up by the india-rubber, and the temperature required in the hot vulcanising processes. It is, however, certain that the quantity of sulphur added to the india-rubber is always in excess of the quantity entering into combination with it. The vulcanising temperature is occasionally given as low as 210° F. This is obviously erroneous, being considerably below the melting point of sulphur. The vulcanising temperature should therefore not be less than 235° F., and it is certainly preferable to vulcanise at a higher temperature than this. The higher the temperature employed the shorter is the time required for vulcanisation. Using 6 per cent. of sulphur at 260° F., the vulcanisation is complete in two hours.—C. O. W.

## PATENTS.

*Improvements in the Manufacture of Linoleum, Kamptulicon, and other similar Floor Cloths, and in the Means employed in such Manufacture.* J. S. Farmer, Salford, and H. L. Storey and L. H. Storey, Lancaster. Eng. Pat. 23,983, December 29, 1892.

A ROLLER is prepared containing grooves corresponding to the fine lines and narrow spaces in a pattern or design. The grooves are filled with the material of which the floor-cloth is made, and the contents of the grooves are discharged on to the foundation fabric of the cloth. The solid colour blocks are then placed in their proper positions, and by means of pressure the material discharged from the grooves is squeezed and caused to fill up the interstices between the solid colour pieces.—R. B. B.



*Improvements in the Treatment of Cotton Cloths for Filtering Purposes.* H. E. Newton, London. From E. de Haen, List vor Hanover, Germany. Eng. Pat. 1770, January 26, 1893.

THE cotton cloths used for filtering acid liquids in filter presses are rapidly destroyed. Wool is too expensive to be commonly employed for this purpose. Nitrated cellulose withstands the action of acids much better than cotton, but the ordinary methods of nitration do not yield a suitable product. The patentee proposes to modify the nitrating process thus:—The cotton is immersed in cold nitric acid at 40°–50° B. for one hour, then steeped in cold sulphuric acid 62°–68° B. for one hour, and well washed. The material thus prepared withstands the action of all acids, and is even more durable than the woollen material sometimes employed for filtering purposes.—R. B. B.

*Improvements in the Method of and Apparatus for Washing Wool and other Fibres.* P. Kelly, Cross Hills, Yorks. Eng. Pat. 1897, January 28, 1893.

THE water or other washing liquid is stored in tanks, and drawn off as required into a cistern, which contains within it a smaller cistern. This inner vessel has perforated sides and bottom, and there is a clear space all round the sides and underneath it. The material is placed in the smaller cistern, and the liquid allowed to flow from the storage tank into the space between the two cisterns, whence it flows through the perforations and saturates the fibre. Within the smaller cistern a plate is caused to alternately rise and fall. On falling it presses on the fibre, squeezing the liquid from it, and on the motion being reversed the pressure is released, and the liquid flows again through the perforations and saturates the fibre. The reciprocating motion of the plate is continued until the washing is complete.

—R. B. B.

*Improvements relating to the Treatment of Fabrics and other Stuffs for the Purpose of Obtaining a Substitute for Ornamented Leather, Tapestry, Brocade, Embroidery, and the Like.* H. B. Lake, London. From the Fratelli Mora, Milan, Italy. Eng. Pat. 16,989, September 9, 1893.

THE materials employed are steeped in water, dried, and then coated with some preparation of a farinaceous, gummy or oleaginous nature. This coating may consist of starch, gum arabic, fish glue, boiled oil, or other substance, either alone or mixed with sawdust, bran, &c. The coating is applied by hand or by immersing the fabric in a bath containing the necessary ingredients. After drying the material is "satined" if a polished surface is required. It may then be embossed by engraved rollers or with hand stamps, and finally is coloured, bronzed, gilded, &c., as required.

—R. B. B.

## VI.—DYEING, CALICO PRINTING, PAPER-STAINING, AND BLEACHING.

*Some Properties of Lead Oxides.* A. Bonnet. Comptes rend. 1893, 117, 518–519.

WHEN cotton is mordanted in an alkaline plumbate, and well washed, the fibre becomes charged with the pure oxide of lead. The oxidising power of this body causes the destruction, more or less, of the fibre. A similar dissociation takes place when plumbites are used, without the inconvenience of oxidation, and the white oxide plays a part in a series of other reactions. Mordanted cotton immersed in baths of logwood, sumac, &c., immediately forms, especially with heat, a coloured lake, black with logwood, yellow-green with sumac. It is also possible with this mordant to deposit on a fabric most of the metallic

oxides by employing suitable baths. Gold, silver, mercury, vanadium, manganese, chromium, iron, cobalt, nickel, and zinc give characteristic reactions if solutions of neutral salts be used. These reactions are so marked that, for example, it is sufficient to use a bath of permanganate of potash containing 0.25 grm. of the salt per litre, to obtain at 80° or 90° a very dark deposit of binoxide of manganese. Traces of vanadium in an ammoniacal chloride solution are at once detected.

This displacement of the oxides is explained by the formation of salts of lead soluble by heating. In the same manner the chlorides of iron, cobalt, nickel, and chromium decompose with heat, depositing upon the material the corresponding oxide; at the same time a chloride of lead is formed in the bath which is soluble with heat, but is precipitated on cooling.—D. E. J.

*Indigo Salt, a New Preparation for the Dyeing of Indigo on Cotton.* E. Fischer. Chem. Zeit. 1893, 17, 1069.

THE indigo blue thus produced is stated to be brighter and much faster to soap and rubbing than ordinary indigo prints. Its use for discharge styles also possesses material advantages over the ordinary indigo. (This Journal, 1893, 908 and 988).—C. O. W.

## PATENTS.

*Process for Bleaching Raw Starch, Starch Gum, and Solutions thereof by Treatment with Chlorine and Ozone combined.* Siemens, Bros., and Co., London. From Siemens and Halske, Berlin, Germany. Eng. Pat. 2597, February 6, 1893.

THE raw starch is made into an emulsion with water, and subjected to the action of currents of chlorine and ozone, either simultaneously, or the treatment with chlorine may precede that with ozone. The bleached starch may then be converted into starch gum by well-known processes.

Or the gum may be first produced from starch and then bleached in a similar manner.

The chlorine may be employed in aqueous solution instead of in the gaseous state.—R. B. B.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Synthetic Formation of Citric Acid by Fermentation of Glucose.* C. Wehmer. Comptes rend, 117, 332.

See under XX., page 57.

*Action of Certain Metals on Acid Solutions of their Chlorides.* A. Ditte and R. Metzner. Comptes rend, 1893, 117, 691.

See under XI., page 43.

*The Production of various Carbides.* O. Mühlhäuser. Dingl. polyt. J. 1893, 289, 164–168.

FIFTEEN elements are known to form carbides. Of these the following are metallic: K, Ca, Ba, Fe, Mn, Cr, U; while the non-metallic are H, Cl, Br, I, O, S, N, B, Si.

The most noteworthy of the metallic carbides are the under-mentioned:—

*Potassium carbide*,  $K_2C_2$ , is prepared by passing acetylene over fused potassium. It is also found as a by-product in the manufacture of potassium.

*Calcium carbide*,  $CaC_2$ , is formed by heating a mixture of calcium, zinc, and carbon to a white heat, or by heating calcium carbonate with magnesium and carbon. Barium

carbide is prepared in a manner analogous to that by which the calcium compound is prepared. All these carbides have the property of evolving acetylene when treated with water.

*Iron carbide* has not hitherto been allotted a definite formula, as the amount of carbon which iron is capable of taking up depends on various conditions. The maximum amount may be taken as 4.6 per cent. of carbon. It is worthy of remark that the substance used by Castner in the preparation of sodium appears to be an iron carbide.

*Manganese carbide* has been investigated by Wahl and Green and by Moissan. The former ascribe to it the formula  $Mn_3C$ , while the latter has prepared bodies with a content of carbon ranging from 6.3 to 14.6 per cent. by means of the electric furnace.

*Chromium carbide*, containing carbon varying from 8.6 to 11.9 per cent. of C, has been prepared by Moissan by the same means.

*Uranium carbide* with 5 to 13 per cent. of C has also been obtained by the same investigator.

Carbides of the non-metallic elements which claim comment are—

*Sulphur carbide* (carbon disulphide) which can be obtained in a manner different from that in which it is manufactured, viz., by heating a mixture of calcium sulphate and carbon in the electric furnace. The following reaction is said to occur:— $2CaSO_4 + 13C = 2CaC_2 + CS_2 + 8CO$ .

*Boron carbide* is obtained by heating a mixture of boric anhydride with excess of carbon in an electric furnace. The progress of the reaction is distinguished by the following stages. The mass softens and decomposition sets in with the evolution of carbon monoxide, which causes the formation of bubbles in the charge until the whole of the boron is converted into carbide. On cooling, the boron carbide is found in rounded masses of metallic lustre, embedded in the excess of carbon. In order to separate it from its matrix, the whole product is powdered and heated to dull redness in a stream of oxygen by which means the whole of the free carbon and a part of that combined with the boron, are oxidised. The residue is powdered and treated successively with caustic soda, water and hydrochloric acid. A powder having the appearance of graphite, and like that substance possessing the property of staining the fingers, is obtained. On heating, the boron carbide becomes spongy and plastic, and on further heating, fuses. It can only with difficulty be burnt in oxygen, but is readily oxidised by lead chromate. It has the composition indicated by the formula  $BC$ . On fusion with sodium carbonate it yields sodium borate and carbon dioxide.

*Silicon carbide* is produced when a mixture of sand, carbon, and common salt is heated in the electric furnace. The charge, consisting of equal parts of sand and carbon with a little salt, is introduced into the furnace and connection between the electrodes made by a layer of powdered carbon, the whole being then covered with a further quantity of the mixture. During the reaction, evolution of gas takes place, and on its cessation the reduction may be considered complete. On examining the contents of the furnace it is found that the carbon core which serves to connect one electrode with the other, has suffered no change, but that there is a zone immediately surrounding the path of the current consisting of a shining black crystalline mass disposed radially, containing about 66 per cent. of free carbon and 33 per cent. of silicon carbide. The free carbon is distinctly graphitic, staining the fingers, &c. The composition of the silicon carbide separated from this matrix is as follows:—Silicon, 68.26; carbon, 30.49; ferric oxide, 0.77; lime, 0.48 per cent.

In order to separate silicon carbide from admixed graphite, it is heated to redness in a stream of oxygen. The properties of the substance obtained in this zone of the reaction are almost identical with those of that formed in the zone immediately exterior to it, the only difference consisting in small variations in optical behaviour. The main product of the reaction occurs, as already stated, immediately exterior to the zone of mixed carbide and free carbon. The crystals, which have a greenish tinge, are disposed radially to the

electrodes of the furnace. A sample analysed had the following composition:—Silicon, 62.70; carbon, 36.26; ferric oxide and alumina, 0.93; magnesia, 0.11 per cent.

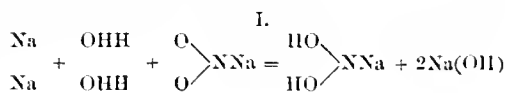
The purification of the carbide is effected by successive treatment with hydrochloric acid, dilute caustic soda and water. The dried powder is heated to dull redness in a stream of oxygen for an hour, and the product treated with hydrofluoric acid together with a little sulphuric acid, whereby it is obtained approximately pure. The substance thus purified has the composition:—Silicon, 69.10; carbon, 30.20; ferric oxide and alumina, 0.46; lime, 0.15 per cent.; and corresponds fairly well with the formula  $CSi$  which requires 70 per cent. of silicon and 30 per cent. of carbon. When pure carbon and silica are used, colourless crystals are obtained, but with the usual materials enough iron is present to give the crystals a greenish or greenish-yellow colour. Silicon carbide is not sensibly soluble in any acid, nor is it appreciably attacked by dilute solutions of the alkalis but is decomposed by fusion with sodium carbonate or caustic soda, sodium silicate being formed and the carbon set free in a condition in which it eventually takes fire, leaving the melt white. Silicon carbide has the curious property of remaining suspended in water for months when finely powdered, but it is precipitated by the addition of an acid or a salt. The zone exterior to that containing the crystals of silicon carbide, consists of a mass of particles of the same size as the original grains of sand. These particles have a similar composition to the silicon carbide already described, and can be purified in the same way but are much softer and can be readily powdered. An analysis of this variety is as follows:—Silicon, 65.42; carbon, 27.93; ferric oxide and alumina, 5.09; lime, 0.38; magnesia, 0.21 per cent.

It appears to be identical with a substance prepared by Schützenberger and is the amorphous variety of silicon carbide formed at a lower temperature than that which is crystalline.

Crystalline silicon carbide is now an article of commerce under the name "Carborundum," and seeing that in hardness it is but little inferior to the diamond it is not surprising that it has been already adopted for grinding and polishing purposes.—B. B.

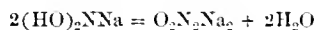
*Hyponitrous acid.* A. Thum. Monatsh. Chem. 1893, 14, 294.

HYPONITROUS acid  $H_2N_2O_2$  is obtained by the reduction of nitrite of soda by sodium or ferrous hydrate. The reaction proceeds in the following manner:—

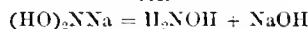


I.

II.



III.



Excess of the reducing agent should be avoided, as it proves detrimental to the yield of hyponitrous acid. If instead of sodium, ferrous hydrate be employed as the reducing agent, also smaller yields are obtained. Anhydrous hyponitrous acid cannot be obtained by decomposing dry hyponitrite of silver by means of sulphuretted hydrogen owing to the liability of this acid to explode spontaneously even at very low temperatures. Solutions of the acid are, however, easily obtained by decomposing the silver salt by means of hydrochloric acid. This solution possesses a very acid reaction and is not altered by boiling with dilute acids or alkalis. Permanganate of potash in the presence of sulphuric acid oxidises the solution quantitatively into nitric acid, whilst in alkaline solution the oxidation only proceeds as far as conversion into nitrite. Nitrous acid completely decomposes hyponitrous acid. Hyponitrous acid is a dibasic acid, as is proved by the existence of an acid barium hyponitrite. It liberates no iodine from an

acid solution of potassium iodide, nor does it prevent such a liberation by the action of nitrous acid. Hyponitrous acid is very indifferent to reducing agents and is not affected by boiling with dilute sulphuric acid and zinc. Reducing agents in alkaline solutions slowly destroy it.—C. O. W.

*New Processes and Apparatus for Concentrating Sulphuric Acid.* M. M. Gerber. *Monit. Scient.* **41**, 366—369.

This paper completes the description given in September 1892 (*see also* this Journal, 1892, 434), of Kessler's process. The process depends upon the action of heated air or gases, passed over or through the acid, whereby the concentration is effected at a temperature considerably below that of the boiling point *in vacuo*. Thus, while concentrated sulphuric acid boils under ordinary pressure at 328° C., and *in vacuo* at about 230°, the chamber acid is concentrated up to 66° B. in Kessler's apparatus at a temperature not exceeding 170° C., and leaves the apparatus at 120°, flowing into a receptacle wherein it gives up its heat to the chamber acid intended for concentration. The effluent gases also escape at a temperature so low as to occasion very little loss of heat.

The apparatus includes a furnace in which coke is burned, the products of combustion being led into a "saturator" divided into compartments, superposed upon which is the "recuperator," with tiers of communicating vessels, containing the acid to be concentrated 2 to 4 c.m. in height, having at alternate ends overflow pipes to successive tiers, and then into the "saturator" wherein the concentration is completed. There are caps to the acid-containing vessels, so arranged that the entering heating gases bubble through the acid in their progress; the arrangement being similar to that of the distilling apparatus of Derome or Savalle. The dome-shaped head to the "recuperator" opens into a pipe, curved so as to lead horizontally to the bottom of a vessel charged with small coke, which filters the gases free from any acid vapours, these condensing, and the liquid being collected. Some slight pressure or suction is necessary to cause the heated gases to bubble up through the layers of acid, both in the "saturator" and the "recuperator," which may be obtained by the draught of a chimney, or as is shown in the drawings of the apparatus, by a steam-jet operating in the horizontal tube leading from the "recuperator" to the condenser. The rectangular trough constituting the "saturator" is formed of an acid-resisting stone, lined with resisting cement, and divided by partitions, slightly dipping into the acid, into compartments, so that the heated gases passed in by a cast-iron pipe pass from compartment to compartment by bubbling under the immersed sides. The "recuperator" may be made wholly of lead; but preferably the uppermost tier, containing the weakest acid, and not subjected to a higher temperature than 110° C., is of lead, while the lower tiers are of similar resisting material to the "saturator."

The fuel used to obtain the heated gases is small gas coke, the consumption of which does not exceed 8 to 10 kilos. per 100 kilos. of acid at 66° B. produced from chamber acid of 52 B. The gases should not enter the concentrating apparatus at so high a temperature as 500° C., as in that case, the strength of the acid obtained is under 66° B., this being accounted for by the tendency to dissociation of the monohydrated acid into the anhydride or pyro-acid and water, as the temperature approaches the boiling point of the acid. The apparatus described has been in use for the past two years in the Clermont-Ferrand works.

—E. S.

*Lead Tetrachloride.* H. Friedrich. *Monatsh. f. Chem.* **14**, 1893, 505—520.

AMMONIUM chloroplumbate,  $\text{Pb}(\text{NH}_4)_2\text{Cl}_6$  or  $2\text{NH}_4\text{Cl}, \text{PbCl}_4$ , is prepared by treating pure lead chloride,  $\text{PbCl}_2$ , with about 20 parts of strong hydrochloric acid, and saturating with chlorine at 10—15° C., whereby almost complete solution ensues, forming a dark yellow clear fluid, smelling strongly of chlorine. To this ammonium chloride is added dissolved in 10 parts of water, both fluids being previously cooled by ice. In a short time a crystalline

precipitate forms, of a yellow colour with slight greenish tinge; if too strong ammonium chloride solution were used, this is apt to contain particles of salammoniac; but if not, it is practically pure ammonium chloroplumbate, furnishing sharp numbers on analysis after collection on a pump-filter, and washing with icecold alcohol which has no action on it. About two-thirds of the theoretical yield are thus obtained.

By dropping this double salt little by little into concentrated sulphuric acid cooled with ice, an oily yellow liquid is ultimately obtained, consisting of lead tetrachloride,  $\text{PbCl}_4$ . The first action gives rise to hydrochloric acid and particles of solid ammonium sulphate, which forms a sort of milky fluid with the sulphuric acid; when the oil has sufficiently accumulated the acid is poured off, and the oil agitated with fresh acid until clear and free from suspended solid matter. Lead tetrachloride thus obtained is a heavy highly refractive fluid resembling antimony pentachloride; it fumes in the air like tin tetrachloride, emitting an odour resembling that of hypochlorous acid, and causing a sweet taste to be perceptible in the mouth. It can be preserved under sulphuric acid, but is liable to explode, especially when heated to 105°, lead chloride,  $\text{PbCl}_2$ , and free chlorine being formed. Two determinations of its specific gravity at 0° gave the values 3.182 and 3.184, mean 3.183. In a freezing mixture it solidifies to a transparent yellowish crystalline mass, which melts at about -15° C. It forms a crystalline compound with hydrochloric acid, probably  $\text{H}_2\text{PbCl}_6$ , when in contact with a small quantity of cooled concentrated acid; larger quantities dissolve it in all proportions, forming solutions from which salammoniac precipitates ammonium chloroplumbate. Contact with large quantities of water forms lead dioxide and hydrochloric acid, a similar result being observed with ammonium chloroplumbate. By cautiously passing a current of dry chlorine through a retort containing sulphuric acid in which lead tetrachloride is suspended, the whole being gently heated, a small amount of volatilised substance can be condensed in a chilled receiver, showing that the compound is volatile. In all these general properties a very close resemblance is noticeable between tin tetrachloride and lead tetrachloride; more especially in that both of these compounds resist the action of sulphuric acid, whilst both tin dichloride and lead dichloride are readily acted on with evolution of hydrochloric acid. Germanium tetrachloride is also unattacked by concentrated sulphuric acid, even on warming; on the other hand antimony trichloride and pentachloride are both decomposed with evolution of hydrochloric acid, as also are both ferrous and ferric chlorides in the anhydrous condition.

Attempts to prepare lead tetrabromide, and the alkaline bromoplumbates proved unsuccessful: on cooling in ice for 10—12 hours a mixture of lead bromide and potassium bromide solution containing free bromine, a reddish brown crystalline body separated; this immediately decomposed on removal from the liquid with evolution of bromine, leaving a white residue containing lead bromide and potassium bromide; whence its composition presumably was that of potassium bromoplumbate,  $\text{K}_2\text{PbBr}_6$ .—C. R. A. W.

*The Specific Gravities of Solutions of Bleaching Powder.* G. Lange and F. Bachofen. *Zeits. angew. Chem.* 1893, 326—328.

This investigation was carried out for the purpose of establishing the relationship existing between the specific gravities of bleach solutions and the available chlorine contained therein. The difficulties attending this research may be reduced to the difficulty of obtaining a fairly representative sample of bleaching powder, since, as is well known, "bleach" gradually loses its available chlorine, and calcium chloride and calcium chlorate are formed as secondary products. The authors base their results on what they consider a fair average sample of well-made bleaching powder, such as is found in the market, and consider that this very fact will enhance the value of their investigation.

In the first instance, a concentrated solution of bleaching powder was prepared and kept protected from air and light.

It was analysed, its specific gravity taken, and other solutions prepared therefrom by suitable dilution. At the conclusion of the investigation, which was carried out with the utmost expedition, the original liquor was tested over again and found to be practically unchanged. The available chlorine was estimated according to Penot's method and the results so obtained checked by the hydrogen peroxide method (this Journal, 1890, 21—24). The results thus obtained being a trifle too high were discarded in favour of those yielded by Penot's method. The chlorine present as calcium chloride was estimated after adding the exact quantity of arsenious acid required by the former test, then acidifying with nitric acid and titrating with silver nitrate, by deducting from the chlorine thus obtained, the quantity of available chlorine as determined by the arsenious acid test. Another sample served for the determination of the chlorate by means of ferrous sulphate and permanganate, deducting from the result the quantity of hypochlorite. The liquor contained:—

	Grms. per Litre.
Available chlorine .....	72.17
Chlorine as chloride .....	6.74
Chlorine as chlorate .....	0.13
Lime .....	65.53

or calculated on the corresponding salts:—

	Grms. per Litre.
CaOCl <sub>2</sub> .....	129.09
CaCl <sub>2</sub> .....	10.54
Ca(ClO <sub>3</sub> ) <sub>2</sub> .....	0.38
Ca(OH) <sub>2</sub> .....	4.21

The specific gravities were taken at 15° C. by means of officially certified hydrometers (a series of 21, ranging from sp. gr. 0.76—2.0), which could be read to 0.0005. The readings were found to be in perfect agreement with pycnometer determinations. The solutions were approximately diluted to the required strength, cooled to somewhat below 15° C., then allowed to stand until the temperature rose just to 15° C., and diluted with water to the proper specific gravities. The results marked by an asterisk are obtained by interpolation. When plotting the results, a slightly convex line is obtained. The uncorrected experimental data are found in column B., whilst in column C. allowance is made for the very slight irregularities of the curve. The values of any specific gravities not here given may be calculated with perfect accuracy by interpolation.

#### SPECIFIC GRAVITIES OF BLEACHING POWDER SOLUTIONS.

Specific Gravity at 15° C.	Available Cl. Grms. per Litre. Observed.	Available Cl. Grms. per Litre. Corrected.	Specific Gravity at 15° C.	Available Cl. Grms. per Litre. Observed.	Available Cl. Grms. per Litre. Corrected.
A.	B.	C.	A.	B.	C.
1.1155	71.79	71.79	1.0600	35.81	35.81
*1.1150	71.50	71.50	1.0550	32.68	32.68
1.1105	68.96	68.40	1.0500	29.41	29.50
*1.1100	68.00	68.00	1.0450	26.62	26.62
1.1060	65.33	65.33	1.0400	23.75	23.75
*1.1050	64.50	64.50	1.0350	20.44	20.44
1.1000	61.17	61.50	1.0300	17.36	17.36
1.0950	58.33	58.40	1.0250	14.47	14.47
1.0900	55.18	55.18	1.0200	11.41	11.41
1.0850	52.27	52.27	1.0150	8.48	8.48
1.0800	48.96	49.96	1.0100	5.58	5.58
1.0750	45.70	45.70	1.0050	2.71	2.71
1.0700	42.31	42.31	1.0025	1.40	1.40
1.0650	38.71	39.10	1.0000	Trace.	Trace.

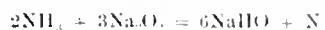
—H. A.

*The Action of Ammonia Gas on Peroxides.* O. Michel and E. Grandmougin. *Ber.* 26, 1893, 2565—2568.

The authors adopted the following method of investigation. The peroxide under examination, contained in a porcelain boat, was heated in an ordinary combustion-tube placed in a gas furnace. Dry ammonia gas was passed through the tube and the permanent gases given off collected over sulphuric acid and analysed.

The following observations were made:

*Sodium peroxide.*—The oxide at first turns yellowish and a small quantity of oxygen is given off. It then fuses, becomes brown, and froths up, giving off at the same time large volumes of practically pure nitrogen. After a time the evolution of gas ceases, the mass sets and again becomes white. The residue in the boat was found to have the following composition:—NaHO, 91 per cent.; NaNO<sub>2</sub>, 6 per cent.; NaNO<sub>3</sub>, 3 per cent. The main reaction evidently, therefore, proceeds according to the equation—



*Barium peroxide.*—In this case the reaction requires a higher temperature. The oxide fuses and pure nitrogen is formed. The residue consists of barium hydrate, no trace of nitrate or nitrite being produced.

*Manganese peroxide.*—The oxide is reduced to MnO, and nitrogen and water are formed. At high temperatures, however, further reduction occurs and nitrous fumes are formed.

*Lead peroxide* turns yellow and is reduced to PbO with violent evolution of gas and water vapour. At the same time ammonium nitrate and nitrite and nitrous fumes are produced.—H. T. P.

#### PATENTS.

*Improvements in the Preparation of Chromic Acid, and in the Extraction of Pure Acids from Alkaline Salts.* E. Placet and J. Bonnet, Paris. Eng. Pat. 22,819, December 12, 1892.

Pure chromic acid is obtained by electrolysis of an alkaline chromate or bichromate, the chromate solution being placed in a vessel within which is a porous vase, the latter being charged with water or the same solution. Electrodes of carbon, or other unattackable conducting body, are used, the positive pole of the source of electricity being connected to the carbon in the main body of the solution, and the negative pole to the carbon within the porous vase. The chromate solution is fed with the salt as required, and the alkaline solution found in the vase is removed from time to time and replaced by the original liquid. The chromic acid separated in the outer vessel is crystallised in the ordinary manner. The separated alkali may be utilised for treating chrome minerals; or in obtaining alkali chromates. A number of applications are specified for the chromic acid, and in this connection reference is made to three patents, Nos. 22,854, 22,855, and 22,856 of 1891.

The process is stated to be applicable generally to the extraction of pure acids from binary compounds.—E. S.

*Improvements in the Manufacture of Cyanides and Cyanogen Compounds.* J. D. Gilmour, Glasgow, Eng. Pat. 24,116, December 31, 1892.

A mixture of carbon, alkali, alkaline oxide or carbonate, or alkaline earth, (but preferably potash), is strongly heated in a current of nitrogen obtained in a subsequent stage of the process. The product, more or less converted into cyanide, is lixiviated, and carbonic acid gas is passed through the solution, which is preferably at boiling heat. The hydrocyanic acid that is disengaged, is condensed and added to strong sodium hydroxide solution to obtain sodium cyanide. The carbonic acid required is obtained by burning carbon in air to obtain a mixture of carbonic acid and nitrogen, which mixture, when led through the solution, where the CO<sub>2</sub> is

absorbed in liberating HCN, leaves nitrogen after condensation of the hydrocyanic acid, which is utilised, as first described, in assisting in the formation of a cyanide.

—E. S.

*Improvements in Apparatus for the Making or Manufacture of Caustic Soda or Caustic Potash, being a Method of Passing the Chlorine and Sodium Gases to the Receiver.* F. W. Petrie, Rochdale. Eng. Pat. 472, January 10, 1893.

THE invention consists in improvements upon Eng. Pat. 15,792, 1889, and especially upon Eng. Pat. 12,977, 1892, relating to the electrolytic production of caustic soda or caustic potash. The object of the present improvements is to remove the gases (chlorine and sodium vapour) from the electrolytic generating chamber, the chlorine by means of a blower or fan connected to a tube entering one compartment of the chamber; and the sodium vapour by a fan intercepting between the chamber and the receiver. The chlorine is conducted to a chamber containing lime, for production of bleaching powder. The receiver for the sodium is called the "caustic soda chamber." A vertical tube communicating with the atmosphere is shown entering the sodium compartment of the electrolytic apparatus, whereby, as the fan rotates, "an admixture of air with the metals is obtained," "facilitating the conversion of the sodium metal into a gas," besides "withdrawing the same from the generating chamber."—E. S.

*Improvements in the Treatment of Manganates for the Production of Permanganates suitable for use for Disinfecting or other Purposes, and in Apparatus therefor.* W. J. Menzies, Malpas, Chester. Eng. Pat. 1213, January 19, 1893.

ALTHOUGH permanganates are excellent disinfectants, their high price forbids their general use. On the other hand, the cheaper manganate of soda must be used in conjunction with sulphuric acid, and this constitutes a serious disadvantage. Another objectionable feature is that the commercial article contains much caustic soda. The object of this invention is to convert manganate of soda into permanganate by means of carbonic acid. The simplest way of doing this is to mix the salt intimately with 1 to 1½ times its weight of bicarbonate of soda; or the conversion may be effected by carbonic acid gas.—H. T. P.

*Improvements in and Apparatus for the Manufacture of Bleaching Powder.* E. K. Muspratt, Liverpool, A. Carey, Browside Gateacre, Lancaster, and V. C. Driffield, Lancaster. Eng. Pat. 1214, January 19, 1893.

IN treating lime with dilute chlorine in compartmented apparatus, excessive heating is prevented by causing the gas to pass from compartment to compartment through tubes which may be connected to cooling coils immersed in water or otherwise. Or, the communicating pipe may be of such length that the heat of the gas may be dispersed. The intermediate cooling apparatus may be modified in described ways, to suit requirements.—E. S.

*Improvements in the Extraction of Carbonic Acid from Mineral Carbonates, and in Apparatus therefor.* A. Van Berkel, Berlin. Eng. Pat. 2274, February 1, 1893.

THE mineral carbonates are strongly heated in a rotating drum set in a suitable heating chamber, on trunnions, one of which is hollow, the opening to the interior of the drum being guarded by a perforated cap. The pipe from the hollow trunnion communicates with a suction pump, by which the gas is drawn off as it forms. The sides of the drum are corrugated or provided with deep hollows, so that as it rotates, the mineral is carried upwards till it falls through the heated space within the drum, and so continuously. The drum is provided with a suitable manhole for charging and discharging.—E. S.

*Improvements in the Manufacture and Production of Salts or Compounds of Alumina or Aluminium.* J. L. Kessler, Clermont-Ferrand, France. Eng. Pat. 2710, February 7, 1893.

THE invention mainly consists in freeing aluminium sulphate from iron by causing it to crystallise with sodium sulphate to form soda alum, the iron being then left in the mother-liquor. As a preliminary treatment, it is preferred to boil the solution of the aluminium salt with a soluble phosphate, or with calcium or aluminium phosphate in small proportion. When the alumina precipitated is white, the quantity of calcium phosphate added is known to be sufficient.

Reference is made to Augé's process for obtaining soda alum, described in the French Patent No. 201,440, and its differences from the present processes are pointed out. The inventor has discovered that a solution of soda alum is liable, under certain conditions, to yield a cheesy magma of minute crystals, which crystals contain less water than the true sodium alum crystals, which latter are larger and transparent. And a crystallisation of either kind may be induced in liquors at certain temperatures or of certain densities, by introducing crystals of the true alum, or a portion of the magma. A number of processes are described in detail, whereby the desired kind of crystals may be obtained. According to one of these processes, a magma as obtained in Augé's process, is in the moist state set to drain with addition of the true alum crystals, when the mass becomes transformed throughout, without stirring, into the desired form of crystals. Also, if the solution of the alum is evaporated until it attains a temperature of 107° to 110° C. In another process, the soda alum is formed at a low temperature by alternate additions of aluminium sulphate and of sodium sulphate. Or a boiling solution of aluminium sulphate at 52° B. is allowed to cool to 90° C., and a cold mother-liquor from a preceding operation is added, or a cold saturated solution of soda alum, sufficient to lower the temperature to 40° C., and having a density of 40° B., and then the required proportion of sodium sulphate crystals are added to form the alum. The cold produced by the solution of the sodium sulphate crystals, causes the "zone of dangerous temperature" at which the objectionable magma is formed, to be passed, and on cooling, the true alum crystals are deposited. These may serve as examples.

Purification of the crystals obtained is effected by continuous washing by processes of displacement or clarification, as used in lixiviating crude soda.

In preparing aluminium sulphate for making soda alum, it is preferred to act on the aluminous matter by sodium bisulphate and excess of sulphuric acid. Details are given.

Soda alum is partly dehydrated and obtained as a translucent porcelain-like salt, by concentrating its solution by means of thick lead steam coil until the temperature rises to 115° C., when it is run out to set.

A salt "enriched with aluminium" is prepared by cooling the soda alum solution to below 10° C., the solution being sufficiently diluted to retain aluminium sulphate, whilst crystals of sodium sulphate separate. The enriched salt "being drained is evaporated in the manner and in the forms customary for sulphate of alumina." It may be rendered basic by addition of alumina.—E. S.

*An Improved Method of Producing Ammonium Formate, and from this Hydrocyanic Acid.* P. R. de Lambilly, Nantes, France. Eng. Pat. 3735, February 20, 1893.

CARBON monoxide is saturated with ammonia and the mixture passed through tubes heated to 100°—130° C. and containing porous substances capable of occluding gases, such as punice or animal charcoal, and either platinised or not. Ammonium formate in the form of vapour is thus produced, and may be condensed and collected if desired, or it may be utilised for the production of hydrocyanic acid by passing the vapours direct through a second set of tubes heated to a temperature of at least 210° C. and also containing some porous material. At this temperature ammonium formate is decomposed into water and hydrocyanic acid.—A. K. M.

## VIII.—GLASS, POTTERY, AND ENAMELS.

### PATENTS.

*Improvements in the Manufacture of Glass Articles in Imitation of Agate.* Count Solms-Baruth, Silesia. Eng. Pat. 10,384, May 26, 1893.

The glass used may have the following composition:—Basalt, 100; soda, 50; borax, 10; carbonate of lime, 20; sand, 80 parts; and chloride of silver, 1 part.

Into the molten glass are introduced pieces of basalt, lava, slag, iron ore, or burnt pyrites, after which dichloride of tin is added to the mixture. Owing to the incomplete solution of the basalt, &c., a turbidity is produced, giving an appearance similar to that of agate. By cooling the outer surface of the glass more rapidly, an improved effect is obtained by the production of a deeply-coloured surface upon a light background.—V. C.

*Improvements in or relating to Apparatus for Drying "Slip" and Evaporating Liquids.* S. Peake, Gillow Heath, near Congleton. Eng. Pat. 15,616, August 17, 1893.

The invention has reference to means for drying "slip" used in the manufacture of tiles, jet and Rockingham ware, and the like. A series of steam pipes connected together in the form of a gridiron are suspended by a chain over the "slip" kiln or other container. The pipe system is lowered into the "slip" to heat it and thereby evaporate the moisture until a sufficient degree of consistency is obtained. In order to conveniently lower the piping, swivel unions are employed at the joints; the condensed water in the steam-pipes is got rid of by means of a steam-trap attached at any suitable point.—E. G. C.

*Improvements in the Manufacture of Enamelled Goods.* H. Claus, Thale, Germany. Eng. Pat. 20,817, November 2, 1893.

In the methods formerly employed the spotting and marking of the metallic surface were obtained by oxidation of the steel or iron by the action of the acid in the enamel, such enamel consisting of silicate, borax, gypsum, and soda. This enamel requires very careful drying in a separate drying room, and the markings produced depend on the structure of the metal, a homogeneous material failing to give the variegated appearance which is desired. In this invention an enamel of the following composition is employed:—Felspar, 130; borax, 125; quartz, 70; soda, 25; saltpetre, 17; fluorspar, 10; antimony, 4 parts; cobalt,  $\frac{1}{2}$  part.

The metallic surface having been covered with the enamel, a mixture of equal parts of blue vitriol and green vitriol is powdered over the surface of the enamel. The whole is air-dried and then fired; the salts of copper and of iron descend through the enamel to the metal plate. Oxidation takes place at the surface of the metal through the action of the alkaline enamel upon the metallic salts, and the spotting is produced. The spotting thus produced is independent of the structure of the metal, but can be varied at will by using larger or smaller grains of the metallic salts. An additional advantage gained by the process is that careful drying in a separate drying room is not necessary.—V. C.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*The Testing of Portland Cement.* P. Cotte. Eng. and Mining J. 56, 722

For the test for fineness of grinding, three sieves are used, Nos. 50, 80, and 200. A well-made Portland cement will leave nothing on the 50 sieve, a residue of from 5 to 7 per cent. on the 80, and from 25 to 30 per cent. on the 200. Some will leave 15 to 20 per cent. on the 80, and 10 to 50 per cent. on the 200, but these should be rejected. The presence of particles of coal shows a careless selection of clinkers, whilst soft yellow grains show under-burned clinkers. The residue should be composed of nothing but hard black grains with sharp angles. The specific gravity of the cement is between 3.050 and 3.175. The most common adulterant is slag, but as its specific gravity is only about 2.8, any objectionable quantity might bring the specific gravity down below 3.050, which is the lowest that can be tolerated.

As to chemical composition, analyses of all kinds show that the constituents vary within the following limits:—

	Per Cent.
Silica .....	20.50 to 26.10
Alumina .....	5.20 „ 10.60
Iron oxide .....	2.10 „ 5.70
Lime .....	51.20 „ 67.31
Magnesia .....	0.33 „ 2.80
Sulphuric acid .....	0.20 „ 1.78

The relation between the silica and alumina on the one hand and the lime on the other constitutes the "degree of hydraulicity," which should not be below 0.43, but which should be raised to 0.46 when the cement is to be used on work in sea water. Oxide of iron should not exceed 4 per cent., and any cement having more than 3 per cent. of magnesia should be rejected. Magnesian cements give good tensile tests and look well for several months after use, but in course of time swelling takes place with almost irresistible force. Two or three per cent. of sulphate of lime can be present without injurious results, if soft water is used, but with sea water the presence of 1 per cent. of sulphuric acid would be dangerous.

The initial setting of Portland cement, gauged neat, should not begin before 30 minutes, and the final setting should not take place before 3 hours, a freshly-made cement usually setting in from 4 to 7 hours, although imported and old material will sometimes take 12 hours or more. Slow setting is almost essential, and a quick-setting cement, if good and well made, can be rendered slow-setting by exposing it to the air in thin layers. The test for stability is an important one, and a briquette, 24 hours after gauging, which is immersed in boiling water for 6 or 12 hours and shows no alteration, may be considered perfectly safe. Cold water is now rarely used for this test, the boiling water bringing out the effects of free lime very quickly. If the cement is mixed with a solution of calcium chloride (10 or 60 grains anhydrous salt per litre) and the cake immersed after 24 hours in the same solution, it will show signs of cracking in a few days if 3 or 4 per cent. of sulphate of lime is present.

The 24 hours' test for tensile strength has no value. Briquettes should be tested after 7 and 28 days, when, if good, they should withstand without rupture strains of 400 and 500 lb. per sq. in. respectively. This is with neat cement, but, as sand is generally mixed in practically, a test of a mixture of sand and cement is much more important. Three parts of cement and one of crushed quartz by weight is generally adopted, but the amount of water is not agreed upon. In Europe 10 per cent. is prescribed, and the mixture is compressed so as to bring out the full strength of the cement. In America the test is conducted more in accordance with the practical use, generally with more water and loose filling, but the results are not so concordant as in the European method. In Europe the crushing strength is sometimes considered more



important than the tensile, and tests are also made of the bending by bars of standard length, of the imperviousness and of adherence, but these have not yet been adopted in the United States.—A. W.

*The Preservation of Timber from the Attacks of Insects.*  
P. P. Dehérain. Comptes rend. 1893, **117**, 694—696.

THE author has observed that the sap-wood of trees used for timber is more subject to the attack of insects, such as boring worms, than the true wood, and farther, that amylaceous sap-wood is the kind most freely consumed by them. Besides this, he has found that the powder left by the consumption of the wood by these insects is free from starchy constituents. In order to deprive the timber of such constituents the plan of removing the bark at the foot of the tree, three or four months before felling, is found to be efficient, a mere ring a few centimetres in depth sufficing, provided that all growth below it be destroyed. Experiments with oaks from which the bark had been removed some months before felling, and others which had been decorticated at the time of felling, and a third series left with the full complement of bark, showed that the timber resisted in the order named. The author is of opinion that the amylaceous matter arising from the leaves is conveyed by the sieve tubes of the liber or inner bark (which run vertically), because the removal of the bark in a helical form is as efficient in securing the absorption of the starchy material as is an annular decortication. The best time to remove the bark is in the spring, the trees being felled in the following October.—B. B.

*The Weathering Properties of Sandstones.* W. Bolton.  
Dingl. Polyt. J. 1893, **289**, 43—48.

THE author points out that the usual criteria for the quality of building stones, such as their strength in compression, their absorptive power for water, and their behaviour when tested for weathering properties in the conventional manner, are not always conclusive. In particular, the compressive strength of the stone is seldom of much value in judging quality, as even inferior stones possess a crushing strength much above anything to which they are likely to be exposed in practice. Attempts to rank building stones according to their specific gravity have proved quite illusory, as no connection can be traced between this property and their mechanical value. A similar negative result has attended endeavours to find a relation between the tensile strength of the stone, its capacity for absorbing water and the force of expansion of the latter which is exerted in freezing. Methods directed to the determination of the capability of stones to resist those influences known collectively as "weathering," comprise the examination of the porosity of the stone as indicated by the quantity of water that it will absorb under standard conditions, the attack of its soluble constituents by hydrochloric acid and the observation of its behaviour when a liquid is caused to crystallise in its pores. With regard to the last-named test, the method formerly employed, which is known as Brard's test, and consists in repeatedly dipping the stone into a solution of sodium sulphate and allowing the salt to crystallise, is valueless as the assumption on which it is based, namely that expansion takes place during the crystallisation of the salt, is erroneous, the reverse being true. Dipping in water and freezing, many times repeated, is a more rational proceeding.

The author, however, prefers to base his judgment on the analysis of the stone, especial attention being directed to the nature of the substances serving as binding material to the particles composing the bulk of the stone. He quotes analyses of sandstones from the same source, some of which had formed part of buildings centuries old, while others had been recently quarried, and claims that he has traced a progressive alteration, which he ascribes chiefly to the action of alkaline sulphates upon the alumina, lime, and magnesia present in the stone, resulting in the formation of soluble sulphates of these bases, their removal from the stone, and its consequent deterioration. He advocates the

comparison of the behaviour of samples of sandstone when used for building purposes, with their composition as determined by analysis, in order to accumulate data for ascribing to each constituent its true influence on the quality of the stone.—B. B.

*On the Manufacture of Hydraulic Mortar in France and Germany.* H. Hanenschild, Thonind. Zeit. 1893, **17**, 418.

THE burnt material is spread in layers of 15 cm. thickness and moistened with water, then another layer is placed on top of this and again moistened. This is repeated until the total thickness attains 2 metres. The slaked mass is then sieved through revolving sieves having 600 meshes per sq. cm., the residue is worked up as Portland cement. These cements harden very slowly on account of the absence of alumina. The tensile strength of a mixture of one part of this cement with three parts of sand after seven days is only 3.3 kilos., after 28 days about 6 kilos., after five years from 25 to 40 kilos. The burnt material contains:— $\text{SiO}_2$ , 23.6;  $\text{Al}_2\text{O}_3$ , 1.4;  $\text{Fe}_2\text{O}_3$ , 0.8;  $\text{CaO}$ , 64.7;  $\text{MgO}$ , 1.4;  $\text{SO}_3$ , 0.5;  $\text{H}_2\text{O}$ , 7.6 per cent.

This product is very finely ground, leaving per sq. cm. on the 900 mesh sieve about 5 per cent., on the 5,000 mesh sieve about 27 per cent. of residue.—C. O. W.

### PATENTS.

*Improvements in the Manufacture of Artificial Building Materials.* F. W. Lübrmann, Düsseldorf. Eng. Pat. 20,004, December 1, 1892.

See under X., page 42.

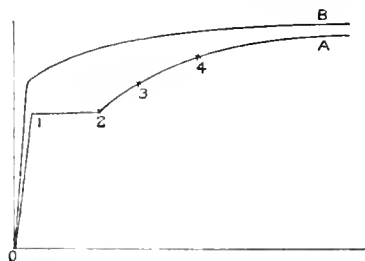
*Improvements in and in the Method of Making Glazed Bricks, Tiles, and the like, and Glazing and Embellishing Walls and other Surfaces.* J. W. Martin, London. Eng. Pat. 19,797, October 20, 1893.

IN this invention the brick or tile is covered with cement, coloured or ornamented with pigments, and glass, roughened on one or both surfaces, is applied to the cement. The coloured cement is visible through the glass, and the effect of coloured marble or stone is thus imitated.—V. C.

## X.—METALLURGY.

*Changes produced in Iron by Permanent Deformation in the Cold.* G. Charpy. Comptes rend. **117**, 850—853.

WHEN a bar of iron is subjected to tension, and plottings made of the increments in length and tensile strain, it is found that a regular curve, analogous to that marked B in the figure, usually results; but with annealed iron and



steel the curve exhibits a zigzag like A. Iodide of mercury subjected to compression exhibits an analogous curve, as the yellow modification passes into the red form, when a certain pressure is reached (Mallard and Le Chatelier); hence it is probable that in the case of iron and steel an analogous molecular transformation takes



place. That this is so is now shown by studying the amount of residual magnetism left after 24 hours' standing after magnetising in bars previously subjected to degrees of tension corresponding with the points marked 1, 2, 3, and 4 respectively.

The figures obtained with bars of soft iron, extra soft steel (0·12 per cent. carbon), and semi-hard steel (0·5 per cent. of carbon) show that the magnetism varies much when the tensile strain corresponds with the rectilinear part of the curve, but is sensibly constant when this region is passed. Hence the effect of permanent deformation in the cold may be regarded as the developing of an allotropic transformation in iron and steel, such as the conversion of  $\alpha$ -iron into  $\beta$ -iron.—C. R. A. W.

#### *Zinc Smelting at the Bertha Works, Virginia.* E. C. Moxham. Eng. and Min. J. 56, 544.

THE smelting is carried out in 10 large Welsh-Belgian furnaces with 140 retorts in each. The process is conducted in the ordinary manner, the long-flame Pocahontas coal being used to fire the furnaces, while a semi-anthracitic coal is employed to mix with the ore in the retorts. The latter are made of selected fireclay, which must be capable of making a tenacious paste, can be worked without cracking, and must be fireproof, so that the retorts will not break or weaken when resting on their ends in the furnace at a white heat. The following are the analyses of two good clays, the first one being slightly the better because it is more elastic and will bend slightly before breaking, at a high temperature:—

	No. 1, St. Louis.	No. 2, Perth Amboy.
	Per Cent.	Per Cent.
Silica (combined) .....	38·10	40·50
Alumina.....	31·53	35·90
Combined moisture .....	11·30	12·80
Titanic acid (TiO <sub>2</sub> ) .....	1·50	1·30
Free silica (quartz) .....	12·70	6·40
Potash (K <sub>2</sub> O).....	0·10	0·28
Soda (Na <sub>2</sub> O) .....	Nil	0·16
Iron oxide (Fe <sub>2</sub> O <sub>3</sub> ) .....	1·92	1·10
Hygroscopic moisture.....	2·50	1·50

—A. W.

#### *The F. L. Bartlett Zinc-Lead Process.* Abstract of report by E. W. Hawker. Eng. and Min. J. 56, 594.

THIS process, for the treatment of argentiferous zinc-lead sulphide ores, consists in separating the zinc and lead by volatilising them in the form of a zinc-lead fume, from which a marketable pigment is obtained, while the silver remains with the non-volatile metals, and is collected in a copper matte. Two methods of treating the ore are adopted, one for ores containing 25 per cent. of zinc and upwards, and the other for those containing 22 per cent. or less. The requisites for the process are (1) cheap flaming fuel, such as bituminous coal; (2) an iron ore, either oxide or pyrites, the latter being especially desirable if it contain silver; (3) a copper ore containing about 3 per cent. of copper for the formation of the matte to collect the silver.

In the first method the ore is crushed to pass a four-mesh screen, mixed with an equal bulk of fine coal, moistened, and charged in lots of 600 lb. into a furnace, of which the grate consists of perforated plates, the charge being spread on the grate to a depth of four inches. The burning is accomplished in about 4½ hours, by air being forced through the charge from below, and by a small quantity being also admitted above the charge, which is not touched during the operation. The sintered mass, containing the non-volatile metals, silver and some zinc, is then mixed with burnt

pyrites, copper ore, fluxes, and fuel in the requisite proportions, and smelted in a water-jacketed furnace of a greater proportional length than the ordinary blast furnace, when argentiferous copper matte is obtained, the bulk of the remaining zinc being volatilised.

In the second method the zinc ore is smelted directly, with the proper mixture of copper ore, fluxes, and fuel, in a special water-jacketed furnace with two rows of tuyers on each side, the upper ones being about 10 in. above the lower. The lower blast is preferably hot and the upper cold. The fuel consists of a mixture of coke and coal screenings, and is fed continuously with the ore in a layer from 12 to 18 in. deep. The lower blast plays upon the molten matter, scorifying it and volatilising all the lead and most of the zinc in the form of fume, which passes off through the unmelted portion of the charge, whilst the upper blast serves to keep up the necessary combustion and prevent the condensation of the volatile bodies. The argentiferous copper matte is tapped with the slag. The latter contains 6 to 10 per cent. of zinc, no lead, trace of copper, and about 1 oz. of silver per ton of ore treated. The matte contains 65 per cent. of copper and 250 oz. of silver per ton.

The fumes from all the furnaces are drawn into chambers by exhaust fans and forced through cooling conduits into long bags, through which the gases pass, leaving the solid contents of the fume behind. This is then refined by subjecting it to a low red heat and constant stirring, when all deleterious volatile matter is removed and a white pigment obtained containing 35 to 40 per cent. of oxy-sulphate of lead and 55 to 60 per cent. of zinc oxide.

In some ores there is a heavy loss of silver, in others very little, those containing copper or iron pyrites losing the least. Generally the salvage is between 70 and 85 per cent., but as much as 95 per cent. has been recovered. The loss should be confined to that in the pigment and that in the slags, but there is a further loss somewhere which at present is inexplicable.—A. W.

#### *Modern Processes for the Manufacture of Aluminium.* A. H. Bucherer. Zeits. ang. Chem. 1893, 17, 515.

See under XI., page 42.

#### PATENTS.

*Improvements in Effecting the Protection of Iron and Steel Surfaces, and in the Furnaces employed therein.* F. S. Barff, Kilburn, and G. Bower and A. S. Bower, St. Neots. Eng. Pat. 3304, July 28, 1881. (Reprint.)

A CHAMBER of cast or wrought iron, or other suitable material, and protected by a fire-brick casing, is heated externally by gas. The products of combustion pass around the chamber, and the waste heat is utilised for generating and superheating steam. The articles of iron or steel to be treated are placed inside the chamber and treated with this superheated steam, or, if desired, by carbonic acid, carbonic oxide, petroleum, or other hydrocarbon, while the chamber is maintained at a high temperature so as to produce a protective surface of magnetic oxide on the articles.—J. H. C.

*Improvements in and relating to the Preparation of Reagents for Working Gold and Silver Ores.* H. H. Lake, London. From Eloy Noriega, Mexico. Eng. Pat. 17,661, October 4, 1892.

THE reagent consists of a concentrated solution of protochloride of copper, or of the double protochloride of iron (ferrous chloride) and copper, prepared by submitting the ingredients, viz., sulphate of copper, chloride of sodium, sulphuric acid, and water, to the action of steam and using metallic copper to neutralise the excess of acid. Ferrous chloride prepared in a similar manner may be also used with or without the addition of cyanide of copper.—J. H. C.

*Improvements in the Manufacture of Artificial Building Materials.* F. W. Lüthmann, Düsseldorf. Eng. Pat. 20,004, December 1, 1892.

BLAST-furnace slags are re-heated in a reverberatory furnace with suitable additions, of limestone, dolomite, clay, quartz, oxide of iron, &c., or with other kinds of slag. The mass is then removed from the furnace and allowed to cool. In most cases, in carrying out the process, it is advantageous to add to the fused mixture a few per cents. of oxide of iron in any convenient form. Data and a formula are given for calculating the proportions of lime and slag that should be used.—J. H. C.

*Improved Application of Aluminium and its Alloys.* W. R. Taylor, Rochester. Eng. Pat. 22,843, December 12, 1892.

THIS invention consists in the application of aluminium or its alloys to the manufacture of brewers' casks, jugs, earboys, water tanks, mineral water bottles and siphons, culinary utensils, milk cans, butter firkins, fermenting and evaporating pans, cocks and taps, pumps and bar fittings, &c. In the case of large vessels, the metal sheet is strengthened by hoops or wooden staves or by applying a layer of wood pulp or other suitable material in a mould.—W. J. P.

*Improvements in the Manufacture of Steel or Wrought-Iron.* P. M. Justice. From the Société anonyme des hauts fourneaux et forges de Dudelange, Grand Duché de Luxembourg. Eng. Pat. 23,286, December 17, 1892.

IN the manufacture of steel and ingot-iron, the metal is carburized in the casting ladle by means of briquettes made of carbon with or without the addition of lime (Eng. Pat. 17,613, 1890, this Journal 1891, 262). The patentee claims protection for the use of such materials in a pulverised form instead of in briquettes. The carburization may be performed by addition of the mixture to the molten metal whilst in the Bessemer converter, ingot moulds, or whilst running into the moulds or furnaces.—W. J. P.

*Improvements in the Purification of Iron.* E. H. Saniter, Wigan, Lancashire. Eng. Pat. 23,534, December 21, 1892.

THIS patent deals with improvements on the processes described in (Eng. Pat. 8612, 8612A; this Journal 1892, 1013 and 1014), and 17,692 of 1892 (this Journal 1893, 844). Mixtures of calcium chloride and fluoride with lime or other oxide, a hydrate or carbonate of an alkaline earth are placed in the casting ladle or other receiver or furnace and the molten iron run on to it with the object of purifying the latter metal. Other chlorides or sodium borate may be used if found desirable. If a large ladle be employed the mixture is best confined in a well at the bottom so that it shall rise slowly through the molten metal.—W. J. P.

*Improvements in the Recovery of Iron from the Ferruginous Residue left in the Production of Amine Bases.* S. S. Bromhead. From Landshoff and Meyer, Grünau, Germany. Eng. Pat. 23,639, December 22, 1892.

THE ferruginous residue obtained in the commercial production of amine bases is heated in a muffle and treated with steam at a red heat. On increasing the temperature to near the melting point (900° C.) the iron is partially reduced by the carbonaceous substances contained in the residue; the reduction is completed by covering the mass with charcoal, coke, or pitchy residues, which do not contain sulphur or by passing carbonic oxide over it. The cooled reduced metal is then sieved and again employed for reduction.—W. J. P.

*A Process for the Elimination of Sulphur from Molten Iron or Steel.* H. J. Phillips, Ebbw Vale. Eng. Pat. 1437, January 23, 1893.

THE molten metal whilst contained in a converter is mixed with blast-furnace slags or manganiferous slags of all kinds, glass, potassium, sodium, calcium, or barium silicates, silicates of alumina or iron, and all other silicates, individually or severally, mixed with basic materials, forming a highly basic slag, which will melt, thereby increasing the chemical affinity between the base and the sulphur contained in the iron; lime, potash, oxide of iron, dolomite, limestone, baryta, soda, alumina, or any cheap base may be used; fluor-spar, rock-salt, or powdered coke may also be added at times with advantage. After puddling or other mixing the iron is run off, the slag being retained by means of dams.—J. H. C.

*Improvements in or relating to the Tinning of Articles made of Cast Iron.* A. J. Boulton, London. From Messrs. Wolf, Netter, and Jacobi, Berlin. Eng. Pat. 2349, February 2, 1893.

THE cast-iron is coated electrolytically with iron and nickel or iron and cobalt, before the coating of molten tin is applied in the ordinary way.—J. H. C.

*Improvements in or relating to the Conversion or Purification of Iron and Steel.* A. J. Boulton. From W. A. Koueman, C. F. Singer, and A. F. Hatch, Chicago, U.S.A. Eng. Pat. 12,588, June 27, 1893.

THE inventors employ a tall fire-brick shell some 30 feet or more in height for the smelting of metallic ores. This furnace is fed with coal and some suitable flux through a hopper at the top, so that the fuel at the upper part of the firebed undergoes distillation; the gas thus formed is conducted off and led again into the furnace below the top layer of fuel, from which it distilled. Here the gas comes in contact with the molten metallic ores or slags which have been charged into the furnace with the fuel, and have sunk down as the fuel burnt, until reaching a portion of the furnace at which an air-blast enters, they melt and percolate through the underlying incandescent fuel; the metal is reduced by the gas and passes out at the lower part of the furnace. Metallic ores or slags may be treated by this process, and scrap iron may be converted into a highly carburized iron.—W. J. P.

*An Improved Process for Producing on Steel, Iron, or other Metals a Durable and Smooth, either Glazed or Dull, Black Surface.* W. T. Thompson, Liverpool. From W. Kiesel, Ludwigsbürg, Germany. Eng. Pat. 20,024, October 24, 1893.

THE material is pressed or worked in the cold into the desired shape, smoothed and coated with balsam of sulphur after it has been heated and boiled in hydrochloric acid. The heating and immersion may have to be repeated at various stages of the work.—J. H. C.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

*Modern Processes for the Manufacture of Aluminium.* A. H. Bucherer. Zeits. ang. Chem. 1893, 17, 515—517.

THOSE methods which consist essentially in the electrolysis of alumina dissolved in fused cryolite, have notable advantages over such as depend on the principles formerly in vogue. The plan in question originated in the laboratory researches of Deville, who in 1859 described a process by which aluminium was prepared by the electrolysis of fused

eryolite, the composition of the bath being maintained by the dissolution of anodes of carbon containing alumina. It is probable that Deville was led to try this method by analogy with his successful process for the manufacture of aluminium by purely chemical means. Just as by the action of chlorine on a heated mixture of alumina and carbon, aluminium chloride is formed, so it might be expected that aluminium fluoride would be produced by the action of fluorine set free by the electrolysis of eryolite upon the mixture of alumina and carbon constituting the anode. In this early process the heat requisite for maintaining the fluidity of the bath was supplied from without. The reasons for the non-utilisation of this method until recent times, are to be found in the facts that an externally heated vessel is soon destroyed under the prevailing conditions; that the content of the bath in alumina cannot be maintained with a difficultly soluble anode of high resistance, such as one composed of alumina and carbon; and that the cost of current was formerly too high. In modern practice all these drawbacks are overcome by the use of pure carbon anodes, by feeding alumina direct to the bath, by internal heating due to the electrolytic process itself, and by the use of cheap current.

Inasmuch as the output of aluminium for a given consumption of energy is dependent on the reactions taking place in the electrolytic bath, the consideration of the nature of these reactions is of primary importance. In processes in which alumina is fed into a bath of eryolite or similar halogen salt, it has been asserted that the main action of the current consists in the electrolysis of the alumina. The author, however, considers that, taking into account the calculated E.M.F. corresponding with the heats of formation of the oxides and fluorides of sodium and aluminium, and also adopting the modern hypothesis of the dissociated condition of electrolytes, the fundamental reaction is the electrolysis of sodium oxide, followed by the liberation of aluminium from its fluoride by the action of the sodium already produced. In support of this view he adduces the fact that when a bath of pure eryolite is electrolysed, on reaching a certain current density sodium is set free, because the quantity of aluminium fluoride is insufficient to react at once with the alkali metal. On increasing the proportion of aluminium salt, however, almost the theoretical amount of aluminium for the current passing, is obtained.

In the Minet process the use of a bath of aluminium fluoride and sodium chloride in place of one of eryolite is claimed. The author finds by experiment that aluminium fluoride is not soluble in sodium chloride (both salts in the fused state), and that the first effect of the electrolysis of such a mixture consists in the production of chlorine and sodium, the latter acting on the aluminium fluoride and liberating aluminium. The ultimate result is the formation of a eryolite bath, prepared in a manner far from economical. When once the eryolite bath has been prepared, the addition of alumina transforms the sequence of reactions into that proper to the Hall or Héroult process.—B. B.

*Action of Certain Metals on Acid Solutions of their Chlorides.* A. Ditte and R. Metzner. *Comptes rend.* 1893, 117, 691—694.

THE author has studied the well-known phenomenon of the formation of crystals of metallic tin on the upper part of a rod of the metal dipping in a solution of stannous chloride, made acid with hydrochloric acid, upon which rests a layer of water. The formation of the crystals does not take place in a homogeneous solution of stannous chloride, and the deposit is most abundant at the plane of junction of the liquids, decreasing with the distance therefrom; as the distance increases the character of the deposit changes, the tin becoming grey and spongy, and finally tailing off to a black powder. The usual explanation is that the phenomenon is one of electrolysis, and this is borne out by the possibility of replacing the tin above the plane of junction of the liquids with a rod of platinum, upon which crystallisation takes place as readily as upon the tin. Moreover, crystallisation can be induced at any predetermined spot on the upper part of the rod by making the liquid at that spot a better conductor by introducing a little stannous chloride

at the point selected. Another indication in the same sense is afforded by the observation that when two bars of tin are placed one above the other without contact, the production of crystals on the upper bar takes place as soon as the progress of diffusion has transferred sufficient tin salt to the upper layer of liquid. The solution of a portion of the tin is of course a necessary preliminary to its deposition elsewhere, whence it follows that no crystallisation occurs when the lower part of the bar is immersed in a neutral solution of stannous chloride.

The deposition of crystals of cadmium can be effected in a similar manner, but in the case of a metal like zinc, easily soluble even in dilute hydrochloric acid, the formation of crystals is not observed. Nickel fails to yield a deposit, this being attributed by the author to the ease with which the metal is covered by a film of hydrogen, which cuts it off from contact with the acid liquid. Metals like bismuth and antimony, which are not appreciably attacked by dilute hydrochloric acid, give negative results.—B. B.

*The Influence of Frictional Electricity upon the Formation of an Amalgam.* G. Staats. *Ber.* 1893, 26, 1796—1797.

IN order to ascertain the influence of electricity of high tension upon the process of formation of an amalgam, a small Leyden jar was used, covered on the outside with tinfoil, and containing mercury to the same height as the outer covering, and having a steel wire terminating in a small silver disc as the means of connection between the charging knob and the mercury in the interior, and the weight of this silver disc, before and after the experiment, was determined. In a second series of experiments, the steel needle with its disc was exposed to a stream of sparks from the conductor of an influence machine for 60 seconds, and then dipped in mercury for a few seconds, and weighed after removal. The mean result of the experiments showed that 23.07 per cent. of amalgam was formed without the use of electricity, and 72.09 per cent. when electricity was employed. In all cases superfluous mercury was shaken off the amalgamated disc before weighing.

—B. B.

*Progress in Electrometallurgy.* *Dingl. Polyt. J.* 1893, 289, 129—135.

*Aluminium.*—One of the most noteworthy features in the recent improvements in the production of aluminium, is the increased purity of the metal, which is said to be procurable with as little as 0.1 per cent. of foreign matter, though trustworthy analyses show 0.8 per cent. to 1.0 per cent. as a more usual amount in the best samples. An indication of the rise in the rate of production of late years is afforded by the increase in the output of the works at Neuhausen, which amounted to 40,538 kilos. in 1890, 168,669 kilos. in 1891, estimated at 300,000 kilos. for 1892, and is expected to reach 1,000,000 kilos. in 1893.

The best methods at present in use for the manufacture of aluminium consist in the electrolysis of a fused electrolyte, e.g. alumina, older processes necessitating the intermediate manufacture of sodium, having been exposed thereby to severe competition, which has caused many efforts to be made to lower the cost of production of the alkali metal (*cf.* this Journal, 1893; 1887, 174). The preparation of the alumina which is used in the Héroult furnace is effected by fusing bauxite with sodium carbonate, extracting the sodium aluminate formed, and precipitating with carbon dioxide, by which means it is obtained in a state of sufficient purity for the purpose in hand. According to Grabau's patent a mixture of aluminium fluoride and sodium carbonate is used as the electrolyte, sodium fluoride and ultimately eryolite being formed as the process progresses. An oxyfluoride, to which the formula  $Al_2O_4F_2$  is assigned, can be substituted for aluminium fluoride. Equations representing the changes said to occur during electrolysis, are quoted.

Bueherer's patent relates to the use of double sulphides of aluminium and the alkalis or alkaline earths, dissolved

in a bath of fused alkaline chlorides and electrolysed (cf. this Journal, 1893, 272). A similar process has been protected by the Aluminium Co. of Neuhausen. The following advantages are claimed for processes of this type:—

(1.) The carbon anode is not attacked, as sulphur is eliminated thereat instead of oxygen, and the temperature is too low to permit of the formation of carbon bisulphide.

(2.) The production of aluminium requires a comparatively low voltage (2·5—3 volts).

(3.) Short-circuiting is avoided, as the aluminium is of higher specific gravity than the bath, and sinks to the bottom of the vessel.

(4.) The sulphur appearing as vapour at the anode can be recovered and utilised.

**Tungsten.**—A method for the preparation of tungsten has been patented by Krieg. It consists essentially in preparing the chloride, transforming this into tungstic acid, and reducing the latter to metal, the first and last operations being performed by electrical means. The preparation of the chloride is effected by using electrodes composed of a mixture of scheelite or wolfram and carbon for the production of an electric arc in the presence of chlorine, the chlorides of the metals present in the ore being thus obtained as a sublimate. Tungstic acid is separated from the product by treatment with hydrochloric acid, by which means tungsten chloride (and silicon chloride) is decomposed and ferric chloride, aluminium chloride, &c., are dissolved. The tungstic acid is freed from gelatinous silica by elutriation, made into electrodes with carbon, and reduced to metal in a closed vessel.—B. B.

*Electrolytic Estimation and Separation.* G. Vortmann. Monatsh. f. Chemie, **14**, 1893, 536.

See under XXIII., page 68.

*Electrical Transport of Heat in Electrolytes.* H. Bagard. Comptes rend. **117**, 97.

See under Analyt. and Scient. Notes, page 71.

## PATENTS.

*Improvements in the Treatment of Ores and Recovery of the Metals contained therein, and Appliances therefor.* F. G. Fuller, London. Eng. Pat. 20,025, November 7, 1892.

FREE-milling and some refractory ores after proper crushing, and sometimes after concentration, are placed in an amalgamating trough, the ends of which are made of conducting material, whilst the sides and bottom are of insulating material. A current of electricity is made to pass through the ore which is caused to move forward between a series of electrically excited amalgamating plates and in contact with a sufficient quantity of mercury or other suitable chemical reagents, such as a chloride of the alkalis, nitric acid, nitric acid vapour and water, sulphuric acid, a bi- or persulphate, &c., together or separately, and heated or not as required. The amalgam is collected, or the solutions produced are drawn off for separate treatment, by chemical re-agents, electricity, or other suitable methods.

In treating refractory ores a series of stationary or revolving "furnaces" is employed. The ores properly prepared by crushing or by a previous roasting are placed in these "furnaces" and heated from hot-air generators connected by means of suitable flues and controlled by dampers. Steam is also supplied, or acid fumes from the roasting of pyrites, the distillation of nitrates or other convenient means. The ores so treated are then leached with water or chemical solutions and the liquors so obtained are treated by any suitable methods for the separation of their contained metals.—J. H. C.

*Improvements in the Preparation of Chromic Acid and in the Extraction of Pure Acids from Alkaline Salts.* E. Placet and J. Bounet, Paris. Eng. Pat. 22,819, December 12, 1892.

See under VII., page 37.

*An Improvement in Plates for Secondary Batteries.* A. Hough, Forest Hill, Surrey. Eng. Pat. 23,750, December 23, 1892.

According to this invention the plates for secondary batteries are constructed as follows:—A good conducting wire, preferably copper, is coated with lead by drawing it through a bath of lead which is not in a fluid condition, but nearly at its setting point. The wire thus coated is drawn down to a suitable gauge, and formed into a gauze network, to constitute the frame of a plate. The meshes of this frame are filled in and covered with lead oxide, preferably a mixture of coarse-flake and finely-ground litharge, applied in a dry state. The plate is then placed between sheets of felt, or the like, moistened with dilute sulphuric acid, and after about six hours it is removed from the felt and left to dry and set for about 12 hours. The litharge is then reduced to spongy lead in the usual manner.—G. H. R.

*Improvements in Apparatus for the Making or Manufacture of Caustic Soda or Caustic Potash, being a Method of passing the Chlorine and Sodium Gases to the Receiver.* F. W. Petrie, Rochdale. Eng. Pat. 472, January 10, 1893.

See under VII., page 38.

*Improvements in Platinised Electrodes.* H. T. Barnett, London. Eng. Pat. 757, January 13, 1893.

SILVER, carbon, or any other suitable material may be used to construct the electrodes of, and a convenient method of manufacture by which the conducting support is a compound, solid, cylindrical, and textile mass, is as follows:—Electric light carbon rods are pasted and dried, and lightly bound with freshly-pasted strips of cotton velvet that have been previously pasted and dried. They are dried slowly in an air-current, and when on the pile are sprayed with chloride of platinum solution, after which they are quickly dried in an oven, and then carbonised, while packed in charcoal dust, in a gas or other muffle furnace.—G. H. R.

*Improvements in connection with Carbon Electrodes.* H. T. Barnett, London. Eng. Pat. 758, January 13, 1893.

This invention consists of the combination of an electrode formed of a number of fine carbon projections outstanding from a carbonaceous or metallic support, and one or more guarding portions attached to, or formed of the substance of the conducting support. The outer surface of the guarding portions is coated with an electricity insulating cement, enamel, or other suitable material.—G. H. R.

*Improvements in or connected with the Manufacture of Electrodes for Electric Accumulators.* C. Pollak, Frankfort-on-Main, Germany. Eng. Pat. 813, January 13, 1893.

THE object of this invention is to effect the more rapid formation of electrodes of the so-called Planté type, by using porous compressed lead obtained by a wet process, by stirring carbonate of lead into a paste with caustic alkali, then reducing this paste (after imparting the necessary shape to it by means of suitable supports, if necessary, and it has become dry) in an alkaline solution by electrolysis, and finally compressing the porous lead thus formed, to a greater or less degree, immediately after the reducing process, and whilst it is still wet.—G. H. R.

*Improvements in Electrolytic Apparatus.* C. Hanbury, London. Eng. Pat. 8176, April 22, 1893.

THIS invention relates to improvements in the construction of electrolytic tanks, and consists in fixing near the opposite ends, or sides, partitions which cut off two narrow portions forming transverse chambers. Each of these chambers is divided into two by a horizontal watertight partition. Between these chambers, at the opposite ends or sides of the tank, and at right angles to them, porous diaphragms, each with an anode and cathode, are fixed so as to form watertight compartments, which contain alternately (except in the first and last) two positive and two negative electrodes. In the space corresponding to every other compartment, a free aperture is made near the top of the partition into the upper transverse chamber at one end of the tank, the lower edge of which aperture dips just below the intended level of the electrolyte. The partition at the other side is similarly perforated near the top in the spaces corresponding to the other alternate compartments. Free openings are made at the bottom of each transverse partition between alternate compartments and the lower transverse chamber, these openings alternating with those in the upper series. The cover of the tank fits closely, and provision is made for drawing off the gas evolved, and for the circulation of the electrolyte.—G. H. R.

*Improvements in Electro-plating.* E. de Pass, London. From H. Thofehn, Paris, France. Eng. Pat. 10,095, May 20, 1893.

ACCORDING to this invention, in order to obtain a smooth deposit of metal upon a cylinder, or a moulded or shaped article, rotating in an electrolytic bath, the surfaces to be plated are showered with a stream of jets, under pressure, of the electrolyte, which acts either continuously or intermittently during the whole of the electrolytic operation. The showering may be effected by means of perforated or slotted tubes or discharge pipes, which may advantageously receive a reciprocating motion. To prevent the electrolyte becoming disturbed during the action of the current, or jets, directed on to the cathodes, the anodes are placed at a suitable distance from the bottom of the tank. The electrolyte is drawn off from the bottom of the tank, and delivered into a reservoir, wherein it is cleansed from impurities, and afterwards restored to its standard strength, and is then again showered on to the cathodes, a constant circulation of the electrolyte being thus kept up.—G. H. R.

*Improvements in Secondary Voltaic Batteries.* E. J. Chubbe and A. W. Southey, London. Eng. Pat. 10,765, June 1, 1893.

ACCORDING to this invention the negative element consists of a pasty mass of peroxide of lead resting on the bottom of a cell or container, of dish or tray form, which is provided with a conducting plate of lead or an alloy of that metal, coated with gold or platinum to prevent oxidation, and embedded in and wholly covered by the pasty mass, except a narrow strip which serves as a contact piece, and is bent so as to pass out of the cell.—G. H. R.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*Macassar Oil.* R. Glenk. Amer. Journ. Pharm. 65, 528.

A SPECIMEN of this oil, obtained from the seeds of *Schleichera Trijuga*, Willd (Sapindaceæ), and sent from Mirzapoor, Hindostan, has been examined by R. Glenk, who describes it as a yellowish-white semi-solid substance, having a faint odour of bitter almonds, and a specific gravity of 0.942. It probably contains hydrocyanic acid, and its

mildly acid taste may be ascribed to partial rancidity. An acid reaction was indicated with litmus paper, and the oil was completely liquefied at 28° C., congealing again at 10°. It was readily saponified by sodium hydrate, even at a low temperature, forming a white, hard soap. With nitrous acid it assumed an orange-red colour, and became viscid, but did not appear to solidify. Concentrated sulphuric acid acquired a reddish-brown colour on addition of the oil. Chloroform, ether, carbon bisulphide, benzene, benzine, and the fixed and volatile oils freely dissolve the oil, but alcohol only slightly affects it.

*Detection of Paraffin and "Lubricating Oil" in the Distillate obtained under Pressure from Tallow Oil.* C. Engler and L. Singer. Ber. 1893, 26, 1449—1451.

THE fact that neither paraffin nor "lubricating oil" was detected in the distillates obtained under pressure from natural glycerides and from the higher fatty acids, and which were examined in the author's laboratory, has been frequently urged against the theory of the formation of petroleum from the remains of animal fat. The authors have in consequence again taken up the subject with special reference to the detection of the above-named substances, and they have succeeded in separating solid paraffin and in obtaining a fluorescent and viscous "lubricating oil." The paraffin was obtained in the form of a white crystalline mass melting at 49—51°.—A. K. M.

*Adulteration of Olive Oil.* V. Oliveri, Le Staz. speriment. agr. ital. 1893, 24, 387.

THE refraction coefficient, saponification number, and iodine absorption of 106 pure olive oils were ascertained and compared with those of other oils. The following are the results:—

	Refraction Coefficient.		Saponification Number.		Iodine Absorption.	
	Mini-mum.	Maxi-mum.	Mini-mum.	Maxi-mum.	Mini-mum.	Maxi-mum.
Olive oil .....	0	2	190.5	195	79	82.2
Cotton-seed oil .....	18		191	195	104	108
Sesame oil .....	15.5		189	190.5	105	107
Colza oil .....	26.5		177	178	99	100
Peanut oil .....	7.5		191	196	101	105
Poppy-seed oil .....	28.5		192	195	133	138

THIS shows that by means of the oleo-refractometer, admixtures of not too small quantities of any of the above oils with olive oil can be detected, as the refraction coefficient would be above 2. Peanut oil is, however, an exception, since a mixture of 25 per cent. of this and 75 per cent. of olive oil with a refraction coefficient 0.25 would give a mixture the refraction coefficient of which would not be above 2. But even in this case the high iodine absorption would betray the adulteration.—C. O. W.

*India-rubber Substitutes.* R. Henriques. Chem. Zeit. 1893, 17, 634.

See under XIII., page 47.

*The Analysis of Bees' Wax.* G. Buchner. Chem. Zeit. 1893, 17, 918.

See under XXIII., page 70.

*The Use of Eurfural for the Detection of Sesame Oil in Oil Mixtures.* V. Villavecchia and G. Fabris. Zeits. ang. Chem. 1893, **17**, 505.

See under XXIII., page 69.

*Detection of Nitronaphthalene in Mineral Oils.* Norman Leonard. Chem. News, **68**, 297.

See under XXIII., page 69.

### PATENTS.

*An Improved Process for Treating and Purifying Sod and other Oils, and Apparatus therefor.* F. N. Turney, Nottingham. Eng. Pat. 1319, January 21, 1893.

For the purification of sod oil (the refuse oil after treatment of chamois leather with cod and other oils) containing water, colouring matter, and impurities derived from the skins, the oil is agitated with a solvent, such as petroleum spirit or carbon bisulphide, and the mixture is allowed to stand. The water and impurities then settle out. The supernatant mixture of oil and solvent is drawn off and distilled, and the solvent condensed for use over again, leaving the purified oil in the still. This oil is useful for currying purposes, and is said not to colour or stain the goods. The process is applicable to the purification of lubricating and other oils.—J. J. K.

*Improvement in the Manufacture of Emulsions.* H. Helbing, London. Eng. Pat. 8196, April 24, 1893.

DEXTRIN is the emulsifying agent employed for oils and liquids which will not mix with water. It is made into a thin paste with water, compounded with the oil, and vinegar is then stirred in to form a homogeneous mass; or the vinegar may be first mixed with the dextrin and the oil added afterwards. Medicinal and flavouring preparations may be mixed with the emulsion.—J. J. K.

*Improved Method of Separating Fats.* M. Ekenberg, Stockholm, Sweden. Eng. Pat. 17,565, September 18, 1893.

By the process claimed, the separation is effected of three different fats and cholesterol contained in neutral wool fat, the melting point of which varies between 38° and 46° C., being generally above 40° C. Wool-fat is melted, cooled slowly to 35° C. in moulds to form thin tablets or cakes, and pressed in hot presses or centrifugal machines; all molten fat at this temperature flows out. The same operation is then repeated at 45° C. The products obtained are, at 35° C. a fat containing about 60 per cent. of cholein melting at 25°–29° C.; at 45° C. a fat containing about 60 per cent. of cholepaluin, melting at 37°–38° C.; the residue in the press consisting of a mixture of cholecerin and cholesterolin, containing 60 per cent. of cholecerin, melting at 49°–55° C. Solvents, such as volatile hydrocarbons, fat oils, and fatty acids, added to the wool-fat are said to facilitate the production of the three fats, and may be separated afterwards; that of the fat oils and acids by saponification with alkali. The cholein may be used for lubricating, the cholepaluin in the manufacture of soap and candles, and the cholecerin as a substitute for beeswax.

—J. J. K.

*An Improved Fat Emulsion and Method of Producing the same.* M. Ekenberg, Stockholm, Sweden. Eng. Pat. 17,566, September 18, 1893.

This relates to the production of an emulsion from cholein, a fat obtained by means described in the preceding patent, No. 17,565, which has the property of forming emulsions with water containing 30 per cent. or more of fat. The melted fat, heated to about 40° C. in a centrifugal mixing apparatus, is mixed with a small quantity of water, allowed

to stand for some hours to lower its temperature to 25°–30° C., and mixed with a quantity of water sufficient to obtain the desired proportion of fat and water. This emulsion may be used as a pomade, as a means of softening leather, or to preserve raw hides.—J. J. K.

*Improved Apparatus for and Process of Making Soap.* M. J. Palmer, Toledo, Ohio, U.S.A. Eng. Pat. 19,298, October 13, 1893.

The saponification is effected in a vacuum chamber, heated by steam, assisted by a revolving agitator, which intimately commingles the fatty matter and alkali. The product is then ejected into a vat and subjected to the action of the electrical current, which disintegrates the fatty globules therein, and effects a complete and rapid saponification, obviating the necessity of time and storage, for ageing the soap.—J. J. K.

## XIII.—PAINTS, PIGMENTS, RESINS, VARNISHES, INDIA-RUBBER, Etc.

### (A.)—PAINTS, PIGMENTS.

*Antimony Blue.* G. Sebor. Casopis pro prumysl Chemicky, 1893, 216.

THE author attempted to find a practically workable method for the preparation of this blue, which was discovered by Boettger in 1871. Sulphide of antimony is dissolved in concentrated hydrochloric acid, and the solution filtered through asbestos. The moderately concentrated solution is heated to the boiling point and mixed with a concentrated solution of potassium ferrocyanide. A small quantity of potassium chlorate or nitric acid is then added and the whole well boiled. The mass is then filtered and washed, and subsequently dried. A very deep fine blue pigment is obtained in this manner. Another blue is obtained by mixing the solutions of antimony trichloride and potassium ferrocyanide without regard to their concentration, and subsequently adding a large excess of water. This yields a fine pale blue. An antimony blue, as pure as it could be obtained, gave the following analytical numbers: Fe, 30.28 per cent.; Sb, 2.422 per cent.; H<sub>2</sub>O, 5.828 per cent.; Cl, 0.712 per cent.; O, 0.323 per cent.; CN, 60.435 per cent. This blue behaves towards acids like ordinary Prussian blue; it is not affected by cold solutions of the caustic alkalis or of ammonia, but by using hot solutions, it is destroyed. It differs from Prussian blue in being insoluble in a solution of ammonium oxalate or tartrate.—C. O. W.

### PATENT.

*A New or Improved Protective Paint.* T. Cohn, Berlin. Eng. Pat. 19,444, October 16, 1893.

This paint consists mainly of oxide of lead, common salt, and a soluble salt of a suitable heavy metal, the whole being stirred up with a little water, washed, and finally ground with oil or varnish. "The oxide of lead combines with the common salt and forms chloride of lead and hydrate of soda. The latter precipitates the oxides or the hydrates of the heavy metallic salts, and thereby forms, with the fatty acids contained in the oils or varnish, saponaceous products. The latter become dissolved in the free oil, and give the paint its elasticity." Convenient proportions are 10 parts of oxide of lead, 2 parts of common salt, enough water to form a thin paste, and 5 parts of chloride or sulphate of zinc. To produce various colours, salts of various other metals, such as copper, iron, &c., may be substituted for the zinc salt.—A. G. B.

## (B.)—RESINS, VARNISHES.

*Kauri Copal Digging in New Zealand.* Chem. Ind.  
1893, 16, 137—138.

Kauri copal is the resin of *Dammara Australis*, and the occurrence of this fir is limited to the province of Auckland, where it appears to have been growing for many thousand years. This resin is of very great importance for the manufacture of varnishes and lacquers, but up to a few years ago, only the fossilised resin was employed for this purpose, the fresh resin being almost valueless. In recent times this resin is also employed for certain purposes. The fossil resin is obtained by digging, mostly on land where no Kauri firs exist at present. The resin is frequently found in quantities of from 50 to 100 lb. together, sometimes even forming blocks weighing from 2 to 4 cwt. The first colonists in Auckland found this resin in enormous quantities when tilling the soil. Its value then being unknown it was burnt as fuel. The first shipment to London of 300 tons was made in 1815, and the resin being found unsaleable, it was sent to America, where its value for the manufacture of lacquers was discovered. Since then the exports have been steadily increasing, and amount to nearly 9,000 tons per annum at present. In some places the resin is found in beds 10 feet in thickness, and till lately the ploughing of native soil was frequently found to be a very remunerative operation on account of the resin brought up by the plough shares.—C. O. W.

*On Xanthorrhoea Resins.* M. Bamberger. Monatsh.  
Chem. 1893, 14, 333.

XANTHORRHOEA resins, which are known in commerce as "Akaroid resin," "Botany Bay resin," "Grass tree gum," "Nut resin," have been repeatedly investigated by Liechtenstein, Laugier, Trommsdorff, and Widmann. Steinhöuse discovered in the yellow resin cinnamic and benzoic acid, and by treating it with nitric acid he obtained large quantities of picric acid. Hlasiwetz and Barth obtained from the same resin, by fusing it with caustic potash, large quantities of para-hydroxybenzoic acid, resorcinol, and a double compound of protocatechuic acid with para-hydroxybenzoic acid and catechol.

*I. Yellow Xanthorrhoea Resin (Xanthorrhoea hastilis Sm.)*—The acid number, saponification number, and iodine number were determined by Schmidt and Erban's method. The figures given refer to resin purified with alcohol, unless stated differently. Acid number, 132—133; saponification number, 220—225; iodine number, 86; methyl number, crude resin, 27·66—28·97; purified resin, 34·73; crude resin (Amsterdam Exhibition), 27·29. Boiling water extracts from the yellow resin 10 per cent. of paraeumaric

acid and smaller quantities of parahydroxybenzaldehyde, a body resembling vanillin, and benzoic acid with 1 per cent. of cinnamic acid.

*II. Red Xanthorrhoea Resin (Xanthorrhoea Australis).*—The acid and saponification numbers of this resin cannot be estimated owing to the intense red colouration of its alcoholic solution. The iodine number of the purified resin was found to be 105·5. Methyl number, crude resin, 60·3—60·9; purified resin, 71·12. Carbonyl number, 0·07. Boiling water extracts from this resin about 2 per cent. of paraeumaric acid, a substance resembling vanillin, and para-hydroxybenzaldehyde.—C. O. W.

*Gardenia Resin.* E. Heckel and Schlagdenhaufen.

Répert. de Pharm. 1893, 49, 145.

THE leaf buds of the New Caledonian *Gardenia G. Oudup Vieil*, *G. Aubryi Vieil*, and *G. Sulcata Gaertn.* possess a protective coating of green resin which is chewed by the natives. Also when dissolved in brandy it is used for dressing wounds and ulcers on account of its antiseptic properties. The resin is, however, chiefly employed for caulking and making water-tight the canoes of the natives. It is insoluble in water, easily soluble in ether, acetone, alcohol, and chloroform. On evaporation, the ethereal solution leaves an opaque, yellow mass, which on exposure to the air hardens completely within three weeks. In its composition and reactions with metallic salts, the resin resembles the tannins. This resin is likely to become very useful both medicinally and as a basis for varnishes.—C. O. W.

## (C.)—INDIA-RUBBER, &amp;c.

*The Manufacture of India-Rubber Waterproof Cloth.* C. O. Weber. Zeits. ang. Chem. 1893, [21], 22.

See under V., page 33.

*India-Rubber Substitutes.* R. Henriques. Chem. Zeit.  
1893, 17, 634—638.

INDIA-RUBBER substitutes prepared by boiling fatty oils with sulphur or by treating them in the cold with chloride of sulphur are distinguished in the trade by the names "brown" (black) and "white substitute" respectively. These two products are entirely different in their composition, they possess a slightly penetrating, oil-like odour and are very insoluble in all the usual solvents. The "white substitute" is distinguished by the high proportion of chlorine it contains, and which can neither be removed by boiling with water nor with acids. This substitute is obtained by treating vegetable oils with chloride of sulphur. Analyses of a number of different substitutes of various descriptions gave the following results:—

	Commercial Products.						Linseed Oil Substitutes from		Colza Oil Substitutes from		Poppy Seed Oil Substitute, from Oxidised Oil.	Castor Oil Substitutes.		Substitute from Oxidised Cotton Seed Oil.
	White Substitute.			Brown Substitute.			Fresh Oil.	Oxidised Oil.	Fresh Oil.	Oxidised Oil.	Oxidised Oil.	With Minimum of $S_2Cl_2$	With Maximum of $S_2Cl_2$	
	A.	B.	C.	A.	B.									
Sulphur in the substitute .	6·4	6·17	8·25	15·48	17·71	9·34	0·78	8·28	6·59	7·68	4·82	10·6	6·23	
Chlorine " " .	5·0	5·86	8·88	0·7	0·36	8·84	4·85	7·62	5·95	7·44	6·70	8·95	5·96	
Water " " .	0·85	1·0	..	..	..	3·02	0·85	..	..	..	..	..	..	
Residue on ignition.....	6·8	5·51	..	..	..	..	..	..	..	..	..	..	..	
Fatty acids .....	30·15	73·58	..	..	..	79·6	81·67	86·89	87·95	74·90	87·95	..	..	
Sulphur in the fatty acids.	6·12	6·45	8·15	14·14	15·20	9·88	4·06	8·34	6·54	8·32	5·92	..	0·44	
Chlorine " " .	0·83	0·43	..	..	..	Trace	0·60	Trace	Trace	..	0·26	Trace	Trace	
Iodine absorption of substitute.	30·9	31·0	32·6	42·0	42·0	56·3	52·6	32·5	26·9	33·6	35·2	21·9	36·3	
Iodine absorption of fatty acids.	91·3	91·2	102·3	129·0	125·6	160·3	$\left\{ \begin{array}{l} 141·2 \\ 121·0 \end{array} \right\}$	101·5	102·8	153·3	$\left\{ \begin{array}{l} 153·2 \\ 147·4 \\ 152·1 \end{array} \right\}$	143·5	91·5	
Acetyl number.....	..	..	..	..	..	21·0	19·6	31·0	..	..	..	105·6	51·3	



The sulphur and chlorine were estimated by dissolving the substitute in fuming nitric acid in the presence of silver nitrate, subsequently fusing with sodium nitrate, dissolving in water and filtering. The sulphuric acid was estimated in the filtrate, the insoluble silver compounds containing the chlorine were estimated by titration with solutions of sulphocyanide and silver. The presence of considerable quantities of chlorine in the white substitute explains the low iodine absorption of these products, although it is not quite as low as had been believed and as will be found by leaving the substitute for 12 hours in contact with Hübl's iodine solution. On saponification with alcoholic potash, the chlorine is completely eliminated from the substitute, whilst the sulphur remains in combination with the fatty acids. The amount of fatty acids contained in the substitute appears rather small. It was however found, that on precipitating the fatty acids from the aqueous solution of the saponified substitute, the mother-liquor, which contains large quantities of hydrochloric acid, contained neither sulphuretted hydrogen, sulphurous, nor sulphuric acids in more than traces. On concentration, until hydrochloric acid begins to escape freely, the liquid becomes turbid and now contains large quantities of sulphuric acid. Part of the fatty acid of the substitute must therefore be contained in it in the form of a sulphonic acid produced by some oxidising influence upon the chloride of sulphur. The free fatty acids obtained from the substitute absorb more iodine than the latter, owing no doubt to the chlorine in them being eliminated on saponification. From the analyses tabulated above, it is evident that the substitutes A and B have been prepared from the same oil, whereas C must have been obtained from a different oil. On the action of chloride of sulphur ( $S_2Cl_2$ ) on oils very little is known. On adding chloride of sulphur to a fatty oil a homogeneous mixture is obtained, and very quickly an energetic reaction accompanied by evolution of heat, vapours of chloride of sulphur and also a little hydrochloric and sulphurous acids, takes place. After a few minutes the mixture congeals to a solid yellow and rather elastic mass which can easily be crushed in a mortar. On exposure to the air, any excess of chloride of sulphur which may be present, evaporates, and a neutral substance is obtained equal in every respect to the commercial substitute. If too little chloride of sulphur be used the product of the reaction is more or less sticky. The following proportions of chloride of sulphur and oil were found to give satisfactory results:—

100 parts of linseed oil	with 30 parts of $S_2Cl_2$ .
" poppy seed oil ..	35 "
" colza oil ..	25 "
" cotton seed oil ..	45 "
" olive oil ..	25 "
" castor oil ..	20 "

There is evidently no relation-ship between the drying power of an oil and the proportion of chloride of sulphur it requires to form a solid substitute. The analysis of substitutes prepared from various oils and using the above mentioned proportions of chloride of sulphur, in no case led to figures similar to those obtained with substitutes A and B, but the substitute prepared from colza oil appear to be identical with substitute C. Oxidised or "blown" oils require less chloride of sulphur for conversion into solid substitute than the natural oils, the amount of chloride of sulphur decreasing with the degree of oxidation of these oils, and it was found that the "soluble castor oil" of commerce, which is really blown cotton-seed oil, by treatment with about 20 per cent. of chloride of sulphur yields substitutes, the analysis of which exhibits almost the same numbers as that of the substitutes A and B. Considering that chloride of sulphur is more expensive than any of the oils available for the manufacture of india-rubber substitute, the preparation of these compounds from oxidised oils offers a distinct economic advantage. About the constitution of these substitutes no definite opinion can be formed yet, but there is little doubt that they are addition-products of the oil and chloride of sulphur, since they contain chlorine and sulphur in the same proportions as this compound. Separation of glycerol does not take place during the formation of the substitutes, or they would become easily soluble in caustic alkalis, which is not the case. It was

further proved that in saponifying the substitutes with alcoholic potash, glycerol always forms one of the products of the saponification. The fact that the iodine absorption of these substitutes is always very much lower than that of the essential oils, shows that the action of the chloride of sulphur upon the oils consists in the conversion of the unsaturated fatty acids of these oils into saturated compounds. In removing the chlorine from the substitute by treatment with alcoholic potash, unsaturated fatty acids are re-formed, as is proved by the high iodine absorption of the fatty acids so obtained, compared with the low iodine absorption of the corresponding substitute. It is, therefore, highly probable that on saponifying the substitute, the chlorine attached to one carbon atom is separated, together with hydrogen, from the next carbon atom, as hydrochloric acid, and with re-formation of double bonded linking between these two carbon atoms. The other possibility, that simply substitution of hydroxyl for chlorine takes place, is very improbable. Indeed, on employing Benedikt's acetyl-test it was found that no such reaction takes place.

"Brown or black substitute," which is obtained by boiling oils with sulphur, contains only minute quantities of chlorine. Also these substitutes are easily saponifiable with alcoholic potash and on precipitating the sulphuretted fatty acids appreciable quantities of sulphuretted hydrogen are evolved, and the proportion of sulphur contained in the free fatty acids is several parts per cent. lower than that of the original substitute. The iodine absorption of these substitutes and of their fatty acids are very high, so that it would appear that these oils are prepared by boiling linseed oil, or mixtures of linseed and colza oil, with sulphur.—C. O. W.

#### *Contributions to the Analysis of India-Rubber Substitutes.*

R. Henriques. Chem. Zeit. 1893, 17, 916.

In a former paper (see preceding abstract) it had been shown that oxidised oils require less chloride of sulphur for conversion into solid rubber substitutes than fresh oils, and, further, that from the iodine absorption of these substitutes and of their fatty acids, the nature of the oils used for their manufacture could be inferred. Glycerides of the saturated fatty acids being indifferent to chloride of sulphur, mixtures of such fats and of the ordinary oils would yield substitutes with comparatively small quantities of chloride of sulphur, and these would give analytical results very similar to those of substitutes prepared from oxidised cotton-seed oil. Boiling alcohol would, however, easily extract from such compound substitutes all the saturated fats, while the substitutes prepared from homogeneous oils yield nothing to alcohol. Possibly saturated and unsaturated glycerides can be separated by treatment with chloride of sulphur, and subsequent extraction with alcohol.—C. O. W.

#### *Deductions from the Analysis of India-Rubber Articles.*

R. Henriques. Chem. Zeit. 1893, 17, 707.

See under XXIII., page 70.

#### *The Manufacture of India-Rubber Waterproof Cloth.*

C. O. Weber. Zeits. ang. Chem. 1893, [21], 22.

See under V., page 33.

#### PATENT.

*Manufacture of Artificial Caoutchouc.* A. F. St. George, Redhill, Surrey. Eng. Pat. 15,544, August 30, 1892.

SUBSTITUTE of turpentine is passed upwards through a heated tube of  $\frac{1}{8}$ th inch or less diameter; the issuing vapour is directed against a spray of hydrochloric acid or other acid, "which has the effect of throwing down solid fragments of caoutchouc." These fragments are thoroughly washed, when they are available for all the purposes to which cleansed, masticated caoutchouc is applicable. The vapour issuing from the tube may be condensed by cooling, and the resulting liquid agitated with hydrochloric acid, when the caoutchouc fragments will be produced.—A. G. B.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

*The Action of Salt on the Putrefying Bacteria of Hides.*  
F. H. Haenlein. Dingl. Polyt. J. 1893, 288, 211—215.

Nor only is it considered unnecessary to remove the salt from salted hides before they are put in the soaks to soften, but even advantageous to add salt to the soaks in which dried hides are being softened. The slight loss of hide substance caused by the action of the salt is compensated for by the checked putrefaction and by the softening action of the salt.

Strips of ox hide, prepared as for tanning, were immersed in sterilised water, and in 2 per cent., 10 per cent., and saturated salt solutions respectively, the vessels being left open to the air. After a week, 0.1 cc. of the saturated and 10 per cent. salt solutions developed only one colony on a Koch's plate, whilst the same quantity of the 2 per cent. solution gave 871 colonies, most of which were micrococci, and did not liquefy the gelatin; the same quantity of the water soak gave innumerable colonies, some of which were micrococci, and others liquefying bacilli.

In a second similar series of experiments the beakers were covered with clock-glasses, made tight with filter paper saturated with mercuric chloride. After eight weeks the saturated and 10 per cent. salt solution produced no colonies; the 2 per cent. solution produced many colonies, but no liquefying bacteria; the water soak produced many colonies, including liquefying bacteria. The hide which had been in the water and in the 2 per cent. salt solution was considerably putrefied, soft, and discoloured; whilst that in the other solutions was still fresh and somewhat harder in the saturated solution than in the 10 per cent. solution.

It was noted that the alkaline reaction of the 2 per cent. salt solution was always stronger than that of the water, so that it would seem that this small quantity of salt considerably alters the character of the fermentation, but whether *per se* or by extinction of the liquefying bacteria remains unsettled.—A. G. B.

*The Course of the Absorption of Tannin in the Layers.*  
C. Sadlon. Dingl. polyt. J. 1893, 288, 256. (Compare this Journal, 1893, 273, 610.)

THE absorption of tannin by the hides in the layers is not in simple proportion to the time of laying away, but is constantly diminishing when no addition of tannin is made. A graphic representation of the absorption gives a parabola. Thus it is unprofitable to leave the hides in a layer beyond a certain lapse of time, for after this the loss of interest is greater than the gain in the weight and quality of the leather.—A. G. B.

*Decomposition of Hides during the Process of Tanning.*  
Von Schroeder and J. Pässler. Dingl. Polyt. J. 1893, 289, 137—141, 210—212, 229—234. Compare this Journal, 1893, 297, 610.

AN investigation, undertaken for the purpose of determining whether hides decompose during the process of tanning, was made by Muntz about the year 1877. His method was essentially that now adopted by the authors, and yielded him results which indicated a loss of 8.6 per cent. of hide substance. This conclusion is untrustworthy because of the small size of the pieces of hide with which Muntz experimented.

The authors have studied the question both for dressing hides (uppers) and for sole-leather. In the first series of experiments 32 dried Buenos Ayres horse hides were put into the soaks for seven days, during which time they were once transferred to the tumblers for 20 minutes and returned to fresh water in the soaks. Six days in arsenical limes followed, after which the hides were unhaird, washed, and given a further six days in pure limes. The fleshed hides were soaked for two days, and bated with pigeons' dung at 25° C. for one day, this bating being followed by tumbling in running water, slicking, trimming, and a further few

minutes tumbling in water. After a final tumbling in water and draining on the horse, the hides were ready for the suspenders.

One of the 32 hides was now cut in half, care being taken that no water was expressed during the cutting, and the halves weighed; the one, weighing 5,110 grms., was tanned with the rest of the batch, whilst the other, weighing 5,170 grms., was retained for analysis.

The hides, including the experimental half, were next placed in the first colouring pit, where the liquor was from pine bark and of about 0.60 B. After frequent handling during the first day, the strength of the liquor was increased to 0.80—0.85 B. Six days later the hides were transferred to the second colouring pit, where the liquor had twice the strength. Here they remained for six days, and were then passed to the third and fourth pits, in each of which a similar period was allowed. The last colour contained quebracho, extracted hot, in addition to the pine, and had a specific gravity of 1.50—1.60 B.

Ten weeks in layers with pine and quebracho liquor at 2 B., completed the tannage. The experimental half hide was then air-dried.

For the analyses, representative strips were cut both from the untanned and tanned half, the portions chosen being the butt (*spiegel*), the middle of the back, and the belly and shoulder. The analytical data were obtained for a piece of each portion, and computed for the whole portions and the whole hide. In the following the figures for the whole hide will alone be given, inasmuch as attention has already been drawn to the slight differences which exist in the composition of different portions of the same hide (this Journal, 1893, 610).

The composition of the untanned half was—

	Per Cent.
Air-dried hide substance.....	26.78
Absolutely dry hide substance.....	22.17

The composition of the anhydrous hide substance was—

	Per Cent.
Fat.....	1.10
Ash.....	1.73
Hide substance.....	97.17
	100.00

Nitrogen.....	17.20
---------------	-------

In the fat- and ash-free hide substance the percentage of nitrogen was 17.78.

The tanned half contained—

	Per Cent.
Air-dried leather.....	67.61
Anhydrous leather.....	38.33

The nitrogen in the anhydrous leather was 10.14 per cent., or 10.18 per cent. in the leather extracted with carbon bisulphide and water.

Assuming that the nitrogen in the anhydrous leather corresponds with the same proportion of hide substance as does the nitrogen in the anhydrous, ash-, and fat-free hide substance, the following may be deduced from the above figures:—

	Hide substance,	Nitrogen,
In 100 parts of hide.....	21.54	3.84
In the leather obtained from this quantity of hide,	21.90	3.89
Difference.....	+0.36	+0.05

It is thus apparent that during the tanning of a horse hide, in the manner described above, there is no appreciable loss, or decomposition of hide substance.

In the following table the proximate composition of the anhydrous leather, of the anhydrous hide, and of the leather obtained therefrom, are shown; the last column gives the gain or loss of the several constituents when the

hide passes into leather—the yield of leather from 100 of hide being 172·9 :—

	Anhydrous Leather.	In 100 Parts of Anhydrous Hide.	In 172·9 Parts of Anhydrous Leather.	Loss or Gain.
	Per Cent.			
Fat.....	0·83	1·10	1·44	+ 0·34
Ash.....	0·45	1·73	0·78	- 0·95
Water extract :—				
Tannins.....	3·57	..	6·17	+ 6·17
Not tannins.....	1·49	..	2·58	+ 2·58
Fat-free hide substance.	..	97·17	98·79	+ 1·62
Tannin in pure leather substance.	..	..	63·14	+63·14
Nitrogen.....	10·16	17·29	17·57	+ 0·28
Sugar, calculated as glucose.	0·11	..	0·19	+ 0·19

It will be noted that there is a decrease of ash; this is attributable to the solution in the tanning liquor of a portion of the lime absorbed by the hide during the unhairing. The slight gain of "fat" is possibly due to the acquisition of some resinous substance from the liquors. The figures show that the total tannin absorbed by 98·79 parts of hide substance is 63·14 + 6·17, or 70·3 per cent., so that the hide is not tanned to saturation, for the authors have already shown (*loc. cit.*) that the maximum absorption of tannin is 100 per cent. The figures given in the paper for the different parts of the hide indicate that the flank is less fully tanned than the other parts, probably because of its greater thickness.

A second series of experiments was conducted on exactly the same lines, with calf skins, the preparation and tannage being as follows:—Seven days in the soaks, with several turnings and renewals of water; seven days in old and fresh limes; unhairing; two days in fresh lime. After being fleshed the skins were bated for a day in pigeon dung, whence they were passed into water at 20° C, and, on the same day, were drained and weighed. They were then passed through five colouring pits, the first two of which consisted of pine and the last three of pine and oak; the specific gravity was gradually increased from 0·99° B. to 1·20° B., and the whole colouring process lasted for 28 days. The skins were finally transferred to the layers, where oak and pine liquor at 1·50° B. was used; here they remained for 44 days. After being in the second colouring pit the skins were slicked on the flesh and trimmed; the portions removed amounted to 4·52 per cent. of the raw weight.

As in the previous series of experiments, three skins were divided before the whole batch went into the colours; three halves were tanned with the rest of the skins, whilst the other three halves served to supply analytical data for the raw skins. It was, of course, necessary to analyse the fleshings and trimmings, referred to above, separately, and to correct the analyses for these.

The untanned skin contained 19·06 per cent. of anhydrous material, which gave the following figures :—

	Per Cent.
Fat.....	0·75
Ash.....	1·11
Hide substance.....	98·14
	100·00
Nitrogen.....	17·45

Thus the true hide substance contained 17·79 per cent. of nitrogen.

The anhydrous fleshings and trimmings, which, it will be remembered, had been slightly tanned in the colours, amounted to 1·23 per cent. of the untanned skins and contained—

	Per Cent.
Fat.....	1·72
Ash.....	1·35
Matter soluble in water :—	
Tannin.....	1·93
Not tannin.....	2·81
True leather.....	92·19
	100·00
Nitrogen.....	12·22

The anhydrous leather amounted to 31·82 per cent. of the untanned skins, and contained 9·67 per cent. of nitrogen.

From the above figures it may be calculated that—

	Nitrogen.	Hide Substance.
100 parts of untanned skin contained.....	3·33	18·71
The leather, fleshings, and trimmings from this contained.....	3·23	18·16
Difference .. .. .	- 0·10	- 0·55

It will be seen that a slight loss is recorded in this balance sheet, whereas in that audited for the horse-hide leather there was a slight gain. It is justifiable to attribute such small differences to experimental errors, and to conclude that in the usual practice of tanning hides for dressing leather, there is no appreciable loss of hide substance.

The following analyses show the difference in composition between calf skin and the leather therefrom; the columns are to be interpreted as follows :—

- Percentage composition of calf leather.
- Percentage composition of dried calf skin.
- Composition of the 6·45 parts of dry fleshings and trimmings from 100 parts of dried skin.
- Composition of the 166·95 parts of dry leather from 100 parts of dried skin.
- Total of C. and D.
- Increase or decrease of E over B.

	A.	B.	C.	D.	E.	F.
Fat.....	0·50	0·75	0·11	0·83	0·94	+ 0·19
Ash.....	0·65	1·11	0·09	1·09	1·18	+ 0·07
Water-soluble :—						
Tannin.....	4·17	..	0·12	6·96	7·08	+ 7·08
Not tannin.....	2·00	..	0·18	3·34	3·52	+ 3·52
Fat- and ash-free hide substance.....	..	98·14	4·43	90·74	95·17	- 2·97
Tannin in the pure leather.....	..	..	1·52	63·90	65·51	+65·51
Nitrogen.....	9·67	17·47	0·79	16·15	16·94	- 0·53
Sugar (as glucose).....	0·18	..	..	0·30	0·30	+ 0·30

On comparison of this table with the similar table given above for horse leather, it will be noted that there is a slight gain of ash in the calf leather, whereas there was a loss in the horse leather; this difference the authors attribute to the more drastic bating and washing which the calf skins receive; these remove the lime more completely than it is removed from the horse hides before tanning. The loss of hide substance is of course magnified by calculation on the dry skin and leather; several unimportant suggestions are made to account for the loss, otherwise than as a decomposition of hide substance.

One hundred parts of the hide substance of calf skin absorbed 78.2 parts of tannin, of which 7.7 parts are easily removed by water.

The third series of experiments is concerned with sole leather. The dry Buenos Ayres hides were put into fresh water soaks, where they remained for nine days with several changes of water. They were then drained and sweated for three days. Unhairing, washing, fleshing, and another washing completed the preparation. The division of the experimental hides was then made.

The hides passed through four suspenders, in which they remained two, three, four, and five days respectively. The suspender liquor was prepared from a sour bark from old layers; it consisted of the extract from this bark diluted with 3, 1,  $\frac{1}{2}$ , and 0 times its volume of water for each of the four suspenders respectively. The extract had a specific gravity of 1.0059 (0.86° B.) and contained—

	Grms. in 100 cc.
Tannin .....	0.226
Not tannin .....	0.916
Total acid (as acetic acid) .....	0.493
Volatile acids .....	0.300
Non-volatile acids .....	0.193

The fifth and final suspender contained 30 kilos. of pine bark for the 7 $\frac{1}{2}$  hides, and was followed by a pit containing 40 kilos. of oak bark and 30 kilos. of pine bark, in which the hides remained for seven days. The first layer contained liquor from 66 kilos. of oak- and 147 kilos. of pine-bark, and the second contained liquor from 100 kilos. of oak-bark, 60 kilos. of pine-bark, and 20 kilos. of valonia; the tanning lasted for 37 weeks and six days.

The experimental hides were divided into butt, shoulders, and belly; representative portions of these were analysed and the composition of the whole portions and of the whole hide computed. The hide contained 28.84 per cent. of dry substance, which was composed of—

	Per cent.
Fat .....	0.28
Ash .....	0.30
Hide substance .....	99.42
	100.00

The nitrogen in the dry hide was 17.76 per cent., or 17.87 per cent. in the ash- and fat-free hide. 100 parts of undried hide gave 52.45 parts of absolutely dry leather, which contained 9.40 per cent. of nitrogen.

Thus a balance sheet may be made out as follows:—

	Hide Substance, Nitrogen.	
In 100 parts of ash- and fat-free hide .....	28.67	5.12
In the leather therefrom .....	27.50	4.93
Difference .....	1.08	0.19

Calculated on the dry hide substance, the loss during tanning becomes 3.77 per cent. The authors, whilst fully aware of the sources of error introduced by the difficulty of determining the moisture in the hide and leather, feel compelled to admit that this is a genuine loss; they attribute its magnitude to the length and severity of the process of tanning sole leather. At the same time the loss is much smaller than was stated by Muntz (see above), and in practice

should not exceed the percentage which the authors have found. A comparison of the three series of experiments may be made:—

	Percentage Loss or Gain of Anhydrous Hide Substance.	Duration of Tanning.
(1.) Horse leather.	+1.67	100
(2.) Calf leather.	-3.04	77
(3.) Sole leather.	-3.77	320

The authors again advert upon the German custom of estimating the yield of leather by the increase in weight of the water-containing hide (compare this Journal, 1893, 610). The butt in this condition always contains the least water, so that it will appear to be the best tanned; as a fact the bellies are always more tanned than the shoulders, and these more than the butts.

The following table shows the composition of sole leather, and the increase or decrease of its constituents over those of the hide. 100 parts of anhydrous hide yield 181.87 parts of anhydrous leather.

	Leather.	Anhydrous Hide Substance.	In 181.87 Parts of Anhydrous Leather.	Loss or Gain.
	Per Cent.	Per Cent.		
Fat .....	0.25	0.28	0.45	+ 0.17
Ash .....	0.37	0.30	1.58	+ 1.28
Water-soluble:—				
Tannin .....	4.73	..	8.60	+ 8.60
Not tannin .....	4.25	..	7.73	+ 7.73
Fat- and ash-free hide substance.	..	99.42	95.65	- 3.77
Tannin in pure leather.	..	..	67.86	+ 67.86
Nitrogen .....	9.40	17.76	17.10	- 0.66
Sugar (as glucose) ..	0.36	..	0.65	+ 0.65

It will be seen that 100 parts of ash and fat free hide substance have absorbed 76.91 parts of tannin.—A. G. B.

## PATENTS.

*Improvements in the Treatment of Leather.* H. Lebl and E. Atlas, Vienna. Eng. Pat. 1216, January 19, 1893.

THE claims in this patent are for producing velvet-leather, having a fine and short nap or down, from ordinary leather by roughening on the grain side, and for smoothing such velvet-leather, where desired, by ironing and pressing. Heretofore velvet-leather has always been produced by roughening the flesh side, which, however, is too coarse for a fine nap. The process of roughening is purely a mechanical one, being performed by friction with some gritty material or by a sand blast.—A. G. B.

*An Improved Process for treating and purifying Sod and other Oils, and Apparatus therefor.* F. N. Turney. Nottingham. Eng. Pat. 1349, January 21, 1893.

See under X11., page 46.

*Improvements relating to the Treatment of Hides and Skins.* H. H. Lake, London. From A. J. Strauss, Paris. Eng. Pat. 2545, February 4, 1893.

THIS invention relates to a process of transforming tanned goat, sheep, calf, and other hides into tawed hides so as to combine the suppleness and other qualities imparted to the hides by tawing, with the strength obtained by tanning.

The tanned goat-skins (one dozen) are submitted to the following operations:—

- (1.) Working in a tumbler with tepid water.
- (2.) Working in a tumbler with a solution of 30 grms. of potash in 60 litres of water for half an hour.
- (3.) Soaking for five minutes in a very weak solution of hydrochloric acid, which has the effect of neutralising the potash.

These three operations constitute the cleansing of the hides "from injurious tanning materials."

- (4.) Pounding and working for 2—3 hours in a tepid bath composed of—

Solution of quebracho (30 "litres" of pulverised quebracho to 150 litres of water).....	15 litres.
Glycerin.....	0.15 ..
Wheat flour.....	75 grms.
Brum.....	30 ..
Alum.....	19 ..
Water.....	30 litres.

- (5.) Two hides are placed flesh to flesh on a marble table and rubbed with an ordinary tanner's grit-stone.

- (6.) The hides thus arranged are rubbed on both sides and brushed with a solution of 100 grms. of ammonia in 15 litres of water.

- (7.) They are then impregnated with a solution of campeachy wood, prepared by boiling 16 kilos. of the wood in 110 litres of water. The hides, still together, are immersed in this liquor, and are then spread on tables to "oxidise the solution of campeachy," the treatment being repeated until a deep chestnut colour has been attained.

- (8.) The hides, still together, are next brushed, on a curved table, with a solution of sulphate of iron and sulphate of copper in the proportion of 60 grms. to 15 litres of water.

- (9.) The hides are then again immersed in a campeachy bath (8 litres of campeachy extract to 100 litres of water), and afterwards separated and rinsed in pure water.

- (10.) Each hide is finally aired, placed on a table, and rubbed on the hair side with a round-edged tool.

The final operations, which are known in tawing, need not be described.—A. G. B.

*An Improved Fat Emulsion and method of producing the same.* M. Ekenberg, Stockholm, Sweden, Eng. Pat. 17,566, September 18, 1893.

See under XII., page 16.

## XVI.—SUGAR, STARCH, GUM, Etc.

*The Composition of Lime-tree Honey.* M. Maquenne, Comptes rend. 117, 127—129.

In dry seasons the leaves of the lime-tree often become covered with a saccharine exudation, known in France as *Miellée* or *Miellat*, on account of its taste. Sometimes this is sufficiently abundant to drop off; apparently its formation is connected with the presence on the leaves of a parasitic insect. Obviously its origin is closely akin to that of various kinds of manna. From polarimetric observations and determinations of the copper reducing power of the saccharine matter before and after hydrolysis, Boussingault concluded in 1869 that ordinary sugar was mainly present, along with invert sugar, and a little dextrin. The author finds that *melezitose* is present in large quantity; by washing the leaves with water and evaporating the solution 100 kilos. of leaves furnished about 1 kilo. of brown syrup of strongly sweet taste, with slightly bitter after taste.

Alcohol precipitated a gummy matter, after removal of which crystals gradually formed, from which ultimately 100 gm. of pure *melezitose* were obtained. The syrup contained something like half its weight of solid matter, so that the *melezitose* probably amounted in all to about 40 per cent. of the total solid matter. Glucose constitutes a large fraction of the remainder; a gummy substance, partially precipitable by alcohol, was also present. Hence lime-tree *Miellée* has a composition closely akin to that of larch manna and the manna of *Alhagi camelorum*.—C. R. A. W.

*The Carbohydrates of the Jerusalem Artichoke.* C. Tauret, Comptes rend. 117, 50—53.

THE juice is treated with lead acetate and the excess of lead eliminated; warm concentrated baryta water is then added in large quantity, whereby a precipitate is formed rich in inulinic matters; additions of alcohol of gradually increasing strength throw down further precipitates. By methodically treating these fractionally with carbonic acid and alcohol, and again fractionally precipitating with baryta and alcohol, dextro-rotatory saccharose, and various levo-rotatory carbohydrates are finally obtained. A litre of juice contains a total of about 160 grms. of saccharose, inulin, pseudo-inulin, inulinin, helianthenin, and synanthrin; the latter two are new products, and are separated from the others by taking advantage of their varying solubilities in alcohol of different strengths. All the constituents named suffer hydrolysis under the influence of acids, or even water, yielding a mixture of levulose and glucose. Helianthenin crystallises in microscopic needles, melting at 176°; its rotatory power is  $\alpha_D^{20} = -23.5^\circ$ . In water it is very soluble, less so in weak alcohol, the less the greater the alcoholic strength; it is fermentable with beer-yeast, but only with difficulty. The author represents its composition (after drying at 110°) as  $12(C_6H_{10}O_5)_3H_2O$ , or preferably  $C_{72}H_{120}O_{72}$ . Synanthrin is amorphous, and represented by the formula  $8(C_6H_{10}O_5)_2H_2O$ , or preferably  $C_{96}H_{160}O_{96}$ ; it melts at 170°, and has the rotatory power  $\alpha_D^{20} = -17^\circ$ . Weak acids hydrolyse both, forming mixtures of glucose and levulose.—C. R. A. W.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*Chemistry of Hops.* C. J. Lintner and A. Bungener, Zeits. ges. Brauw. 1893, 357.

According to the authors, the crystals obtained from Haiduck's  $\alpha$ -resin are different from H. Bungener's bitter acid from hops, but appear to be identical with the bitter principle obtained by Lerner.—A. K. M.

*Influence of Acidity in Worts on the Composition of "Low-Wines."* L. Lindet, Comptes rend. 117, 122—125.

It is a common practice with distillers to acidulate worts with a mineral acid before fermentation, with the object of preventing the development of certain undesirable organisms; more alcohol is thus formed, since the production of lactic and butyric acids is diminished. In certain cases, however, the formation of these organic acids is desired; mineral acids (such as sulphuric, hydrochloric, and hydrofluoric acids) are then not added; under these circumstances a wort, practically neutral before yeasting, gradually becomes acid to an extent exceeding 3 grms. per litre, expressed as sulphuric acid.

The author finds that the addition of mineral acids also diminishes the proportion of bases, volatile acids, and compound ethers formed during fermentation, but increases the quantity of higher alcohols developed, this being the general

result in a number of carefully made duplicate experiments where a given wort (from maize, barley, &c.) was divided into two portions, each treated absolutely alike throughout, excepting that in one case a small percentage (e.g., 1.5 gram. per litre) of sulphuric or hydrofluoric acid was added, and in the other not. In a manufacturing experiment with 3,000 kilos. of potatoes, one half fermented in the presence of hydrofluoric acid, and the other half without mineral acid, the following comparative figures were obtained on systematic fractional distillation of the "low-wines" produced by the first distillation of the wash:—

	Acidulated with Hydrofluoric Acid.	Not Treated with Acid.
	Per Litre of Alcohol. Cc.	Per Litre of Alcohol. Cc.
Higher alcohols insoluble in water	2.05	1.65
Amylic alcohol contained therein.	1.43 Gm.	1.00 Gm.
Bases.....	0.107	0.127
Volatile acids (calculated as acetic acid).	0.78	1.37
Compound ethers (calculated as acetic ether).	0.43	1.47

—C. R. A. W.

*The Estimation of the Ready-formed Sugars in Malt.*  
J. Jais. Zeit. für das gesau. Brauw. 16, 1893, 357—360 and 365—367.

THE author finds that in the extraction of the malt by alcohol, in the ordinary method of determining the above, the acid of the malt is without augmentative influence on the amount of the substances reducing Fehling's solution. The amount of extract obtained from a malt is of course conversely as the strength of alcohol employed, and this, the author shows, is due to diastatic action taking place on the already existing starch products in the presence of the weaker alcohols, and not to greater solubility of the sugars in them; for, on extracting a malt with boiling alcohol (94.4 per cent.), and at the same time killing the diastase and then extracting with water, the total amount of sugars contained in the two extracts is equal to that obtained by digesting a malt of ordinary diastatic activity for 15 minutes with cold water. In the case, however, of low-dried and green malts even this short extraction with water was sufficient to bring about a considerable degradation, and hence an increase on the amount of sugars obtained by the preceding method.

The author recommends the following method for estimating the ready-formed sugars in any kind of malt:—10 grms. of the malt are digested with 500 cc. 94 per cent. alcohol for 1—2 hours, in a boiling water-bath, in a flask fitted with an inverted condenser. The alcoholic solution is filtered off when cold and the residue washed with alcohol, dried at 60° C., and extracted with water at 55° for a considerable time, with constant shaking. The sugars are then estimated in the two extracts. Ether cannot be used to separate the fat from the alcoholic extract, as it dissolves an appreciable amount of cane-sugar. By extracting the residue, after evaporating off the alcohol *in vacuo*, with hot water, and filtering the solution when cold, the greater part of the fat was left on the filter. A blank experiment performed with soluble starch proved that no diastatic action takes place under these conditions. The enpic reduction of the extracts was calculated as invert-sugar, the increase on digestion with  $\frac{1}{4}$ th normal hydrochloric acid (Meissl's method) as cane-sugar, while the further increase on inversion according to Sachsse's method was calculated as gum.

The alcoholic extract contained the greater part of the cane-sugar and but very little gum, of which latter the aqueous extract contained a large amount. The total amount of cane-sugar found in the two extracts corresponded to that found in a hot-water mash of the same malt. The

cane-sugar contained in these extracts cannot be estimated by the polariscope owing to the levo-rotatory action of the gum they contain.—J. G. W.

*The Degradation of Starch by the Action of Diastase.*  
C. J. Lintner and G. Dull. Ber. 26, 16, 2533—2547.

THE authors find that five products only are the result of the action of diastase upon starch, viz., three dextrans, which they term amylo-, erythro-, and achroo-dextrin, and two sugars, isomaltose and maltose, so that, according to them, Brown and Morris's hypothesis regarding the transformation of starch is no longer tenable, their so-called amyloins consisting partly of mixtures of dextrin and isomaltose and being partly identical with them (this Journal, 1893, 851—855).

Their process of degradation was conducted as follows:—100 parts of potato starch were mashed with 500 parts of water at a temperature of 70° C., with the addition of air-dried malt in varying amount up to the extent of 6 parts, according to the dextrin it was desired to obtain. The dextrins and sugars were isolated by stopping the diastatic action at the necessary point by boiling and then repeatedly fractionating the product with alcohol.

*Amylodextrin* ( $C_{12}H_{20}O_{10}$ )<sub>54</sub>, was obtained by stopping the degradation while iodine still gave a blue reaction and then saturating with hot 10 per cent. alcohol. The dextrin separated out on cooling, and, after purification by repeated fractionation with 40—50 per cent. alcohol, was obtained as an extremely light, white powder, only slightly soluble in cold water, but readily so in hot. It did not reduce Fehling's solution, gave a deep blue colour with iodine, and had a specific rotary power  $[\alpha]_D = 196^\circ$ . Its molecular weight, determined by Raoult's method, corresponded to the above formula.

*Erythrodextrin* ( $C_{12}H_{20}O_{10}$ )<sub>17</sub>.  $C_{12}H_{22}O_{11}$ , was prepared by stopping the diastatic action when iodine gave a reddish-brown colouration, saturating with hot alcohol and filtering. The filtrate was concentrated to a syrup and repeatedly treated with 70 per cent. hot alcohol to remove the sugars. It is easily soluble in water, but scarcely so in 50 per cent. alcohol. Its reducing power is 1 per cent. that of maltose and its  $[\alpha]_D = 196^\circ$ . With iodine it gives a reddish-brown colouration.

*Achroodextrin* ( $C_{12}H_{20}O_{10}$ )<sub>5</sub>.  $C_{12}H_{22}O_{11}$ , was obtained by stopping the degradation when iodine ceased to give a colouration, and evaporating the product to a syrup, which was then treated a few times with 60—70 per cent. alcohol to remove traces of erythrodextrin. The remainder was evaporated to a syrup and the sugars extracted by treating successively with 90, 85, and 80 per cent. alcohol. It is very readily soluble in water, but is scarcely so in 70 per cent. alcohol. Its reducing power is 10 per cent. that of maltose, its  $[\alpha]_D = 192^\circ$ , and it gives no reaction with iodine solution.

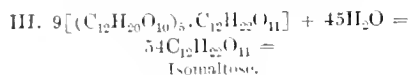
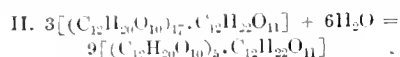
*Isomaltose*,  $C_{12}H_{22}O_{11}$  (probably  $C_{12}H_{22}O_{11} + H_2O$ ), was isolated by stopping diastatic action when a red iodine reaction was obtained. The crude syrup was fractionated with 85—90 per cent. alcohol, all fractions having  $[\alpha]_D = 140^\circ$  being collected, concentrated, dissolved in hot methyl alcohol and precipitated with absolute ethyl alcohol. All fractions having  $[\alpha]_D = 140^\circ$  were again united and the operation repeated until no trace of maltose could be detected in the resulting body by means of the osazone. Isomaltose has not yet been obtained in a crystalline form, but the authors are in hopes of doing so from methyl alcohol solutions. It is extremely soluble in water, in 80 per cent. alcohol, and still more so in methyl alcohol, but in hot 95 per cent. ethyl alcohol it is scarcely soluble. It readily caramelises on exposure to heat and has an intensely sweet taste. Its reducing power is 80 per cent. that of maltose, and its  $[\alpha]_D = 140^\circ$ . It is fermented by yeast with greater difficulty than is maltose, into which it is converted by the action of diastase. Its osazone crystallises from water in spherical aggregates of extremely fine yolk-coloured needles, which soften at 138—140° C. and melt at 150—153° C. It is much more readily soluble in

water and alcohol than is maltosazone, from which it is also easily distinguished by its melting-point and its crystalline form.

The numbers obtained with isomaltose by Raoult's method point to its formula as being  $C_{12}H_{22}O_{11} + H_2O$ .

The authors propound the following theory of starch-transformation. Starch is a union of high molecular complexes and under the action of diastase, dilute acids, or combined heat and pressure, splits up into its high molecular components, of which amyloextrin is the first to be liberated. Amyloextrin is hydrolysed into three molecules of erythroextrin, and the latter again into three of achroodextrin, from which by further hydrolysis isomaltose results, which is then converted into its isomer maltose.

These stages are represented by the following equations:—



These stages, however, proceed simultaneously.

The authors attempt to explain the stoppage of the degradatory process which takes place under restrictive conditions, as for instance, at a temperature of  $70^\circ C.$ , by the supposition that in the conversion of erythroextrin into achroodextrin under unfavourable circumstances part only of the resulting achroodextrin is further degradable, the remainder "taking a form which withstands diastatic action." Under the same conditions the conversion of isomaltose into maltose is restricted.—J. G. W.

#### *The Preparation of Betaine and Choline from Malt-Combs and Wheat Germs.* E. Schulze and S. Frankfurt. Ber. 26, 1893, 2151—2155.

BETAINE and choline can be obtained from the above in the following manner:—The material is extracted with water, lead acetate added so long as a precipitate is produced, the solution acidified with sulphuric acid and repeatedly filtered. Phospho-tungstic acid is then added and the resulting precipitate washed and treated in the cold with milk of lime. The filtrate from the insoluble calcium compounds is treated with carbonic anhydride to remove the excess of lime, filtered, neutralised with hydrochloric acid, evaporated to a syrup and the latter extracted with hot 90—95 per cent. alcohol. Alcoholic mercuric chloride solution is added to the extract which is then allowed to stand for several days, at the end of which the separated mercuric double salt is removed and crystallised from water. The portion of the salt difficult of solubility, contains the betaine while the easily soluble part consists of the choline compound. They can be separated from each other by repeated fractional crystallisation from water, or, by decomposing with sulphuretted hydrogen and treating the hydrochlorides so obtained with cold absolute alcohol. The choline salt dissolves while the betaine compound remains behind.

Three kilos. of wheat embryos yielded 5—6 grms. of betaine hydrochloride, the yield of the choline salt being considerably less. Malt rootlets yielded a somewhat less amount of the betaine salt but a rather larger quantity of the choline compound.—J. G. W.

#### *The Development and Ripening of the Cider Apple.* L. Lindet. Comptes. rend. 1893, 117, 696—698.

THE author has studied the gradual transformation of starch into sugars in the case of the cider apple by making analyses of samples taken from the same tree from July to November

inclusive. The following figures for the extreme terms of the series will illustrate the changes that occur.

	July 24.	November 3.
Average weight of an apple.....	Grm. 21'5 Per Cent. 1'8	Grm. 76'5 Per Cent. 0'8
Starch.....	1'1	2'9
Saccharose.....	0'4	9'4
Invert sugar.....	0'5	0'2
Acidity as malic acid.....	0'4	0'2

He has also examined the ripening of apples that have been picked and allowed to ripen in the dark, and has found that corresponding progressive changes take place.

—B. B.

#### *The presence of Boric Acid in Beer.* J. Brand, Zeits. ges. Brauw. 1893, 16, 197.

A CONSIGNMENT of German export beer had been rejected by the chemist of the custom house of Montevideo on the ground that it contained boric acid. This acid, however, occurs naturally in beer and is derived from the hops, in which it was first observed by Delton. The quantity of boric acid thus present is very small and to judge by the intensity of the turmeric paper test, less than 50 milligrms. per litre. For analysis at least 100 cc. of beer should be employed and examined by Gooch's method. The quantity of boric acid naturally present will turn turmeric paper only faintly red. This stain on moistening with alkalis turns bluish-green, which proves that it is due to boric acid. An addition of 5 milligrms. of boric acid in 100 cc. of beer (an amount which with regard to its antiseptic action, can scarcely be considered) will give by Gooch's method a very intense reddening of turmeric paper.—C. O. W.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY AND WATER PURIFICATION, DISINFECTANTS.

### (A.)—CHEMISTRY OF FOODS.

*Saffron and its Adulterants.* E. Vinassa. Archiv. der Pharm. 1892, 231, 353, and Dingl. Polyt. J. 290, 17.

See under XXIII., page 71.

*Analysis of the Butters of Commerce.* C. Viollette. Comptes. rend. 117, 856—858.

THE author's determinations of the weight *in vacuo* of 1 cc. of genuine butters of various origin, margarines, and mixtures of the two have led him to the belief that by means of a density determination alone commercial butters may be rapidly sorted into three classes; viz., those undoubtedly adulterated with margarine, those doubtful, and those that may be considered practically pure. Mixtures of margarine and butter have uniformly the density due to the constituents without contraction, &c.; at  $100^\circ$  1 cc. weighs (*in vacuo*)—

	Grm.	Grm.
Genuine butter.....	0'86320 to 0'86425	
Margarine.....	0'85790 to 0'85865	

Animals fed chiefly on hay furnish butters of lower density than those from cows allowed grass, cake, and roots, &c. Out of 500 samples, only two (from high fed



beasts) give still higher values, 0.86530 and 0.86540; whilst one, from a cow fed on straw and dry hay, gave a lower figure, 0.86277. Accordingly he has had constructed an extremely delicate areometer by means of which the density at 100° of the substance examined can be at once determined with reference to these three classes, the stem having three corresponding marks upon it. A quantity indicating 0.0001 gram. per cc. represents 1.4 mm. of length on the stem. Conveniently, three smaller areometers may be employed, No. 1 with three marks representing respectively 0.86215, 0.86266, and 0.86320 gram. per cc.; No. 2 and No. 3 arranged for substances of lower density, 0.857 to 0.86266; for these a table is provided giving the proportions of margarine and butter with which the observed density approximately corresponds. Substances yielding figures lower than 0.86266 but higher than 0.86215 (first and second marks on No. 1) may possibly be pure butters, but are more likely to contain margarine; they are therefore considered doubtful, and should be subjected to chemical examination.—C. R. A. W.

*On the Internal Temperature of Bread as it comes from the Oven.* M. Balland. *Comptes rend.* 117, 519–521.

In a previous paper the author advanced the opinion that the internal temperature of bread when taken from the oven is from 97° to 100°, and does not exceed the latter temperature. In his experiments he used loaves of 1 kilo. in weight and the thermometer was inserted in a hole made with a bodkin. The author has since then carried out other experiments, using carefully-graduated thermometers, and inserting them in the bread before baking. The experiments were carried out in the central laboratory of the War Department at the Invalides, and at the military bake-house at Billy.

*Laboratory of the Invalides.*—Here there is a Biabaud oven for 60 kilos. of bread, and it is furnished with a pyrometer.

*1st Experiment.*—With two similar long-shaped loaves A and B, of 750 grms., left in the oven for 40 minutes. The temperature of the oven when the bread was placed in it was 300°, and when the bread was taken out 265°.

A.....	100°·2
B.....	99°·8

*2nd Experiment.*—With two similar loaves of the same weight and shape, A being left in the oven 32 minutes and B one hour. The initial temperature of the oven was 302°, and 275° when A was taken out, and 225° when B was taken out.

A.....	100°·8
B.....	101°·0

*3rd Experiment.*—With two similar loaves of the same weight and shape, A being left in 28 minutes and B one hour. The initial temperature of the oven was 294°, 256° when A was taken out, and 225° when B was taken out.

A.....	101°·0
B.....	101°·0

*4th Experiment.*—With two similar long-shaped loaves of 1,500 grms. A was taken out after 75 minutes and B after 85 minutes.

A.....	99°·2
B.....	100°·6

*5th Experiment.*—With two similar loaves of 2 kilos. left in the oven for 70 minutes.

A.....	100°·8
B.....	100°·8

*6th Experiment.*—With small elongated loaves of from 150 grms. to 200 grms., left in the oven for from 20 to 40 minutes, the temperature was found to be between 100°·5 and 101°·5; on one occasion it reached 105°, but in this case the bulb of the thermometer was partly in the crust.

*7th Experiment.*—With rolls of the same weight it was found in like manner that the temperature only exceeded 101°·5 when the thermometer was inserted in the crust.

*Bake house at Billy.*—The Lamoureux oven used by the author can bake 270 kilos. of bread, and the batches succeed each other continuously, day and night. There is no pyrometer, but the temperature is certainly not less than that of the oven at the Invalides.

1. With the loaves in ordinary use (which join other loaves in four places and weigh 1,500 grms. after 45 minutes baking) the temperature was from 100°·5 to 101°·5.

2. With three similar loaves, baked separately, the temperatures were 102°, 102°, and 103°·6. In the latter case the bulb of the thermometer touched the crust.

3. With a loaf of 750 grms. made with biscuit dough, the thermometer registered 101°.

4. With soldiers' biscuits the results were uncertain, the thermometers being too near the crust.

From these experiments, carried out in different ovens and with loaves of different size and shape, the author concludes that the temperature of the crumb of the bread during baking reaches to 100° or 102°, while that of the crust is much higher. Beyond 100° the aqueous vapour, imprisoned by the crust, is under pressure, and when the pressure is lowered by making a hole in the crust, as in the author's first experiments, the temperature does not exceed 100°.—D. E. J.

## PATENTS.

*Improvements in the Manufacture of Yeast.* F. M. Pelsky, Paris. Eng. Pat. 20,787, November 16, 1892.

A MIXTURE of green barley and wheat-malt flours is kneaded with water and allowed to turn sour. It is then digested at a temperature of 62°–65° C., and cooled. Pressed yeast is then added and the mixture divided into two parts. To one part tartaric acid and subsequently sodium bicarbonate are added, and to the other ammonium carbonate. The two are then mixed and allowed to stand. The resulting product is washed with a solution of calcium phosphate and pressed.—J. G. W.

*Process of curdling Milk and producing Cream and Butter.* M. Julien, sen., and A. Brin, London. Eng. Pat. 22,115, December 2, 1892.

SKIMMED milk and any fatty matter, such as margarin or olive oil, are emulsified by subjecting them, separately or in a state of mixture, under very high pressure (about 250 atmospheres), and in the form of very fine jets ( $\frac{1}{32}$  inch diameter), to violent impact, by projecting the jets either against one another or against a common surface of impact. The apparatus for so doing is the subject of another patent, 14,840, August 2, 1893. Butter is prepared from the curdled milk in the usual manner.—J. G. W.

*Improvements in the Utilisation of Flours which are rich in Diastase.* L. Briant, London, and W. H. Walker, Southport. Eng. Pat. 23,748, December 23, 1892.

SINCE the introduction of high-class flours, bread has become less moist and of less flavour, this being due to the fact that in the milling of flours the germ, which is markedly diastatic, is entirely or almost entirely separated. The present invention consists in the separation of diastase from low-grade unmalted wheaten and other flours, which are now practically rejected for bread-making, and adding the diastase so obtained to higher-class flours during the process of making them into bread.

The process employed for extracting the diastase is as follows:—The flour is extracted with water at a temperature not exceeding 120° F., for 1½ hours, with constant stirring, a small quantity of either sulphurous acid or salicylic acid being added as an antiseptic. The extract is filtered and evaporated without delay *in vacuo* to a specific gravity of about 1.450, at a temperature not exceeding 120° F. The extract is used in bread-making in the proportion of from 6 oz. to 1 lb. per sack of flour.—J. G. W.

*Improvements in Milk-Testers.* M. Pedersen, Dursley. Eng. Pat. 1888, January 27, 1893.

THIS improved milk-tester for the estimation of the quantity of cream by means of centrifugal action, consists of a cylinder, closed at one end and open at the other, into which slides another cylinder, open at the lower end and partially closed at the other by a plug of glass tubing of narrow bore. When filled with milk and placed on the centrifugal disk, with the glass plug towards the centre, the cream rises into the narrow bore of the glass plug, the lower end of which is bevelled to facilitate its so doing. The length of the column of cream is then measured off.

—J. G. W.

*Improvements in the Preservation of Alimentary and other Substances, and Apparatus therefor.* C. A. Sahlstrom, London. Eng. Pat. 1954, January 28, 1893.

THE substance to be preserved is placed in baskets within a special receiver and covered with a preservative solution, the nature of which varies according to the character of the substance to be preserved. The receiver is then exhausted, with the result that all the gases contained in the tissues of the organic substance are removed. The atmospheric pressure is then restored by very slowly admitting air into the receiver, and then a pressure of 2–6 atmospheres applied, in order that the tissues may be properly impregnated with the preservative fluid. This is then run off and the substances packed in common boxes or barrels, with or without preserving fluid.

The following solutions are recommended for the several purposes:—For preserving fish for a few days, a 1–2½ per cent. solution of common salt, or pure sea water. For preserving fish for a longer time, the previous solution, with the addition of 1–2½ per cent. of boracic acid or borax,  $\frac{1}{100}$ – $\frac{1}{50}$  per cent. of glycerin, and  $\frac{1}{1000}$ – $\frac{1}{100}$  per cent. of essence of cloves. For salting or pickling fish or meat, a stronger solution of salt with only a very small proportion of glycerin and boracic acid. For preserving eggs, a smaller percentage of boracic acid and a larger one of glycerin. For preserving fruits, a solution containing 2–5 per cent. of sugar,  $\frac{1}{2}$ –2 per cent. of glycerin, 1–1½ per cent. of boracic acid, and  $\frac{1}{1000}$ – $\frac{1}{100}$  per cent. of essence of cloves. For preserving meat, melted tallow, either alone or mixed with 1–2½ per cent. of boracic acid.

It is claimed that by this method the substances after treatment contain only a trace of the ingredients of the preservative solution, retain almost unaltered their original appearance and taste, and require no special arrangements for conveyance to distant markets.—J. G. W.

*An Improved Extract of Cocoa.* T. R. Allison, London. Eng. Pat. 1117, February 24, 1893.

THE shells only of the fruit of the cocoa plant are macerated with water, with the addition of bran. The whole is then heated to a point just short of boiling for three hours, filtered and concentrated. It is claimed that the addition of bran effects a better extraction of the essential principles of the shells, and obviates the need of skimming off the cocoa fat.—J. G. W.

*Improvements in Centrifugal Separators for Cream and Butter.* A. Wahlén, Bainbridge, U.S.A. Eng. Pat. 11,026, June 6, 1893.

THE milk is received into an auxiliary separating vessel in which any foreign substances are arrested, and, as the milk is thrown over the edge of this vessel by centrifugal action, partial separation of the cream from the watery portions also takes place. The materials pass into the main separating vessel, consisting preferably of a hollow conical vessel having a partially flat bottom, the centre portion of which is upwardly conical, and provided with one or more downward openings, around which externally is a cylindrical lip.

In the preparation of butter, the cream passes downwards through these openings, and, being discharged from the cylindrical lip, is received upon the conical under surface

of the bottom, and is there spread out by centrifugal action in such a thin layer that the watery particles pass off and the buttery globules coalesce; the separated materials being received into an annular chamber below the centrifugal separating vessel.

In the preparation of cream, a moveable receiver below the butter-separator is raised and receives the cream from the annular lip so as to prevent any further separation of the fatty and watery constituents being effected. The cream is discharged by a suitable spout from the intercepting vessel.—J. G. W.

*Improvements in the Manufacture of Dough from Cereals, and Apparatus therefor.* F. J. Gelinck, Riga. Eng. Pat. 15,067, August 5, 1893.

THE corn, after washing and freeing from all impurities, is steeped in water at 50° C. for 1½ hours and then crushed between a pair of rollers. The crushed mass is conducted into a ribbed casing of peculiar shape, in which a conveyer-screw rotates. By means of the screw-thread and the ribs of the casing, the crushed mass is further reduced, any pieces which may have escaped crushing being cut up by rotating knives. The whole is then passed through a perforated plate into another similar compartment in which further reduction takes place, it being finally passed through a plate having smaller perforations, when the dough is ready for use.—J. G. W.

*Method of Purifying Water by Oxide of Tin.* H. von der Linde and C. Hess, Uerdingen-on-the-Rhine, Germany. Eng. Pat. 19,216, October 12, 1893.

THE water to be purified is filtered through materials impregnated with oxide of tin. This substance, it is stated, acts as an extremely energetic oxygen carrier, and effects the rapid oxidation of the organic impurities through the agency of the dissolved oxygen present in the water.

—H. T. P.

## (B.)—SANITARY CHEMISTRY.

### PATENTS.

*Improvements in the Treatment and Utilisation of Sewage, and Plant therefor.* A. O. Jones, Harrogate, Yorks. Eng. Pat. 21,291, November 23, 1892.

A PROCESS of treating sewage so as to obviate the dissemination of offensive odours, and to utilise the solid and liquid constituents as completely as possible. The sewage is filtered in a closed chamber through a filter-bed supported on a raised false bottom. During filtration either cold or hot air is drawn through the apparatus, or the latter itself may be heated, the noxious vapours given off being passed into a destructor furnace to render them harmless. The material of the filter-bed is afterwards employed, in conjunction with the solid portion of the sewage, for the manufacture of artificial fuel, or the production of heating gas, &c., in the manner indicated in two earlier patents (Eng. Pats. 14,253, 1890, and 11,764, 1891; this Journal, 1891, 846, and 1892, 597). In the first case the filtering material may be peat, brick-dust, mould, &c.; in the second, coal-dust, town refuse, soot, &c. The liquid portion of the sewage may be sprayed into the furnace, or it may be refiltered and aerated until sufficiently pure and discharged into a water-course. Another form of apparatus is described in which the liquid portion of the sewage is evaporated by maintaining a partial vacuum in the outer chamber, the vapours given off being conducted to a furnace, &c., whilst the sludge is periodically withdrawn at the bottom of the chamber.—H. T. P.

*Improvements in Apparatus for the Treatment of Sewage Matter.* M. Nadein, B. Ewdokimoff, and S. de Baschmakoff, St. Petersburg, Russia. Eng. Pat. 21,931, November 30, 1892.

THIS is an improvement on a previous invention (Eng. Pat. 18,504, 1891) and relates to a novel device for separating the solid and liquid constituents of sewage.

For this purpose the liquid, &c., collected from water-closets, sinks, &c., is caused to flow over a parabolically curved metallic surface, arranged vertically, convex side outwards. The liquid being spread out in a comparatively thin layer, follows the contour of the surface and is received at the lower extremity of the surface in a chute. The solid portions of the sewage, however, when they reach the centre of the curved surface, glance off, and fall perpendicularly into a special receptacle. Details and drawings are given showing the manner of adapting the above contrivance to an ordinary dwelling-house and to a street gully.—H. T. P.

### (C).—DISINFECTANTS.

*Tests of a Disinfectant for Use in Railway Sanitation.* W. T. Sedgwick. Tech. Quart. 1893, 6, 2, 143.

THE disinfectant used by the Pennsylvania Railroad Co. is stated to consist of a solution of the chlorides of zinc, copper, and mercury, together with a little turpentine to act as a tell-tale and indicate where it has been used. The specification to which it is made requires that each 8 oz. bottle shall contain 2,400 grains of zinc chloride, 120 grains of cupric chloride, 10·5 grains of mercuric chloride, and 10 drops of a mixture of equal parts of terebene and turpentine. The author examined the effect of this disinfectant upon various typical bacteria, using methods well adapted to prevent the inhibition of the growth of the bacteria by disinfectant carried over from the vessel in which they had been exposed to its action to that in which their cultivation was to be carried out; the following are the chief results arrived at.

*Bacillus typhi abdominalis.*—The Eberth bacillus of typhoid fever was killed by an exposure of 12 seconds to the disinfectant diluted to one-half its original strength.

*Spores of bacillus subtilis.*—The spores were destroyed by exposure to the disinfectant of the same strength for 17 minutes.

*Streptococcus of pseudo-diphtheria.* The destruction of the organism was effected in 12 seconds under the same conditions of dilution as before.

*Staphylococcus pyogenes aureus.*—An exposure of 10 seconds sufficed.

*Koch's cholera spirillum.*—An exposure of 10 seconds sufficed.

*Spores of bacillus anthracis.*—An exposure of 20 minutes was necessary.

A practical test of the disinfectant with a typhoid stool was made and sterilisation found to be effected in 3½ minutes. The author attributes the disinfecting action of the preparation chiefly to the presence of mercuric chloride.—B. B.

### PATENTS.

*An Improvement in the Manufacture of Sulphate of Copper.* G. F. Strawson, Newbury, Berks, and W. Fream, Downton, Wilts. Eng. Pat. 23,342, December 19, 1892.

A METHOD is described and claimed for the manufacture of sulphate of copper for insecticidal purposes, &c. The hot concentrated solution of copper sulphate obtained in the usual way is treated with one-tenth per cent. of some repellant oil, preferably eucalyptus oil, and allowed to crystallise, being continuously stirred until cold. After some time, any excess of oil is skimmed off, and the crystals dried for use.—H. T. P.

*Improvements in the Treatment of Manganates for the Production of Permanganates suitable for use for Disinfecting or other purposes and in Apparatus therefor.* W. J. Menzies, Malpas, Chester. Eng. Pat. 1213, January 19, 1893.

See under VII., page 38.

*Improvements in Compositions of Camphor with other Substances for making Solid Objects of any desired Shape or Colour.* H. F. Verdun, Pouligny St. Pierre, France. Eng. Pat. 15,767, August 19, 1894.

THE camphor is powdered and mixed with a material such as plaster, cement, or the like. The paste thus obtained may be moulded or pressed into any desired shape. By mixing camphor with wood saw-dust or pulverised leather, different articles of furniture may be manufactured from the composition. Bands to be placed round the stems or roots of vines so as to keep off phylloxera, are to be made from the composition. Any suitable colouring material may be added.—A. G. B.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*The Phosphoester of Benzoylamido Phenyl Acetic Acid.* F. Weiss. Ber. 26, 1699, 1699—1702.

THE above-named ester was prepared by F. Bayer and Co. of Elberfeld in 1890. It, as well as the benzoylamido-phenyl acetic acid, is employed as an intestinal disinfectant. The ester in question was prepared by the action of phosphorus oxychloride on the calculated quantities in mixture, of benzoyl amidophenyl acetic acid and phenol. It has the composition—



and forms small white needles with a melting point of 131° C., which are nearly insoluble in water. In manufacturing this substance the interesting observation was made, that if, during the action of the phosphorus oxychloride the temperature were allowed to rise above 100° C., not the desired ester, but quite a different body was formed. This latter body is formed from the ester by the separation of one molecule of water, and it appears in fine needle-shaped crystals, insoluble in water, but tolerably soluble in ether, alcohol or glacial acetic acid. These crystals melt at 101° C.—W. S.

*Basic Salicylate of Bismuth.* Schubardt. Pharm. Zeit. 1893, 38, 250.

THE following properties and tests should be responded to:—White powder, odourless, tasteless, insoluble in alcohol and water. On heating 1 grm. of the salicylate with 20 cc. of water to the boiling point, and filtering, a colourless, faintly acid filtrate should be obtained, in which barium nitrate and silver nitrate produce only a slight opalescence, and from which, after addition of hydrochloric acid, on cooling, no salicylic acid should be precipitated. On shaking 0·5 grm. with 25 cc. of dilute sulphuric acid and 10 cc. of ether, a clear solution should be obtained without any effervescence. On ignition, a residue of from 62 to 65 per cent. of  $\text{Bi}_2\text{O}_3$  should be obtained.—C. O. W.

*Occurrence of Betaine and Choline in Wormseed.* E. Jahns. Ber. 1893, 26, 1493—1496.

THE author finds that wormseed (*Artemisia Gallica*, Willd) contains about 0·5 per cent. betain and approximately 0·1 per cent. choline.

Wormseed (*Semen Contra*) is the source of santonin, the vermifuge principle of certain of the *Artemisia* family.

—A. K. M.

*Synthetic Formation of Citric Acid by Fermentation of Glucose.* C. Webber. Comptes rend. 117, 332—333.

CERTAIN moulds or fungi (of species the characteristics of which are as yet somewhat uncertain) have the power of converting sugars, especially glucose, into citric acid. Under

favourable conditions 50 per cent. of the glucose used can be converted into citric acid without formation of secondary organic products. The spores of the *citromyces* (*C. pfefferianus* and *C. glaber*) capable of producing this change are abundant in the air; by proper selection and cultivation they have been readily obtained at Hanover and at Thann (Alsace). The production of citric acid in this way is susceptible of being utilised industrially. Accordingly the process is patented.—C. R. A. W.

*A New Test for Esrine.* S. J. Ferreira da Silva.

Comptes rend. **117**, 330.

See under XXIII., page 69.

*The Metallic Compounds of Gallanilide.* P. Cazeneuve.

Comptes rend. **117**, 47—50.

GALLANILIDE, being less oxidisable than pyrogallol, admits of the formation of metallic derivatives that can be obtained in a state of sufficient purity for analysis. A disodium derivative and analogous lime, lead, and baryta compounds are obtainable, indicated by the general formula—

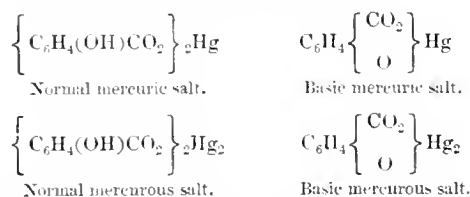


Zinc derivatives can be formed of three classes, viz., where 1, 2, and 3 hydroxyl hydrogens are respectively displaced by metal. Alkaloids appear to combine with gallanilide, forming insoluble compounds; thus, insoluble salts of quinine and strychnine are obtained.—C. R. A. W.

*Mercurial Salicylates.* H. Lajoux and A. Grandval.

Comptes rend. **117**, 44—47.

SALICYLIC acid, being simultaneously a phenol and a carboxylic acid, is capable of forming two classes of salts, according as the hydrogen of the phenolic hydroxyl is or is not replaced by metal; thus in the case of the mercurous and mercuric salts the compositions are—



Normal mercuric salicylate is obtained by precipitating, in the cold, a solution of a mercuric salt with normal sodium salicylate; at a boiling temperature mercuric chloride forms the basic salt, the normal salt being decomposed into basic salt and free salicylic acid; the same change ensues when the normal salt, prepared in the cold, is boiled with water. A simpler way of preparing the basic salt is to dissolve salicylic acid in boiling water and add, little by little, well-washed, recently precipitated mercuric oxide; when the yellow colour, due to the addition of one portion of mercuric oxide, has disappeared, another portion is added. The basic salt is white, and insoluble in water, alcohol, ether, and chloroform; when suspended in cold water it is not decomposed by sulphuretted hydrogen until after long standing, becoming gradually yellow, and finally black. It is slightly soluble in ammonia and in solutions of sodium chloride and potassium iodide; still more so in caustic soda. These solutions are not precipitated black by sulphuretted hydrogen; but, on standing, a yellow precipitate forms, consisting of a compound of sulphide and salicylate of mercury, salicylic acid being set free. Similarly, the basic salt, digested with a weak alkaline sulphide solution, forms the yellow double salt, which only blackens very slowly. Cold dilute hydrochloric acid is without action on the basic salt, but complete decomposition is brought about by

hot concentrated acid. Solutions of the basic salt in caustic soda or potassium iodide solution, when subjected to dialysis, leave the salt behind in an amorphous condition.—C. R. A. W.

*Cinchonine.* O. Hesse. Annalen, **276**, 88—124.

THE author has submitted cinchonine to the action of sulphuric and hydrochloric acids under various conditions, and has obtained 10 isomerides of the base  $\text{C}_{19}\text{H}_{21}\text{N}_3\text{O}_2$ , namely  $\alpha$ - and  $\beta$ -isocinchonine, apocinchonine, apocinchonine, isapocinchonine, diapocinchonine, diacinchonine, homocinchonine, pseudocinchonine and cinchonine. In addition to these there is cinchonine itself, Pum's  $\beta$ - and  $\gamma$ -cinchonine and also Jungfleisch and Léger's cinchonidine and cinchonidine.  $\beta$ -cinchonine may, however, possibly be identical with homocinchonine, and  $\gamma$ -cinchonine likewise bears a close resemblance to isapocinchonine.

The experiments described show how remarkably easily one base may be converted into another, and that such a change may actually occur during the process of the extraction of a base from the plant, or during its purification.

—A. K. M.

*Cinchonidine.* E. Jungfleisch and E. Léger. Comptes rend. **117**, 42—44.

THE supposed isomeride of cinchonine designated cinchonidine is a mixture, or possibly a compound, of cinchonidine and apocinchonine; on treatment with methyl chloride it furnishes a mixture of two kinds of crystals—one consisting of clinorhombic prisms containing  $4\text{H}_2\text{O}$ , found to be identical with apocinchonine chloromethylate; the other made up of fine needles, much more soluble in water, and identical with cinchonidine chloromethylate. The free base, although exhibiting remarkable constancy in its physical properties, notably in rotatory power, is nevertheless gradually broken up by crystallisation from alcohol; with a minimum of boiling solvent, cinchonidine separates; but with a large excess of alcohol, the crystals first deposited consist of cinchonidine, whilst apocinchonine accumulates in the mother-liquors.—C. R. A. W.

*Alkaloids of Papaveraceæ.* E. Schmidt. Arch. Pharm. **1893**, **231**, 136, 161, 174.

THE suggestion that a distinct chemical relationship exists between plants morphologically related, has not been strictly confirmed by these investigations. Of the numerous opium alkaloids, only Hesse's protopine was observed in the other papaveraceæ, *Chelidonium majus*, *Stylophoron dianthillum*, *Sanguinaria canadensis*, *Eschscholtzia californica*. Bardet and Adrian's observation of morphine in the last-named species could not be confirmed. The roots of *Sanguinaria canadensis* were found to contain the following alkaloids. (1.) *Chelerythrine*,  $\text{C}_{22}\text{H}_{17}\text{NO}_4 + \text{C}_2\text{H}_5\text{OH}$ , crystallises with one molecule of alcohol, which is not given off at  $150^\circ \text{C}$ . The hydrochloride,  $\text{C}_{22}\text{H}_{17}\text{NO}_4 \cdot \text{HCl} + 5\text{H}_2\text{O}$ , crystallises in brilliant yellow needles. The hydroiodide forms yellow needles and contains no water of crystallisation. (2.) *Sanguinarine*,  $\text{C}_{20}\text{H}_{15}\text{NO}_4 + \text{H}_2\text{O}$ , forms white needles with melting point  $213^\circ \text{C}$ . It is characterised by the blood-red colour of its salts. The hydrochloride contains five, the nitrate one molecule of water of crystallisation. (3.)  $\gamma$ -*Homochelidonine*,  $\text{C}_{21}\text{H}_{21}\text{NO}_5$ , together with  $\beta$ -*Homochelidonine* and protopine, occurs in the ammoniacal mother-liquor obtained from the precipitation of the crude alkaloids. (4.)  $\beta$ -*Homochelidonine*,  $\text{C}_{21}\text{H}_{21}\text{NO}_5$ , was obtained in minute quantities. It was also observed by Hesse in the roots of *Chelidonium majus*. (5.) *Protopine*,  $\text{C}_{20}\text{H}_{17}\text{NO}_5$ , was first isolated from opium by Hesse. Its melting point is  $270^\circ \text{C}$ . It forms colourless salts. The above formulae for *Protopine* and *Chelerythrine* were confirmed by G. König.—C. O. W.

*Investigations on Narceine.* Martin and Freund and (in part) G. B. Frankforter. Annalen, **277**, 20—58.

NARCEINE is indicated by the formula,  $\text{C}_{22}\text{H}_{27}\text{NO}_5 \cdot 3\text{H}_2\text{O}$ , the hitherto generally received formula,  $\text{C}_{22}\text{H}_{29}\text{NO}_5 \cdot 2\text{H}_2\text{O}$ , being inexact. As formerly pointed out by Alder Wright

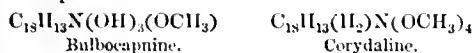
and Beckett, it stands in very close relationship to narcotine, gentle oxidising agents furnishing the same hemipinic acid in each case; accordingly the question arises, is narcine actually pre-contained in poppyeapsule juice, or is it formed from narcotine during the processes adopted in manufacturing morphine from opium? On the whole, the authors incline to the former view. A large number of narcene derivatives are described, amongst others, metallic derivatives such as *potassium narcene*,  $C_{23}H_{29}KNO_3$ ,  $C_2H_5O$ , which crystallises with alcohol of crystallisation. Narcene contains three methoxyl groups, and has the constitution of a substituted phenylbenzyl ketone.—C. R. A. W.

*Investigations on the Alkaloids contained in the Root of Corydalis cava (Schwegg).* M. Freund and W. Josephi. *Annalen*, 277, 1—19.

The authors conclude, from their own work and that of Dobbie and Lauder, and of E. Merck, that the root of *Corydalis cava* contains the following alkaloids:—

	Melting Point.
Bulbocapnine, $C_{13}H_{19}NO_3$ .....	139°
Corydine, amorphous .....	65°—75°
Corydaline, $C_{23}H_{29}NO_3$ .....	134°
Corycavine, $C_{23}H_{29}NO_3$ .....	215°
Corytuberine, $C_{19}H_{25}NO_3$ .....	Above 200°
Corybulbine .....	208°

Corydaline forms a crystallisable hydrochloride and hydriodide; with methyl iodide it unites to form a crystalline compound, which on treatment with silver chloride and caustic potash in succession forms *methyl corydaline*,  $C_{23}H_{29}NO_3$ ; this also combines with methyl iodide. It contains four methoxyl groups, whereas bulbocapnine contains but one: probably the two are related, as indicated by the respective formulæ—



Corycavine contains no methoxyl group. Various salts of corydaline, bulbocapnine, corycavine, and corytuberine are described.—C. R. A. W.

*The Alkaloids of Gelsemium Sempervirens.* A. R. Cushny. *Ber.* 26, 1893, 1725—1727.

In this paper the author describes some of the properties of the alkaloids occurring in *Gelsemium sempervirens*. *Gelsemine* (Gerard, *Pharm. J.* and *Trans.* (3), 13), is a white, non-crystalline, strongly alkaline, bitter substance. Its hydrochloride is crystalline, easily soluble in water, slightly so in alcohol. It gives no colour reactions with nitric or sulphuric acids. But if to a solution in the latter, an oxidising agent be added ( $MnO_2$ ,  $K_2Cr_2O_7$ , &c.), an intense red colour is developed, passing gradually into green. The gold and platinum double salts are soluble in hot water, from which they crystallise on cooling. *Gelsemine* produces in frogs spasmodic contractions followed by paralysis of the extremities of the motor nerves. Analysis assigns to the hydrochloride the formula,  $C_{19}H_{23}N_3O_{14} \cdot 2HCl$ .

*Gelseminine* (Thompson, *Pharm. J.* and *Trans.* (3), 17) is colourless, amorphous, strongly alkaline, insoluble in water, soluble in alcohol, ether, and chloroform. Its salts are readily soluble, amorphous, yellowish substances. *Gelseminine* gives with nitric acid a green, and with sulphuric acid a yellow coloration, changing on addition of oxidising agents to violet and finally to green. *Gelseminine* is highly poisonous, not only to amphibia, but also to warm-blooded animals. One mgrm. of this alkaloid suffices to kill a rabbit, whilst 0.5 grm. *gelsemine* has no effect whatever upon it. The platinum double salt of *gelseminine* has the formula,  $C_{19}H_{23}N_3O_{14} \cdot HClPtCl_4$  (compare this *Journal*, 1893, 618).—H. T. P.

*Derivatives of Toly-antipyrine.* G. Liebert. *Pharm. Zeit.* 1893, 38, 251.

*Toly-antipyrine hydrochloride* is obtained as a crystalline body by passing a current of hydrochloric acid through a solution of toly-antipyrine in benzene.

*Chloral hydrate toly-antipyrine* is formed when hot solutions of molecular proportions of chloral hydrate and toly-antipyrine are mixed together. It separates as a crystalline precipitate.

*Ferric chloride toly-antipyrine* is obtained by substituting ferric chloride for chloral hydrate in the last process, alcoholic solutions being used. A precipitate is formed consisting of a yellowish-red amorphous powder, which is the ferric chloride compound referred to.

*Monobromo- and mono-iodotoly-antipyrine* are obtained by treatment of the toly-antipyrine dissolved in chloroform, with bromine or iodine, as the case may be.—C. O. W.

*Gardenia Resin.* E. Heckel and Schlagdenhauffen. *Répert. de Pharm.* 1893, 49, 115.

See under XIII., page 47.

*The Preparation of the Oak Tannins, with Special Reference to the Use of Acetone as a Solvent.* H. Trimble and J. C. Peacock. *Pharm. Jour.* 1893, 53, 317—318.

THE usual solvent for tannins is official ether, sp. gr. 0.750, which is equivalent to a mixture of alcohol and ether. Critical experiments have been made on oak bark with this solvent and with acetic ether, water, and acetone. Acetone was found to be the most suitable, and the following process is therefore recommended:—The powdered oak bark is well moistened with acetone, packed in a glass percolator, and the menstruum poured on until it begins to drop from the lower orifice, when the latter is closed with a cork, and the bark allowed to macerate for 48 hours, evaporation being prevented by covering the percolator with a glass plate smeared with vaseline. The cork is then removed and acetone poured through until the number of litres of percolate amount to one-half the number of kilos. of bark used. The acetone is then distilled off over a water-bath, at first at ordinary pressure, but afterwards under reduced pressure. The dark-coloured residue is warmed with water until nearly all of it has dissolved; the solution is cooled and filtered, and the clear filtrate diluted with water until no further precipitation occurs. The precipitate, consisting of the bulk of the anhydrides, is filtered off, and the filtrate successively agitated with acetic ether. The extracts are mixed and the acetic ether distilled off, under reduced pressure, in order to obtain the tannin in a porous condition. The product is treated with cold water, the solution is filtered, and the filtrate is again extracted with acetic ether. This process is repeated until the tannin is completely soluble in water. The tannin now possesses an odour of acetic ether, which is removed by solution in official ether, sp. gr. 0.750, and distillation of the solvent. For the removal of the last trace of resin and crystalline principles, digestion with absolute ether should follow.

It was found in some cases that by dissolving the acetone residue in a mixture of four parts of water and one part of alcohol, instead of water alone, a smaller quantity of anhydride was formed.

From a sample of powdered nut-galls commercial ether extracted 59.77 per cent. of solids, whilst acetone extracted 62.24 per cent. of the same.—A. G. B.

*The Localisation of the Active Principles of the Myronette.* L. Guignard. *Comptes rend.* 117, 861—863.

INDIGENOUS species of the family of the *Rosaceæ* contain in their stems and leaves, and especially in their roots, a nitrogenous sulphurised essential oil, apparently identical with that of black mustard. This, however, does not pre-exist as such, but as a glucoside, which becomes decomposed under the influence of the special ferment, *myrosine*, contained as cellulose distinguishable under the microscope. In

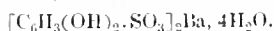
these respects the miguonettes exactly resemble the other cruciferous plants previously examined by the author; these results are entirely at variance with those previously recorded by Spatzier, who found that myrosine was not contained in the roots (though present in the aerial organs and the seed); and that the oil was present in the roots as such, and not as glucoside.—C. R. A. W.

*Geraniol.* Ph. Barbier. Comptes rend. 117, 120—122.

THE essential oil of *Andropogon Schannanthus* (Hindoo geranium oil) contains about 75 per cent. of geraniol,  $C_{15}H_{25}O$ , easily separated in an approximately pure condition by fractional distillation *in vacuo*, the impurity being a trace of a sesquiterpene, which increases its density and gives it a slight laevo-rotatory power. Pure geraniol is obtainable by converting into acetic geraniol ether by means of acetic anhydride at 150°, and saponifying the product. The ether boils at 129—130° under 14.5 mm. pressure, and has the specific gravity at 0°, 0.9388; pure geraniol boils at 126°—127° under 16 mm. pressure, and has the specific gravity at 0°, 0.9012; gaseous hydrochloric acid forms a dihydrochloride,  $C_{15}H_{25}Cl_2$ , boiling at 142°—143° under 16 mm. pressure, and breaking up into hydrochloric acid and dipentene (characterised by its tetrabromide) on boiling with acetic solution of potassium acetate. Geraniol appears thus to be the stable stereo-chemical isomeride,  $C_{15}H_{25}O$ , and linalol the unstable one.—C. R. A. W.

*Action of Sulphuric Acid on Pyrocatechol and Homopyrocatechol.* H. Cousin. Comptes rend. 117, 113—115.

PYROCATECHOL and twice its weight of sulphuric acid mixed and allowed to stand in the cold for some days become a solid crystalline mass of pyrocatechol sulphonic acid. The free acid melts at 53°—54°, and is soluble in alcohol and in ether; the barium salt is—



It yields a fine green coloration with ferric chloride, becoming red with alkalis. The potassium salt—



is very soluble in water, but insoluble in cold alcohol. If pyrocatechol and sulphuric acid be digested on the water-bath, the same sulphonic acid is formed, together with a little of a disulphonic acid. This latter is obtained in quantity by heating together on the water-bath for half an hour a mixture of one part pyrocatechol and five of fuming acid containing 30 per cent. of anhydride. The potassium salt of this is  $C_6H_3(OH)_2 \cdot (SO_3K)_2 \cdot H_2O$ . At temperatures above 100°, ordinary sulphuric acid chars pyrocatechol.

Homopyrocatechol only yields a monosulphonic acid, whether acted on by oil of vitriol or by 30 per cent. fuming acid. This acid is soluble in alcohol and ether; it melts at 93°—94°. The barium and potassium salts are respectively  $[C_7H_5(OH)_2 \cdot SO_3]_2Ba, 3H_2O$ , and  $C_7H_5(OH)_2 \cdot SO_3K, H_2O$ .—C. R. A. W.

*Oil of Spike (Lavandula Spica).* G. Bouchardat. Comptes rend. 117, 53—56.

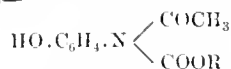
THE essential oil examined, had the sp. gr. 0.920, and the deviation +1°, 28' for 10 cm. thickness. Its odour was disagreeable; with alcoholic potash at 100° only traces of organic salts were formed, chiefly acetate (*Lavandula vera* oil contains the acetic and valeric ethers of geraniol and linalol in quantity). On continued fractional distillation it furnished a large proportion of distillate passing over at 120°—126° *in vacuo*, or near 200° at ordinary pressure; by treatment of this with hydroxylamine in alkaline alcoholic solution and distilling off unaltered linalol, camphoroxime was obtained crystallised in the residue, borneol being contained in the mother-liquor; these were ultimately separated and recognised. From fractions passing over at 145°—150° and 150°—160° borneol was isolated. A dihydrochloride,  $C_{20}H_{31}Cl_2$ , boiling at 145°—150° was also obtained by means of hydrochloric acid, apparently the dihydrochloride of

geraniol. The fraction passing at 160°—190° was chiefly a hydrocarbon,  $C_{20}H_{34}$ , of the copaiba family. Previous experiments having shown that eucalyptol,  $C_{20}H_{34}O_2$ , is also present (boiling point near 175°), it results that five distinct isomerides of this formula are contained in oil of spike; viz., eucalyptol, laurel camphor, linalol, geraniol, and terpinol; this last being obtained as a dihydrochloride (melting at 48°—50°) from fractions passing over at 130°—135 and 135°—140°.—C. R. A. W.

## PATENTS.

*Improvements relating to the Production of Acetyl and Propionyl Compounds of Parahydroxyphenylurethanes or their Ethers.* J. F. Freiherr von Mering, Halle, Germany. Eng. Pat. 22,757, December 10, 1892.

THE parahydroxyphenylurethanes,  $HO \cdot C_6H_4 \cdot NHCOOR$ , or their ethers,  $RO \cdot C_6H_4 \cdot NHCOOR$ , are converted into their acetyl derivatives—



by heating them for about an hour with an equal weight of acetic anhydride, either under pressure or with inverted condenser; the excess of acetic anhydride is distilled off and the product allowed to crystallise. In the place of acetic anhydride either glacial acetic acid or acetic chloride may be employed. The propionyl derivatives are similarly prepared, propionic acid, anhydride or chloride, being then employed.

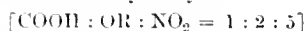
The products are chiefly intended for medicinal purposes, and are said to exhibit a powerful antipyretic and analgetic action.—A. K. M.

*Production of Ethers of Sulphuric Acid.* Newton and Son, London. From the Farbenfabriken vormals F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 824, January 13, 1893.

THE class of ethers to which this patent refers are mixed ethers containing one alkyl group and one aromatic group. They are obtained by the interaction of alkylsulphonic chlorides  $RO \cdot SO_2Cl$  with phenols, derivatives of phenols, naphthols, their analogues, homologues, and substitution derivatives in the presence of alkalis. These mixed sulphuric ethers are not decomposed by water, and they are stated to possess powerful and valuable medicinal properties.—A. K. M.

*Manufacture of Amido-alkylsalicylic Acid and Acetamido-alkylsalicylic Acid.* Abel and Imray, London. From Kuehler and Boff, Crefeld, Germany. Eng. Pat. 3182, February 13, 1893.

NITROMETHYL- or nitro-ethylsalicylic acid—



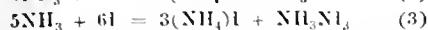
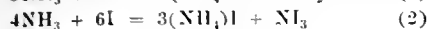
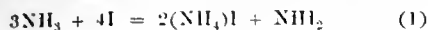
obtained by treating alkylsalicylic acid with nitric acid of sp. gr. 1.2, is converted into the corresponding amido-alkylsalicylic acid by treatment with the ordinary reducing agents. The corresponding acetyl-derivatives are readily obtained by treating the amido-compounds with glacial acetic acid, acetic anhydride dissolved in acetic acid, or acetyl chloride. It is preferable, however, to prepare them directly from the nitro-compounds by treatment with tin and glacial acetic acid. The products are applied to medicinal uses.—A. K. M.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*Iodide of Nitrogen.* Szuhay. Ber. 1893, 26, 14, 1933—1945.

AFTER recapitulating previous researches that have been made on the formation and composition of iodide of nitrogen, and pointing out their inconclusive character, the author describes his own investigations on the same subject, together with experiments on the reactions of iodide of nitrogen with various substances.

The mode of preparation adopted by the author consisted in adding an excess of aqueous ammonia to a solution of potassium iodide nearly saturated with iodine. The product is washed with a 1 per cent. solution of sodium sulphate, as when washed with water it is somewhat decomposed, and ammonia is inadmissible because interfering with the subsequent determination of the ratio of iodine to nitrogen in the wet precipitate. By using weighed quantities of iodine and potassium iodide, and determining the amount of iodine present in the iodide of nitrogen formed and in the mixture of ammonium iodide and potassium iodide removed by the process of washing, it was found that half the total iodine entering into the reaction was present as iodide of nitrogen and half as ammonium iodide, leaving a choice of the following three equations to represent the reaction:—



Decision between these was arrived at by decomposing iodide of nitrogen with standard sulphurous acid and determining thus the quantity of  $\text{H}_2\text{SO}_3$  required for the decomposition, and also estimating the ammonia and iodide existing in the products of decomposition. The result was that equation (1) was found to represent the change, whence it appears that the composition of iodide of nitrogen prepared in the manner described above corresponds with the formula  $\text{NH}_2$ .

Having regard to the fact that nitrogen forms with negative elements compounds of acid character, the author endeavoured to replace the hydrogen in "iodide of nitrogen"  $\text{NH}_2$  by a metal. A silver compound was prepared by acting upon  $\text{NH}_2$  with silver oxide or an ammoniacal solution of silver nitrate, to which the formula  $\text{AgN}_2$  was assigned. Evidence was obtained of the existence of similar compounds containing K and Ba in place of Ag, but the corresponding lead salt was too unstable to allow of definite conclusions being drawn concerning its composition. The slightly acid character of  $\text{NH}_2$  allows of its comparison with azoimide, a body of similar constitution.

In the course of the investigation it was shown that if alcohol be added to a mixture of 5 grms. of iodine and 2 grms. of mercury amide chloride, the mixture explodes very violently in consequence of the formation of iodide of nitrogen. If a little carboic acid be present, not iodide of nitrogen, but iodoform, is produced. If amyl alcohol, chloroform, carbon bisulphide, or glycerin be added instead of phenol, gas is evolved, but there is no explosion even after 24 hours. According to Böttger, a mixture of 4 grms. of "white precipitate" ( $\text{N}_2\text{HgCl}$ ) and 6 grms. of iodine, mixed with 60 grms. of alcohol in a porcelain dish, will explode after 30 to 45 minutes. Flückiger found that four atoms of iodine and three molecules of "white precipitate" mixed together will give rise to an explosion in a very brief space of time, yielding  $\text{N}$ ,  $\text{NH}_4\text{Cl}$ ,  $\text{NH}_3$ ,  $\text{HgCl}_2$ , and  $\text{HgI}_2$ . All iodides of nitrogen, from whatever sources, explode on drying.—B. B.

## PATENTS.

*Improvements in the Manufacture of Matches.* W. P. Thompson, Liverpool. From E. Lagneau, Lessines, Belgium. Eng. Pat. 3267, February 14, 1893.

THIS specification describes a methodical system of making matches, which "allows of the green shafts being inserted just as they come from the cutting apparatus, directly into the frames, and of these shafts, held in these frames, under-

going successively all the necessary operations up to the complete finishing, such operations being the drying of the green match shafts in the frames themselves and various dippings for the purpose of coating them with the various chemical ingredients."

The chief characteristic of the process is the drying of the green shafts of the matches in the frames themselves. The mechanical means employed are fully illustrated and described in the original specification. W. M.

*Improved Plugging or Tamping Compound for Blasting Cartridges, also applicable for keeping the Packages of such Cartridges Dry.* C. D. Abel, London. From the Westfälisch-Anhaltischen Sprengstoff Actien Gesellschaft, Coswig, Germany. Eng. Pat. 7572, April 13, 1893.

THIS invention relates to "a compound for plugging or tamping shot holes and for keeping packages of blasting material dry, consisting of a mixture of a neutral powder or absorbent medium, such as infusorial earth, sand, gypsum, brick earth, and the like, with a hygroscopic salt or salt solution, such as chloride of calcium, bisulphate of soda, &c., by means of which on the one hand, the deterioration of blasting materials by moisture during transport and storage is prevented, and on the other hand when it is used as tamping, the flame on firing is surrounded by vapour produced by the vapourisation of the hygroscopic substance which prevents the ignition of fire-damp."—W. M.

*An Improved Safety Explosive, and Method or Process of Manufacturing the same.* W. Evelyn-Lianet, Elsternwick, Victoria, Australia. Eng. Pat. 19,931, October 23, 1893.

THE object of this invention is to produce a safe and economical explosive. The ingredients proposed are tar, picric acid, saw-dust or wood-meal, and potassium chlorate which has been fused until it has lost one-third of its oxygen. The explosive is prepared by heating the tar to about 260° F., adding from one-third to one-half its weight of picric acid. The inventor terms this mixture "prepared tar." To two parts of this "prepared tar" is added one part of sawdust or meal—the mixing taking place in a heated vessel. The resulting product is termed "prepared sawdust." This "prepared sawdust" is mixed with the pulverised "perchlorate of potassium" in a steam-jacketed pan at 220° F. until the mass becomes quite black.—W. M.

*An Improved Gunpowder.* M. E. Leonard, Manchester. New Jersey, U.S.A. Eng. Pat. 20,066, October 24, 1893.

THIS invention relates to the preparation of a smokeless explosive. The ingredients are nitroglycerin, gun-cotton, lycopodium, and a neutraliser of free acid, such as urea or diinitrobenzol. For the United States 30 calibre rifle the patentee has found the following proportions satisfactory:—

150 parts by weight of nitroglycerin, 50 of gun-cotton, 10 of lycopodium, and 4 of urea crystals.

"For use in cannon where a further deterring and moisture proof effect is desired, 7 parts by weight of cotton-seed oil are added to the ingredients above named."—W. M.

## XXIII.—ANALYTICAL CHEMISTRY.

## APPARATUS, ETC.

*Mercury Thermometer for High Temperatures.* M. V. Recklinghausen. Ber. 26, 1514—1517.

(Compare this Journal 1893, 865 and following abstract.)

THE author describes some tests which he made with one of the new mercury thermometers for measuring temperatures



up to 550° C. It was made of Jena glass (No. 59, III.) and the space above the mercury was filled with compressed carbon dioxide. In cases where the whole of the stem of the thermometer was not heated a correction was applied for the projecting portion. If  $t_0$  be the mean temperature and  $a$  the length of the projecting thread of mercury,  $t_1$  the observed boiling point, and 0.000156 the coefficient of expansion of mercury in glass, then the corrected boiling point  $t$  is:—

$$t = t_1 + 0.000156 a (t_1 - t_0)$$

(See Kohlrausch, *Leitfaden der prakt. Physik*, page 68, "Physical Measurements." Churchill Sect. 22 E.)

The boiling point of sulphur was determined on different days and found to lie between 447°—448° C., even although the thermometer was used to determine still higher boiling points between the observations. When the whole of the stem of the thermometer was immersed in the sulphur vapour, slightly higher results were obtained (451°·5—452°). The author therefore recommends that before determining the boiling point of any substance, the boiling point of sulphur should be taken under precisely the same conditions of experiment, and the deviation from the true boiling point (448° C. by the air thermometer) applied as an additional correction. The boiling points of several other substances, with boiling points ranging from 379° to 475° C., were also determined. By means of the new thermometer the author is trying to decide whether or not  $P_2S_3$  exists. He melted phosphorus and sulphur in the proportions given below and determined the boiling point of the mixture with the following results:—

Mixture.	Boiling Point.
$P_2 + S_3$	522°·5—525° C.
$P_2 + S_2$	545° — 546°·5 C.
$P + S_2$	516° — 518° C.

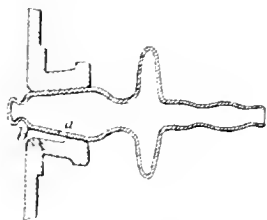
—J. S.

#### Mercury Thermometer for High Temperatures. A. Mahlke. Ber. 26, 1815—1818.

As the correction (see preceding abstract) for the projecting part of the stem of mercury thermometers for measuring temperatures up to 550° C. (this Journal 1893, 865) is sometimes as much as 20° C. wrong, the author recommends the use of an accessory thermometer, with capillary bulb, which is placed alongside the high temperature thermometer and so constructed that the correction can be read off directly.—J. S.

#### A New Stop-cock for Vacuum Desiccators. O. Ernst. Ber. 25, 1893, 1698—1699.

THIS stop-cock forms at the same time the passage for the air and is made out of glass tubing. At one end it is blown out into a stopper, the extremity of which is sealed. The other end is fluted to facilitate connection with the pump. A little above the stopper two arms are blown out, which serve to form a handle with which to turn the stop-cock. In one side of the stopper a small hole ( $a$ ) is drilled,



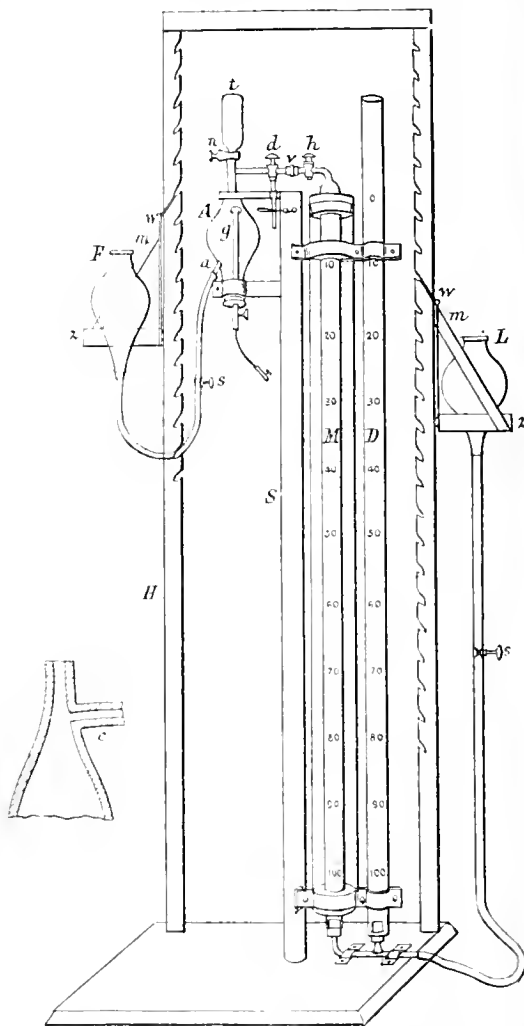
while in the tubulure of the desiccator is made a small notch ( $b$ ), which opens into the interior of the vessel. In one position of the stop-cock these form a connection

between the pump and the desiccator, which a slight turning of the tap is sufficient to close. The inner end of the stopper is formed into a small knob for the purpose of carrying a leathern washer in order to obviate any danger of the stop-cock falling out. A mixture of lanolin and a little vaseline is recommended for greasing the stopper.

—J. G. W.

#### Gas Analysis Apparatus. F. Fischer. Zeits. ang. Chem. 1893, 17, 511—512.

The apparatus consists of a laboratory vessel A connected by a tubulus  $c$  (see enlargement at left side of Fig.) with a measuring tube M and a levelling tube D. A is provided with a tubulus  $a$  by means of which it is coupled with a reservoir F, so that it can be filled or emptied at will. It has also a funnel  $t$  for the introduction of reagents, and a platinum spiral  $g$  for the combustion of gaseous mixtures. The side tubulus  $c$  is shaped as shown in the enlargement in the lower part of the figure, with the view of preventing any drop of liquid from the funnel  $t$  finding its way to the measuring tube M. The three-way



cock  $d$  serves for introducing the gas to be analysed into the apparatus. The platinum spiral  $g$  is carried by a stem, as shown in the figure, which works air-tight through the lower part of the laboratory vessel, so that it can be lowered out of the way of the reagents, dropped from the funnel  $t$ , when not in use. The current requisite for heating it can be supplied by a couple of Leclanche cells or by

three dry cells, there being no risk of overheating and fusion with a battery of such moderate power.

The capabilities of the apparatus and its mode of working are intelligible from the figure. It suffices to say that the author prefers to estimate CO by combustion together with the hydrogen and hydrocarbons of the gas under examination, rather than to rely on its absorption with cuprous chloride, and uses air in place of oxygen at this stage of the operations. The apparatus can be used with mercury or water according to the degree of accuracy to be attained.

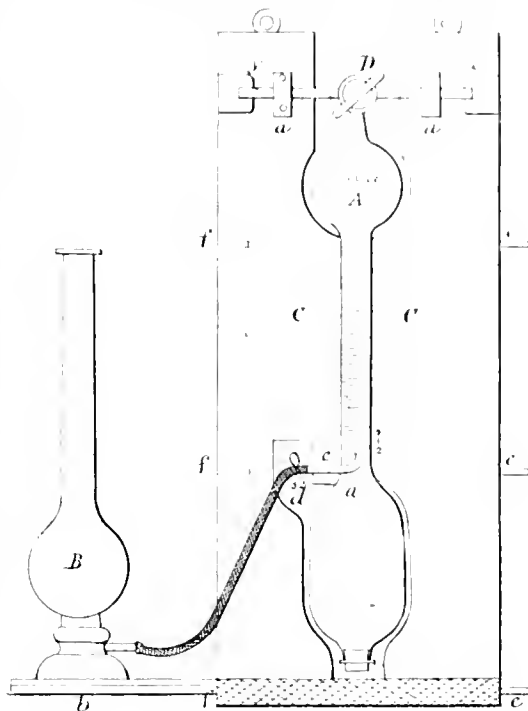
—B. B.

*Apparatus for the Analysis of Gases for Carbonic Acid, Oxygen and Carbon Monoxide.* G. Pfeiffer. Chem. Ind. 16, 462—463.

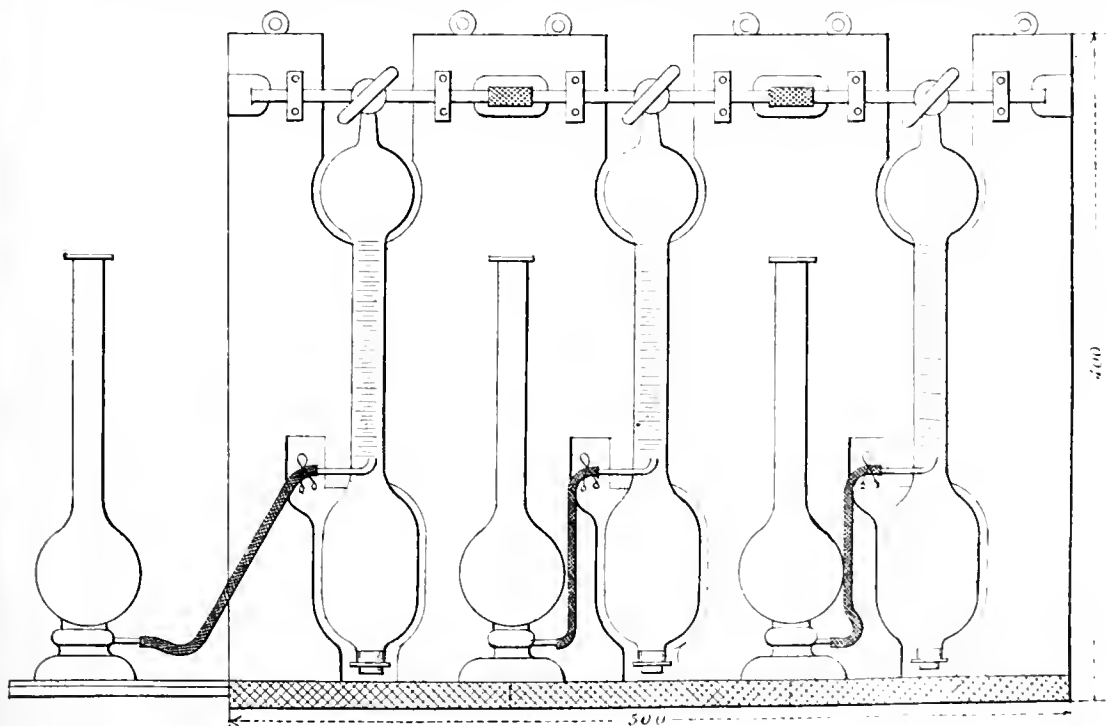
A CONVENIENT form of apparatus, suitable for use in chemical works. It consists of the measuring and absorbing burette A and the aspirator B, which fits into a wooden stand heavily weighted with lead, and connected with the burette by means of a rubber tube 100 cm. long. Tap D has two rectangular perforations enabling the operator to connect the burette with the capillary tubes *x* or *y*, or to disconnect these altogether. The burette is suspended on the sheet iron strips *a a* which connect the capillary tubes with the wooden stand, and may be easily swung round the axis *x y*. The cylindrical part of the burette is charged with the absorbing material. The foot of the stand is provided with a board *b* which may be drawn out on either side and serves as an aspirator stand. The burette is prepared for use by connecting a short rubber tube provided with a funnel to the end *c*, and thus charging up the cylindrical portion as far as *e* with the absorbent solution. End *c* is then connected to aspirator B which is charged with water, slightly acidulated with hydrochloric acid. By opening clip *d*, acidulated water is admitted into the burette and accumulates above the heavier absorbing liquid; the apparatus is thus filled up as far as D.

The gas to be analysed is drawn into the burette by lowering the aspirator, and the level adjusted to O under atmospheric pressure, by opening tap D. This is then closed. By lowering the aspirator, the remaining part of the water is drawn out as far as *c* when the clip *d* is closed

and the contents of the burette agitated by rotation round the sheet iron bands. Two or three rotations are sufficient to complete the absorption, and a reading may be taken after opening *d* to equalise the levels. For a second determination D is opened, the burette filled with acidulated water and D reversed to admit a fresh quantity of gas.



By means of this burette a great number of determinations may be made in a very brief space of time, and



no fear need be entertained as to errors caused by the action of acidulated water on the absorbing material. Nevertheless, care must be taken to maintain the acid reaction of the water in the aspirator, which is preferably coloured by means of methyl orange.

Oxygen determinations are preferably carried out by means of phosphorus sticks, in which case the cylindrical part may be easily warmed up between the hands. For carbon monoxide determinations the use of a solution of cuprous chloride in hydrochloric acid, prepared according to Winkler, is recommended.

The stand C C of each burette is provided with attachments *e* and mortices *f*, enabling the operator to connect two or three burettes so as to form one apparatus.

#### CHECK ANALYSES.

Burettes as Described.			Bunte's Burettes.			Hempel's Burettes.		
Per Cent.	Time required for Experiment.		Per Cent.	Time required for Experiment.		Per Cent.	Time required for Experiment.	
	Mins.			Mins.			Mins.	
CO <sub>2</sub>	12.6	12	12.5	33	29	12.6	29	
O	6.0		6.2			5.8		
CO	1.5		1.3			1.4		
CO <sub>2</sub>	10.0	13	10.2	35	32	10.1	32	
O	6.5		6.5			6.3		
CO	4.0		3.7			3.9		
CO <sub>2</sub>	15.0	11	15.0	32	30	14.8	30	
O	3.5		3.8			3.6		
CO	1.6		1.4			1.5		

—H. A.

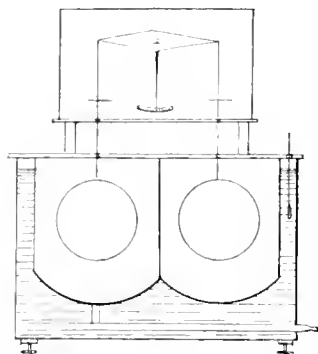
*An Apparatus and Method for Determining the Density of Gases suitable for Industrial Purposes.* M. Meslans. *Comptes rend.* 1893, **117**, 386—390.

Attention to the analysis of gases has not hitherto entered very largely into industrial operations, it is yet desirable to know the composition of gaseous products which are given off at various stages of many of these operations, either in order to understand the nature of the reactions which take place, or to watch the development of the process.

The author believes that the determination of the density of these gaseous products will afford valuable indications of, and, in a large number of cases, with as much accuracy as analysis, will give the composition of simple gaseous mixtures. He has therefore endeavoured to devise a rapid method of determining the density of gases requiring no further skill than is required in the use of ordinary hydrometers.

His method consists in immersing two hollow spheres of equal weight and volume, the one in air, the other in the gas whose density is to be determined. The spheres are suspended from the arms of a balance and hang in separate compartments of a box with double walls of metal. The space between the walls is filled with water to establish equality of temperature in all parts of the apparatus. From the bottom of one of the compartments there issues a copper tube (see Fig.) surrounded by the water of the box. This serves to admit the gases under consideration which thus assume the temperature of the apparatus. The mixture is previously well dried and equal quantities of desiccating material are also placed in the two compartments of the box. The spheres (which are of glass or polished metal) are equilibrated in air and the gaseous mixture is then admitted. To restore equilibrium it is necessary to place a suitable

weight *P* in one of the pans of the balance. This weight *P* represents the difference in the loss of weight sustained by



the two spheres. The loss of weight sustained by the sphere immersed in air is—

$$p = \frac{0.001293 v H}{(1 + at) 760}$$

and by the other is—

$$p^1 = \frac{0.001293 v H d}{(1 + at) 760}$$

where *d* is the density of the gaseous mixture under consideration—

Thus—

$$P = p^1 - p = \frac{0.001293 v H}{(1 + at) 760} (d - 1)$$

whence—

$$d = 1 + P \frac{760}{0.001293 v} \cdot \frac{1 + at}{H}$$

The expression  $\frac{0.001293 v}{H} = K$  is the constant of the instrument, the volume *v* of the spheres being determined with great care. The density is, therefore, given by the very simple formula—

$$d = 1 + P K \frac{1 + at}{H}$$

and it is easy to draw up a table giving for all ordinary values of *t* and *H* their value of  $K \times \frac{1 + at}{H}$ .

For practical purposes the apparatus is so modified that when a slow but continuous current of gas is made to pass through it, the variations in density are indicated at any instant by the position of the pointer of the balance on a suitably divided scale.

The object which the author had in view in devising the apparatus was the systematic study of the combustion which takes place in various systems of furnaces and the regulation of the supply of air to these. The supply of air is in numerous cases ten times the necessary amount, which results in waste of fuel. The author attributes this to the ignorance of the composition of the gaseous products of combustion and the consequent neglect to properly regulate the supply of air. With a few exceptions we may consider the gases of furnaces to consist of the normal product of combustion (79 vols. of nitrogen and 21 vols. of carbon dioxide), which has a density of about 1.0875, and of air in excess. The density of the gases varies with the excess of air. The indications of the apparatus enable us to so modify the admission of air to the furnace as to prevent wasteful combustion.

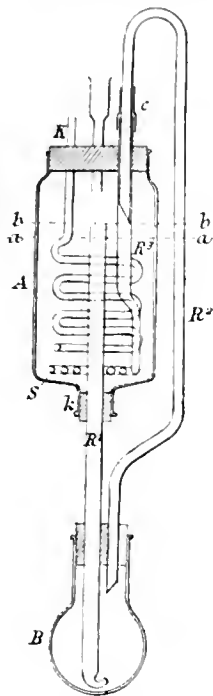
The author also proposes to use the apparatus for determining the quantity of fire-damp in fiery mines.

—D. E. J.

*New Extraction Apparatus for Organic Chemical Purposes.* C. T. L. Hagemann. *Ber.* **26**, 1893, 1975—1977.

The author finds the extraction apparatus shown in the accompanying figure to give excellent results in the extraction

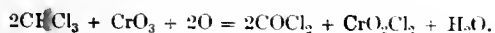
of liquids by light solvents. The solvent is contained in the flask B, which is heated in a water-bath and contains a small piece of porous earthenware; the latter causes boiling to occur more regularly than platinum clippings. The vapour passes up R<sub>2</sub> into a spiral tube S, pierced with small holes at its lower part; the vapour condenses and the solvent rises in small drops through the solution contained in A, forming a layer upon the surface. The solvent, as it accumulates, passes down the tube R into B. The tube R is connected to the vessel A by an india-rubber stopper and may be placed at any convenient height. The apparatus can be employed for the extraction by solvents heavier than the solution by raising the spiral S to the upper part of A,



and attaching a condenser to it; R is then enclosed by a wider glass tube open at both ends and filled with the solvent. By this arrangement the condensed solvent drops through the solution and rises into the wider tube encircling R, down which it then passes.—W. J. P.

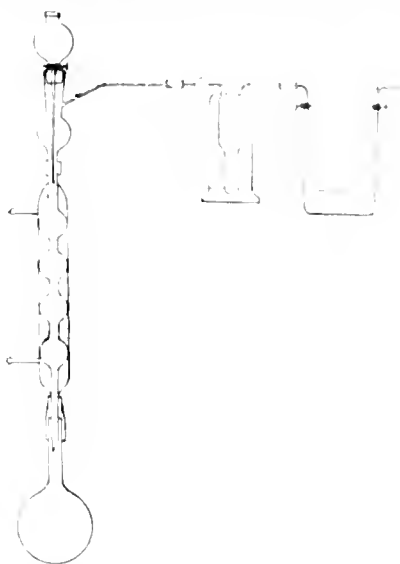
**Oxidation of Chloroform by Chromic Acid and the Preparation of Carbonyl Chloride (Phosgene) from Carbon Tetrachloride.** H. Erdmann. Ber. 1893, 26, 1990—1994.

EMMERLING and Lengyel's method for preparing carbonyl chloride by oxidising chloroform with potassium bichromate and concentrated sulphuric acid gives a poor yield; a somewhat better yield is obtained by employing fuming sulphuric acid in the process. The equation representing the reaction given by the above-named authors is erroneous, the following being the correct one:—



The action of fuming sulphuric acid containing 80 per cent. of sulphur trioxide on carbon tetrachloride affords a far better means of obtaining carbonyl chloride. Carbon tetrachloride (100 cc.) is kept briskly boiling in a 300 cc. flask (see figure) and the fuming acid (120 cc.) allowed to drop down the attached condenser from a dropping funnel so as to come in contact with the vapour: the liberated oxychloride is washed by passing through sulphuric acid contained in a washbottle and condensed in the stoppered vessel shown in the figure, by a freezing mixture. The last portions of

the oxychloride are driven over by heating the residual liquid; the process gives a 90 per cent. yield of the crude product, which may be rectified by distillation, the distilling



flask being heated by the hand and the vapour again passed through sulphuric acid and condensed in a freezing mixture. The residue from the oxidation is a mixture of chlorosulphonic acid and pyrosulphuryl chloride; it may be separated by fractional distillation, or may be used, without separating, for sulphonations.—W. J. P.

#### Rules for the Preparation of Standard Sperm Candles.

The Metropolitan gas referees have recently issued the following particulars concerning the methods to be adopted in manufacturing standard sperm candles for photometric work:—

(1.) All candles to be used in the testing places shall be made with the materials hereinafter prescribed, and shall, when made, be examined and certified by the gas referees.

(2.) The wicks shall be made of three strands of cotton plaited together, each strand consisting of 18 threads. The strands shall be plaited with such closeness that, when the wick is laid upon a rule, and extended by a pull just sufficient to straighten it, the number of plaits in four inches shall not exceed 34, nor fall short of 32. Each wick shall be of suitable length, and looped ready for fixing in the mould. After having been bleached in the usual manner and thoroughly washed, the wicks shall be steeped in a liquid made by dissolving one ounce of crystallised boric acid in a gallon of distilled water, and adding two ounces of liquid ammonia. They are then to be gently wrung or pressed till most of the liquid has been removed, and dried at a moderate heat. Twelve inches of a wick thus made and treated shall weigh not more than 6.5, nor less than 6 grains. The weight of the ash remaining after the burning of 10 wicks which have not been steeped in boric acid, or from which the boric acid has been washed out, shall be not more than 0.025 grain. Wicks made in accordance with this prescription shall be sent to the office of the gas referees, by whom they will be examined and certified. The wicks so certified are to be used by the candle-maker in the condition in which they are returned to him.

(3.) The spermaceti of which the candles are made shall be genuine spermaceti, extracted in the United Kingdom from crude sperm oil, the product of the sperm whale (*Physeter Macrocephalus*). It shall be so refined as to have a melting point lying between 112° and 115° F. An account of the method by which the melting point of the spermaceti is to be determined is given in the appendix. Since candles made with spermaceti alone are brittle, and

the cup which they form in burning has an uneven edge, it is necessary to add a small proportion of beeswax or paraffin to remedy these defects. We therefore prescribe that the best air-bleached beeswax, melting at or about 144° F., and no other material, shall be used for this purpose, and that the proportion of beeswax to spermaceti shall be not less than 3 per cent., nor more than 4½ per cent.

(4.) The candles made with the materials above prescribed shall each weigh, as nearly as may be, one-sixth of a pound, and will be found to answer to the following test:—Immerse a candle, taper-end downwards, in water of 60° F. with a brass weight of 40 grains attached to the wick by a small piece of thread. When a further weight of two grains is laid on the butt end of the candle, it will still float; but with a weight of four grains it will sink. As the rate of burning of a candle is affected by the force with which the wick is pulled when it is set in the mould, the strain commonly applied by an experienced maker of candles has been measured, and is found to be about 24 oz. The candles shall be sent to the office of the gas referees, by whom samples from each batch will be examined and tested. Each batch of candles shall be accompanied by a specimen of the spermaceti (unmixed with beeswax) which was used in making them. Packets of candles approved by the gas referees will be sealed by them, and certified for use in the testing-places.

(5.) The candles are to be used by the gas examiners as heretofore, in accordance with the half-yearly "notifications" of the gas referees. The results are to be corrected as usual, on the assumption that for small variations the light of a candle varies directly with its consumption; and if any candle in a packet certified by the gas referees is found by a gas examiner to burn at a rate exceeding 126, or falling short of 114 grains per hour, the testings made with that candle are to be rejected.

#### *Method of Determining the Melting-Point of the Spermaceti.*

As various methods are used by different refiners of spermaceti for determining the melting-point, which lead to different results, it must be noted that the temperatures here given as the limits within which the melting-point of a sample of refined spermaceti should fall—viz., 112° to 115° F.—have been found by the following method, which is known as the capillary tube method:—

A small portion of the spermaceti is melted by being placed in a short test-tube, the lower end of which is then plunged in hot water. A glass tube drawn out at one end into a capillary tube about 1 mm. in diameter is dipped, narrow end downwards, into the liquid spermaceti, so that, when the tube is withdrawn, 2 or 3 mm. of its length are filled with spermaceti, which immediately solidifies. The corresponding part of the exterior of the tube is also coated with spermaceti, which must be removed. The narrow part of the tube is then immersed in a large vessel of water of a temperature not exceeding 100° F. The lower end of the tube which contains the spermaceti should be 3 or 4 inches below the surface, and close to the bulb of a thermometer. The upper end of the tube must be above the surface, and the interior of the tube must contain no water. The water is then slowly heated, being at the same time briskly stirred, so that the temperature of the whole mass is as uniform as possible. When the plug of spermaceti in the tube melts, it will be forced up the tube by the pressure of the water. The temperature at the moment when this movement is observed is the melting-point.

#### *Method of Washing Wicks.*

As it is found to conduce to the regular burning of candles that the wicks should have been as far as possible cleaned and freed from mineral matters, it is recommended that the candle maker, before steeping the wicks, shall wash them first in distilled water made alkaline with between 1 and 2 per cent. of strong liquid ammonia, then in dilute nitric acid containing about 10 per cent. of strong acid, and then repeatedly in distilled water.

### PATENTS.

*Improvements in Milk Testers.* M. Pedersen, Dursley. Eng. Pat. 1888, January 27, 1893.

See under XVIII. A., page 56.

*Improvements in Balances.* W. Thomson, Baron Kelvin, Glasgow. Eng. Pat. 2198, February 1, 1893.

THE invention has reference to improvements in knife edges for balances. Knife edges in this arrangement are not straight, but convex or concave in form, and called for convenience axe edges and sickle edges respectively. The sickle edges may form part of the inner edge of a ring of agate with its inner edge sharp all round. To give great hardness against lateral motion, balance beams may be supported from two sickle-edged hooks by a straight knife edge on one side and a sickle knife edge on the other.

—E. G. C.

*Improvements in the Production of Vacuum and Means for that Purpose.* J. Dewar, Cambridge. Eng. Pat. 439, January 9, 1893.

See under I., page 23.

### INORGANIC CHEMISTRY.— QUANTITATIVE.

*Separation of Lead from Tin and Antimony by Bromine.* P. Jannasch and W. Remmler. Ber. 26, 1893, 1422—1425.

CONTINUING their work on the quantitative separation of the metals of the sulphuretted hydrogen group by means of bromine vapour, the authors describe in this paper the process for the separation of lead from tin or antimony. The apparatus employed resembles a Liebig's drying tube. The body (about 20 cc. capacity) is made of hard glass and is connected at one end with an upright wide tube, and at the other with a long delivery tube bent first upwards and then down at an acute angle. Into this apparatus the weighed substances, in the form of metal or any compound, are brought, and mixed with 6—10 times their weight of sulphur. A current of sulphuretted hydrogen is then passed and the temperature raised, the sulphides finally remaining behind. The addition of 10—15 per cent. of iodine to the sulphur greatly aids the sulphurisation of refractory compounds. Bromine vapour is then led over the sulphides, the volatile bromides formed being distilled off into hydrochloric acid by heating with a small gas flame. Lead bromide remains in the apparatus; it is converted into chloride by means of chlorine water, and precipitated as sulphate. The tin is weighed as dioxide after repeated evaporation with nitric acid. No numbers are given for antimony.—J. W.

*Separation of Lead and Silver in Ammoniacal Solution by means of Chromic Acid.* By P. Jannasch. Ber. 1893, 26, 1500.

ABOUT 0.5 GRM. each of the nitrates of lead and silver are dissolved in 100 cc. of water, with addition of 2 cc. of dilute nitric acid. The boiling solution is precipitated in a large porcelain basin with a boiling 10 per cent. solution of potassium bichromate (about 5 cc. for each half grm. of nitrate); 15 cc. of dilute ammonia (1:3) are then added with constant stirring, and the resulting lead chromate precipitate warmed for 15—20 minutes on the water-bath. The precipitate is filtered off at once on cooling, washed three or four times with cold weak ammonia, and finally with water. The lead chromate is weighed as such.

The ammoniacal filtrate is carefully acidified with nitric acid and the silver precipitated as chloride. The results given are very accurate.—J. W.

**Quantitative Separation of Metals by means of Hydrogen Peroxide.** P. Jannasch. Ber. 1893, **26**, 1496—1499, 2329—2331. P. Jannasch and J. Lesinsky. *Ibid.* 2331—2336.

**Separation of Lead and Silver.**—About half a gram. of each of the salts is dissolved in 50 cc. of water, 2 cc. of strong nitric acid are added, and the lead is precipitated in the cold, as brown hydrated peroxide, with a previously prepared solution consisting of 15—20 cc. 2 per cent. hydrogen peroxide, and 15 cc. concentrated ammonia. After the precipitation, 5 cc. of cold saturated ammonium carbonate solution are added, and the whole stirred for 10 minutes before filtering. The precipitate is washed in the cold first with ammoniacal water and then with water alone. The lead may be weighed directly as oxide by placing the moist precipitate together with the filter paper in a platinum crucible, which is carefully heated in a small air bath of nickel. A stream of oxygen may be necessary to remove the last traces of carbon. Nitric acid is then added, the nitrate formed being afterwards evaporated to dryness and decomposed by careful heating.

The silver is precipitated from the filtrate by means of hydrochloric acid after evaporation and treatment with excess of nitric acid. The results are accurate.

**Separation of Silver and Bismuth.**—This separation is conducted precisely as in the preceding case, with the exception that ammonium carbonate need not be added after precipitation of the hydrated bismuth peroxide. The bismuth is incinerated as above and weighed as sesquioxide. Results fairly good.

**Behaviour of Copper Solutions.**—If a neutral solution of a copper salt is treated with dilute ammonia until the first precipitate just dissolves, an olive-green peroxide is precipitated on addition of hydrogen peroxide. No precipitation takes place if ammonia is present in excess. Tartaric acid, caustic soda, and hydrogen peroxide give a precipitate of cuprous oxide when boiled with a solution of a cupric salt.

**Separation of Lead and Copper.**—The precipitation of the lead is effected in this case with 60 cc. of 2 per cent. hydrogen peroxide and 20 cc. of concentrated ammonia in the cold, 5 cc. of saturated ammonium carbonate solution being afterwards added. The precipitate is washed first with a cold mixture of hydrogen peroxide (1 vol.), concentrated ammonia (1 vol.), and water (6—8 vols.), then with dilute ammonia warmed to 80°, and lastly with water of the same temperature. The lead precipitate is incinerated in a porcelain crucible, converted into nitrate, and weighed as oxide.

The filtrate is evaporated to drive off the excess of ammonia, treated with sulphuric acid, evaporated, diluted, and the copper contained in it finally precipitated as sulphide.

When large excess of hydrogen peroxide is used, the brown flocculent precipitate of hydrated lead peroxide changes to white pearly scales, which may be further treated precisely as in the case of the amorphous precipitate. The results are good.

**Separation of Lead and Zinc.**—The separation is effected in exactly the same manner as the separation of lead and copper. From the filtrate the zinc is precipitated as carbonate after all the ammonium salts have been removed. The zinc oxide obtained on ignition is usually contaminated with silica, which must be determined specially by dissolving the whole in hydrochloric acid, evaporating, &c. Results fairly good.

**Separation of Lead and Nickel.**—The precipitation is effected with 75 cc. of 2 per cent. hydrogen peroxide and 20 cc. concentrated ammonia, subsequent addition of ammonium carbonate being unnecessary. The lead precipitate is treated as in the case of the separation of lead and copper.

The nickel is precipitated by means of caustic soda from a hot solution containing hydroxylamine hydrochloride. Results fairly good.—J. W.

**Determination of Phosphorus by the Molybdenum Method.** F. G. Myhlertz. *Eng. and Min. J.* **56**, 339.

IN the form of a letter criticising a previous paragraph on the above subject, the author thinks it is erroneous to call the turbidity formed on dissolving the ammonium phosphomolybdate in ammonia "an impurity." By its removal, as sometimes recommended, a source of error is introduced, due to a loss of phosphorus amounting to some thousandths of 1 per cent. An examination of the turbidity invariably reveals both phosphorus and iron. It consists probably of a phosphate of iron formed by the contact of ammonium phosphate and minutely divided ferric hydrate upon dissolving the ammonium-molybdate salt in ammonia. The presence of iron is due to incomplete washing previous to dissolving. The formation of the turbidity is obviated by adding citrate of ammonia to the ammonia, when the iron is kept in solution, and, moreover, the final magnesium pyrophosphate is absolutely white, not "almost white," and also free from molybdenum. To remove the turbidity or to allow it to remain introduces an error. The former by loss of phosphorus and the latter by the introduction of iron as ferric pyro-phosphate into the ignited final product.

—A. W.

**Notes on the Estimation of Copper as Copper Sub-Sulphide.** R. Wegscheider. *Monatsh. f. Chem.* 1893, **14**, 315.

THE majority of analytical handbooks contain the statement that sub-sulphide of copper is perfectly stable at high temperatures, provided access of air be prevented, or the ignition be carried out in a current of hydrogen. Pickering, however, and after him Uhl, found that at a red heat sub-sulphide of copper is reduced to metallic copper by a current of hydrogen, sulphuretted hydrogen being formed at the same time. It appears that these statements passed quite unnoticed. A repetition of the experiments gave results which proved that at a dull red heat the interaction between sub-sulphide of copper is practically nil, but at higher temperatures reduction ensues to such a degree as to vitiate the results of the analysis. This reduction always proceeds very slowly, so that if the heating at a high temperature be of sufficiently short duration, the results obtained will be approximately correct. In order to avoid the careful regulation of the temperature required for the ignition of copper sulphide in a current of hydrogen, sulphuretted hydrogen was substituted for the latter. No reduction of the sulphide occurs under these circumstances, even at very high temperatures, but, on the other hand, it was found impossible in this way to effect a complete conversion of the copper sulphide into the sub-sulphide.—C. O. W.

**The Volumetric Determination of Copper by Means of Sodium Sulphide.** A. Borntrager. *Zeits. ang. Chem.* 1893, **17**, 517—524.

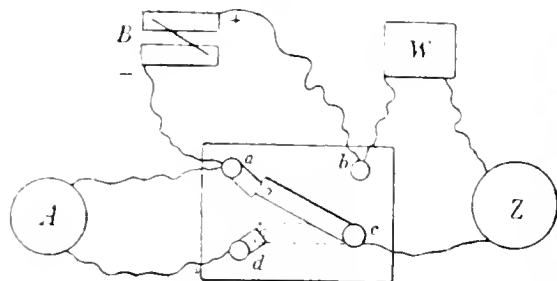
THE process of titrating copper by means of a solution of sodium sulphide is too well known to need description. The author finds that the operation should be carried out cold, as irregularities occur when the solution is used hot, as recommended by Pelouze and Kuenzel. The solution to be titrated should be ammoniacal, and the flask containing it vigorously shaken after each addition of sodium sulphide, as otherwise the precipitated sulphide may not ball together and allow the colour of the supernatant liquid to be seen. The accurate determination of the endpoint must be effected by the use of an external indicator. The author has examined the behaviour of various indicators, such as sodium sulphide and potassium ferrocyanide, depending on the disappearance of the reaction with copper, and that of an alkaline lead solution and sodium nitroprusside depending on the appearance of the reaction with sodium sulphide, and finds that one class cannot be substituted indifferently for the other. The cessation of the reactions for copper occurs appreciably before the beginning of the reactions for a sulphide. Potassium ferrocyanide and alkaline lead solution are the most sensitive indicators for their respective classes. Whichever be adopted, the standardisation of the sulphide solution and the titration of the copper solution under examination must be carried out with the same indicator.

The titration of copper with sodium sulphide can be performed in the presence of zinc, for although a white precipitate of zinc sulphide takes place on the addition of the standard solution, yet this reacts completely with the remaining copper salt on shaking, and the whole of the copper is precipitated before the zinc is permanently separated. The method is therefore valid for the analysis of zinc-copper alloys. The determination of copper in commercial preparations of copper sulphate, is conducted as follows:—5 grms. of the sample are dissolved in water, any iron present oxidised with nitric acid, the solution supersaturated with ammonia, filtered if necessary, and an aliquot portion titrated. Examples of the analysis of various mixtures sold under fancy names for agricultural use, are given, but illustrate no point of sufficient novelty to merit abstraction.—B. B.

*Electrolytic Analytical Estimation and Separation.*  
G. Vortmann. *Monatsh. f. Chemie*, **14**, 1893, 536—552.

When solutions of salts of iron, zinc, cobalt, or nickel are electrolysed under suitable conditions, the metals are precipitated quantitatively; whilst under other conditions separations may be effected, one being precipitated and another not. Bunsen cells, containing instead of nitric acid a saturated solution of potassium dichromate acidulated with sulphuric acid, serve conveniently as electromotors; or preferably small accumulators of 2 volts and 25 ampère-hours power. Two of these are arranged in series as indicated in Fig. 1, where B is the battery, A an ampère-

Fig. 1.

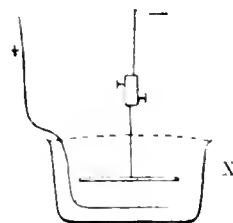


meter, W a variable resistance, and Z the decomposing cell containing the fluid to be electrolysed. When the switch contact-maker C is in the position indicated, the current simply flows through W and Z; but when placed in the position indicated by the dotted line so as to make contact with Z, the ampèremeter A is also in circuit; the resistance of this is practically inappreciable as compared with the rest of the circuit, so that sensibly the same current flows whether A is included in the circuit or not; A is switched in when a current reading is to be made, but is otherwise left out of circuit to avoid damage through over use. The amount of resistance, W, included, depends on the current-density required, i.e., on the amperage per 100 sq. cm. of cathode surface (written N.D. 100 for shortness = normal density per 100 sq. cm.); thus with a cathode consisting of a platinum dish exposing a surface of 125 sq. cm., to obtain a current density of say 0.4 ampère per 100 sq. cm., or N.D. 100 = 0.4 ampère, the resistance must be so adjusted that the current flowing is 0.5 ampère.

Well silvered copper capsules may be employed as cathodes, conveniently of such dimensions as to weigh about 45 grms., holding 150 cc., and exposing about 115 sq. cm. of surface. Fig. 2 represents another form of cell, where the containing vessel is a glass dish, X; the cathode, a silvered copper disc, 5 cm. diameter, furnished with a silvered copper wire 5 cm. long, fixed at the centre of the disc perpendicularly to its plane; and the anode a strip of platinum foil with a platinum wire attached. Platinum discs instead of silvered copper ones are more convenient in certain cases. When the electro-deposition is judged to be complete, the cathode is removed from the fluid, disconnected, washed with water, dried, and

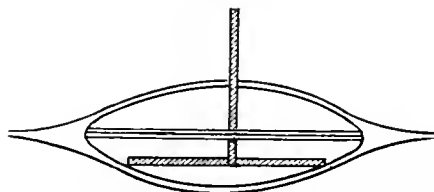
weighed; a similar one is coupled up in its place, the current being passed as before; and so on, until no more gain

Fig. 2.



in weight of the cathode is observed. In order to avoid loss by detaching particles of metal, the cathode is preferably dried inside a pair of watch glasses held by a spring clip, Fig. 3, the upper watch glass having a small

Fig. 3.



hole drilled through its centre to enable the wire to project, and the clip being furnished with a corresponding notch.

Test experiments made with known weights of different metallic salts yielded very satisfactory numbers. Thus, with zinc salts dissolved in caustic soda containing tartrate (N.D. 100 = 0.3 to 0.6 ampère), sensibly exact valuations of the quantity of zinc present were obtained by weighing the metal thrown down on the cathode; and similarly with iron from a similar solution (N.D. 100 = 0.3 to 1.0 ampère). With cobalt, potassium iodide must also be added, otherwise cobalt peroxide is thrown down at the anode; with this precaution and using a concentrated soda solution, fair values are obtained when N.D. 100 = 0.3 to 0.6 ampère. With nickel, the solution with caustic soda and tartrate does not deposit metal at the cathode, although a portion is thrown down as peroxide at the anode; this may be obviated by adding potassium iodide. In potassium cyanide solution only incomplete precipitation as peroxide is brought about; but all the metal present may be obtained as such at the cathode by employing excess of sodium carbonate along with tartrate or citrate as the solution (N.D. 100 = 0.3 to 0.4 ampère). By taking advantage of these differences, nickel may be separated from zinc, iron, and cobalt, the latter being deposited from caustic soda and tartrate solution, whilst the nickel is not thrown down. In similar fashion, zinc and iron may be separated, using caustic soda and tartrate solution, and a platinum dish as cathode. The iron is first thrown down (N.D. 100 = 0.3 to 0.8 ampère) along with a little zinc, from which it may be freed by redissolving and repeating the electrolysis. Another method is to dissolve in excess of potassium cyanide, when zinc only is deposited (N.D. 100 = 0.3 to 0.6 ampère), the ferrocyanide produced not being decomposed by the current. Cobalt, nickel, or copper may be determined in presence of iron by adding excess of ammonia and electrolysis; the precipitated ferric oxide is not decomposed, but need not be filtered off, the other metal being obtained free from iron as deposit on the cathode.—C. R. A. W.

*The Action of Zinc and Magnesium upon Metallic Solutions and the Determination of Potassium thereby.* A. Villiers and F. Borg. *Comptes rend.* 1893, **116**, 1524—1527.

THE method of determining metals by their precipitation in the metallic state by the action of zinc or magnesium



upon their solutions, is practically confined to a limited number, e.g. copper and platinum, and is not without drawbacks, since the precipitates are apt to contain the precipitating metal in the form of an alloy. The purity of the precipitating metal has an influence on the quantity of the metal precipitated, which is not proportional to the amount of foreign metal present. Thus a sample of zinc prepared by the electrolysis of an ammoniacal solution of the sulphate, precipitated 100·92 per cent. of the theoretical quantity of platinum when allowed to act upon a solution of the chloride; another sample, that had been distilled and contained only traces of carbon, precipitated 110·39 per cent., and a third, of commercial quality containing 1·1 per cent. of foreign matter, precipitated 119·12 per cent. of platinum. Magnesium, on the other hand, in the form of ribbon, such as is commonly sold, can be used for determining copper, gold, platinum, or potassium, the last-named having been first obtained as potassium platino chloride. (Commercial aluminium, which would be in some respects convenient as a precipitant, is too impure for analytical use.) The precipitation of copper can be carried out in a slightly acid liquid in the presence of metals of the alkalis and alkaline earths, the precipitate being granular and easy to wash. In the presence of zinc the estimation is inaccurate, as alloys are formed even when the liquid is sharply acid with hydrochloric acid. In the case of potassium, separation is effected as usual as in the form of potassium platino chloride which, is then washed, dissolved in boiling water, the solution made slightly acid with hydrochloric acid, and treated with successive fragments of magnesium until the latter is no longer discoloured while dissolving. The platinum does not stick to the sides of the beaker and is easy to wash free from soluble salts, and may be ignited and weighed in the customary manner. This method of reducing potassium platino chloride is preferable to the use of sodium formate or of formaldehyde. The authors point out that when the platinum instead of the original precipitate is weighed, the accidental presence of the double bromide replacing a portion of the chloride does not affect the accuracy of the results.—B. B.

#### *The Valuation of Commercial Chloride of Sulphur.*

C. O. Weber. *Zeits. ang. Chem.* 1893 [21], 22.

See under V., page 33. (Manufacture of India-Rubber Waterproof Cloth.)

### ORGANIC CHEMISTRY.—QUALITATIVE.

*Basic Salicylate of Bismuth.* Schubardt. *Pharm. Zeit.* 1893, 38, 250.

See under XX., page 57.

*A New Test for Eserine, and a Green Colouring Matter derived from that Alkaloid.* S. J. Ferreira da Silva. *Comptes rend.* 117 330—331.

A FRAGMENT of eserine (or a salt thereof) the size of a grain of sand is placed in a porcelain capsule and dissolved in a drop or two of fuming nitric acid. The clear yellow solution becomes darker and finally orange on heating on the water-bath; on evaporating to dryness with stirring, the residue becomes a pure green substance soluble in water or strong alcohol with green colour. From these solvents it separates, apparently unchanged, on evaporation. Sulphuric acid dissolves it to a green-coloured non-fluorescent solution. 0·005 grm. of alkaloid can thus be readily detected. The author proposes to term the green colouring matter *Chloreserine*.—C. R. A. W.

#### *Detection of Nitronaphthalene in Mineral Oils.*

N. Leonard. *Chem. News*, 68, 297.

NITRONAPHTHALENE, the  $\alpha$ -derivative, is frequently added to mineral oils for the purpose of removing the fluorescence

or "bloom." The following method for its detection depends upon the reduction of nitronaphthalene to naphthylamine.

A small quantity of the oil is gently warmed with zinc dust and dilute hydrochloric acid, and the mixture agitated from time to time. During this process the fecal odour characteristic of  $\alpha$ -naphthylamine will be perceived. After the reduction is complete, the acid aqueous liquid is drawn off by the aid of a separator. A portion of this liquid, when neutralised with ammonia, gives with boric chloride a blue precipitate, which rapidly becomes purple. The remainder of the solution may be rendered alkaline with soda and extracted with ether. The latter is then evaporated, and the residue dissolved in a little alcohol. On the addition of a drop of a solution of sodium nitrate, acidified with acetic acid, a yellow colour is produced, which is changed to crimson by hydrochloric acid.

#### *The Use of Furfural for the Detection of Sesame Oil in Oil Mixtures.* A. Villavechia and G. Fabris. *Zeits. ang. Chem.* 1893, 17, 505—506.

THE well-known reaction with sugar and hydrochloric acid which has long been in use for the detection of sesame oil has been studied by the authors, who have identified various constituents of the oil other than the glycerides forming its bulk, and now give a description of such bodies as they have succeeded in isolating. By preparing a barium soap from sesame oil, extracting with alcohol, evaporating the extract to dryness, and again extracting with petroleum ether, the following substances have been obtained.

(1.) A higher alcohol of the formula  $C_{25}H_{52}O$ , with a melting point of  $137^{\circ}C$ , and a rotatory power of  $[\alpha]_D^{20} = -34^{\circ}23'$  where  $c = 5\cdot013$ .

(2.) A crystalline substance of the formula  $C_{11}H_{22}O_2$ , with a melting point of  $123^{\circ}C$ , and a rotatory power of  $[\alpha] = +68\cdot36$  where  $c = 24\cdot15$  in chloroformic solution. This substance is called by the authors *sesamin*. A similar body has been isolated from sesame oil by Tocher (*Chem. Zeit.* 1893, 121), and has had assigned to it a melting point of  $118^{\circ}C$  and the formula  $C_{15}H_{30}O_2$ .

(3.) A thick, non-crystallisable oil, free from nitrogen, and containing the substance which causes sesame oil to give its characteristic reaction with sugar and hydrochloric acid. A very small quantity of this substance when dissolved in alcohol gives a strong crimson colour with these reagents.

The authors attribute the colour reaction to the agency of furfural produced by the action of hydrochloric acid on sugar, and accordingly substitute the former for its raw materials in carrying out the reaction.

Inasmuch as furfural itself gives a violet shade with hydrochloric acid, it is necessary to use a dilute solution, e.g. 2 per cent., in alcohol. 0·1 cc. of the furfural solution is placed in a test-tube and 10 cc. of the suspected oil and 10 cc. of hydrochloric acid, sp. gr. 1·19, added, the mixture being shaken for half a minute and allowed to settle. Should sesame oil be present, even in smaller quantity than 1 per cent., the aqueous layer at the bottom of the tube becomes of a definite carmine colour. In the absence of sesame oil the lower layer is either colourless or has, at most, a dirty yellow colour, which may appear when a rancid olive oil is being tested. A modification of the test consists in using only 1 cc. of hydrochloric acid, and, after the shaking mentioned above, inducing the separation of the oily and aqueous layers by the addition of 10 cc. of chloroform. The process has been tried on a number of animal and vegetable oils, and its reliability ascertained.

—B. B.

#### *Note on an Abnormal Melting Point.* E. J. Bovan. *Analyst.* 1893, 18, 286—287.

IN the course of an examination of a crystalline fatty body obtained from pure lard by fractional crystallisation from ether, the melting point was determined by filling capillary tubes with the molten substance and proceeding in the

usual manner. When an observation was made with a tube which had been filled only a few minutes previously the melting point of the substance was found to be 47°; but when a tube which was filled at the same time was used for an observation on the following day the melting point was found to be 61.5°. After the substance had become solid in this second tube, it was found to immediately melt when the tube was immersed in water at 50°. Other experiments made with test-tubes internally coated with the substance, showed that after standing for 24 hours the melting point was above 50°; by melting one-half of the substance in such a tube, allowing it to solidify, and plunging the tube into water at 50°, this half melts whilst the other half remains solid.

It is thus apparent that the substance has two widely differing melting points; that it passes slowly and spontaneously from the state of low melting point to that of high melting point; and that the change from high melting point to low melting point can be brought about rapidly by heating.

When the temperature of the substance is gradually raised the passage from low to high melting point is procured. Thus, a tube of the recently-heated substance is immersed in water at 43°: the substance remains solid, but melts when the temperature of the water has reached 47°; when the temperature rises to 53° the substance is again solid, and finally melts at 62°. Compare *Allen's Commercial Organic Analysis*, 3 (2), 570.—A. G. B.

## ORGANIC CHEMISTRY.—QUANTITATIVE.

*Adulteration of Olive Oil.* V. Oliveri. *Le Staz. speriment.* agr. ital. 1893, 24, 387.

See under XII., page 45.

*The Analysis of Beeswax.* G. Buchner. *Chem. Zeit.* 1893, 17, 918.

Hübl's method is, for practical purposes, the easiest and best for the analysis of beeswax. It is, however, possible to produce mixtures containing no trace of beeswax, which still give normal results by Hübl's method. Such a mixture may contain, for instance, 35 stearic acid, 165 Japan wax, 300 ceresin or paraffin wax. Such mixtures may even show the proper melting point and right specific gravity. In the first instance the wax should be remelted in distilled water, and if the latter exhibit an acid reaction (as is often the case with bleached wax), this operation must be repeated several times. The dried wax is then subjected to Hübl's test.

*A wax giving normal numbers.*—Acid number (19—21), 20; ether number (73—76), 75; saponification number (92—97), 95; acid number (3.6—3.84), 3.75. This may be a pure beeswax or a mixture. To decide which, the following addition tests are applied:—

1. *Stearic acid test.*—Boil 1 gm. of wax with 10 cc. of 80 per cent. alcohol, cool to 18° or 20° C. filter, add water to the filtrate and shake. Stearic acid separates out in flakes and floats on the surface; the liquid becomes clear, 1 per cent. of stearic acid may be detected in this manner. If from 7 to 8 per cent. of the acid are present, it separates out as a cream-like mass.

2. *Rosin test.*—5 grms. of the wax are boiled for one minute with about 20 grms. of nitric acid (sp. gr. 1.32—1.33), an equal volume of water and subsequently excess of ammonia are added, and the whole is filtered. The filtrate is yellow if the wax be pure; reddish brown, if resin is present. The method will indicate the presence of 1 per cent. of resin.

3. *Glyceride test.*—The residue obtained after the Hübl test, whilst still warm, is evaporated on the water bath until all the alcohol is expelled. Water is then added, the solution is filtered, concentrated, and tested with potassium bichromate. The odour of acrolein indicates glycerol.

4. If the above three tests give negative results, the Hübl test having given normal figures, no ceresin or paraffin wax can be present, and the sample may safely be pronounced pure.

*B. Wax not giving normal numbers with Hübl's test.*—In this case, the wax has certainly been adulterated, except in cases where only the acid number is too high, and in absence of stearic acid or resin. (With chemically bleached wax, the acid number may rise as high as 24). The nature of the adulterant is estimated by the above mentioned tests and their quantities calculated from the figures obtained by Hübl's test. In certain cases, an estimation of the glycerol and of the paraffin wax by Buisine's method, may be desirable. (*This Journal* 1892, 756, 471, 729, 860, 575, 165, also 1890, 771, and 1888, 871).—C. O. W.

*Contributions to the Analysis of India-Rubber Substitutes.* R. Henriques. *Chem. Zeit.* 1893, 17, 916.

See under XIII., page 48.

*Deductions from the Analysis of India-Rubber Articles.* II. R. Henriques. *Chem. Zeit.* 1893, 17, 707.

In a former paper (*this Journal*, 1893, 467) it was shown that mixtures of india-rubber and such substitutes as are prepared by the action of sulphur or chloride of sulphur upon fatty oils, can be qualitatively separated by means of alcoholic potash. In a later paper the nature of these rubber substitutes was investigated (*this Journal*, 1894, see page 47), and it was found that the characteristic component of the "white substitute" is the large quantity of chlorine it contains, while the "black substitute" contains much more sulphur, but no chlorine. With one exception, presently to be mentioned, no chlorides are used at present, or are ever likely to be used in the manufacture of india-rubber articles. The chlorine which alcoholic potash extracts from india-rubber articles, together with the substitute, can, therefore, decide the question whether "white" or "black" substitute has been present. The percentage of chlorine in the finished article is, however, much less than it ought to be according to the amount of substitute present. Two samples, containing respectively 5.3 and 12 per cent. of substitute, were found to contain respectively only 0.5 and 0.37 instead of 3.7 and 0.9 per cent. of chlorine. Evidently in the vulcanisation process a large quantity of chlorine is liberated in some form. If the substance extracted from the india-rubber contain no chlorine, it may either be derived from "black substitute" or consist of free oil. In the case of the extract being derived from "black substitute" it must at least contain 10 per cent. of sulphur, but if it consist of free oils, at most only traces of sulphur will be present, as it was ascertained by special experiments that free sulphur at vulcanisation temperature has no perceptible action upon oils. An estimation of the chlorine and sulphur in the alcoholic extract permits the analyst to pronounce as to the presence of "white substitute," "black substitute," or sulphur. If, however, the rubber article has been vulcanised with chloride of sulphur, the india-rubber combines with the chloride of sulphur much in the same way as vegetable oils do, and contains both sulphur and chlorine in about the same proportions in which they form chloride of sulphur. The analysis of two samples of thin rubber sheeting prepared from para-rubber and vulcanised with chloride of sulphur gave the following figures:—

	I.	II.
	Per Cent.	Per Cent.
1. Ignition residue of crude rubber.....	0.46	0.46
2. Sulphur.....	5.19	0.5
3. Chlorine.....	5.61	0.57
4. Extracted by alcoholic caustic soda....	2.9	2.2
5. Ignition residue of extract.....	0.67	0.90

A considerable quantity of the extract consisting of sulphur and chlorine, it follows that india-rubber vulcanised by Parkes' method yields very little extract to alcoholic caustic soda, and india-rubber articles of this description can be analysed by the methods as those vulcanised by the ordinary process. Sample I. was over-vulcanised and rather hard, Sample II. was under-vulcanised and still somewhat sticky. The analysis of a sheet of cut rubber before and after vulcanisation gave the following figures:—

	Cut Sheet Rubber.	
	Unvulcanised.	Vulcanised.
	Per Cent.	Per Cent.
1. Ignition residue.....	0.18	0.18
2. Sulphur.....	..	1.07
3. Chlorine.....	..	0.89
4. Extracted by alcoholic soda ..	1.94	1.66
5. Sulphur in extract.....	..	0.57
6. Chlorine in extract.....	..	0.55
7. Rubber extracted.....	1.94	0.54

A number of commercial articles manufactured from cut sheet (patent) rubber gave the following figures:—

	I.	II.	III.
	Per Cent.	Per Cent.	Per Cent.
1. Ignition residue.....	0.67	..	0.2
2. Sulphur.....	3.68	2.37	1.86
3. Chlorine.....	3.63	2.51	1.33
4. Residue on extraction.....	84.3	92.1	94.5
5. Ignition residue of extract.....	1.8	..	0.88
6. Sulphur in 4.....	1.92	1.77	0.83
7. Chlorine in 4.....	1.31	1.54	1.17
Dissolved:—			
S + Cl.....	4.08	1.57	1.19
Organic matter.....About	12.50	6.5	5.0

Sample No. I. was a specimen of cheap gas tubing (black). Nos. II. and III. were similar tubing, but of better quality. From the alcoholic extract of Sample I., by precipitation with acid and extraction with ether, a heavy oil was separated which on analysis gave the following figures:—

	Per Cent.
Sulphur.....	6.29
Chlorine.....	0.55
Iodine absorption.....	92.0

It will be observed that these figures are almost identical with those obtained in analysing the fatty acids of the substitutes made from colza oil or cotton-seed oil, and there is no doubt that such substitutes were contained in the above samples. It is frequently believed that articles made of cut sheet rubber must necessarily be pure, but as a matter of fact they may contain as much as one-third of the before-mentioned substitutes.—C. O. W.

*Saffron and its Adulterants.* E. Vinassa. Archiv. der Pharm. 1892, 231, 353; Diagl. Polyt. J. 290, 47.

To detect adulteration in commercial saffron, the author recommends the employment of the following tests:—

I. *Microscopic and Micro-Chemical Examination.*—A small quantity of the sample is mounted in paraffin oil and examined under the microscope; if all the small particles

are equally coloured, the sample is genuine, but the presence of white or yellowish grains, resembling those of saffron in form, will indicate adulteration with already extracted saffron.

Pure saffron, treated with concentrated sulphuric acid and examined under the microscope, exhibits blue markings, which gradually change to a dirty violet colour, whereas most of its adulterants show characteristic violet or cherry-red ones.

If the sample be clarified with chloral hydrate and then extracted with water, examination with the naked eye will detect admixture with the grosser adulterants, such as sandal wood, logwood, and safflower. On examining a little under a high power, the form of the pollen grains, being most characteristic of the particular species of plant, will afford a good idea as to the genuineness of the sample or the nature of its adulterant, as will also the presence of hairs, crystals, &c.

II. *Chemical Examination.*—This consists in estimating the amounts of ash and water. A genuine sample should not contain more than 15–16 per cent. of water and 8 per cent. of ash. A larger amount of ash indicates the presence of sand, gypsum, or heavy spar.

For the estimation of the colouring power of the saffron, Procter's method may be employed. This consists in extracting 1 part of saffron with 1,000 parts of water for four hours, and filtering off 50 cc. A 10 per cent. solution of potassium bichromate is then added to 50 cc. of water until the colour of the mixture is equal to that of the saffron extract. As a rule 5 to 6 cc. of the potassium bichromate solution should be necessary.

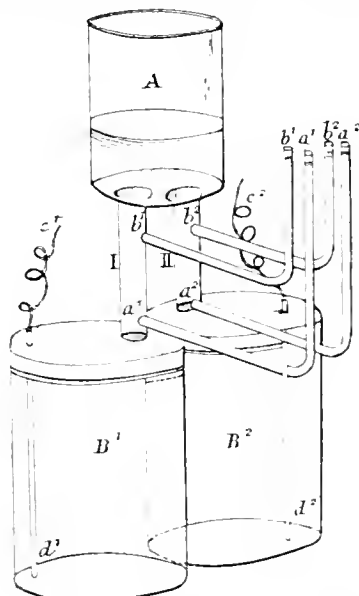
III. *Physical Examination.*—If the saffron be strewn on the surface of water, contained in a shallow dish, the presence of dark points will indicate adulteration with sandal wood, logwood, or safflower. Goppelsroder's method of capillary analysis is of great value in this connection.

—J. G. W.

## ANALYTICAL AND SCIENTIFIC NOTES.

*Electrical Transport of Heat in Electrolytes.* H. Bagard. Comptes rend. 117, 97–100.

By means of the arrangement shown in the figure the author has been enabled to demonstrate that the electrical



transport of heat discovered for metals by Thomson in 1854 can also occur in certain saline solutions. B<sub>1</sub> and B<sub>2</sub> are two glass vessels closed by corks through which pass two glass tubes, I and II, each 1 cm. diameter and 6.5 cm. long, these being inferior prolongations of the upper reservoir A. The lower ends of these tubes are closed with

parchment paper;  $a_1a_1'$  and  $b_1b_1'$ , also  $a_2a_2'$  and  $b_2b_2'$ , are side tubes fused to I and II respectively, and communicating with them by means of very fine holes at  $a_1$  and  $b_1$ ,  $a_2$  and  $b_2$  respectively.  $d_1, d_2$  are two electrodes, by means of which a current can be passed from  $B_1$  to  $B_2$  through I, A, and II, or in the opposite direction. The whole apparatus being filled with the fluid to be experimented with, the upper part is enclosed in the centre of an annular metal box forming a steam jacket, whilst the lower vessels  $B_1$  and  $B_2$  are kept relatively cool by means of a current of water, the tubes I and II being thickly coated with non-conducting material. By-and-bye an equilibrium of temperature is attained amongst the various layers of fluid; at this stage the resistance-ratio of the two columns of fluid between the holes  $a_1, b_1, a_2, b_2$  is ascertained. The current being then passed through the apparatus, heat is developed (Joule effect), which may cause some slight alteration in the value of the resistance-ratio of the two columns owing to want of perfect symmetry; but even if this be the case, the new ratio must be independent of the direction of the current. If, however, a Thomson effect be produced, it will affect each tube in the opposite way, since the current ascends one tube and descends the other; so that the resistance-ratio will be altered in a way dependent on the direction of the current; according as the current passes one way or the other the numerical value of the resistance-ratio will increase or diminish. With a solution of zinc sulphate (23.7 per cent.) the values obtained indicate that the substance is *positive*, i.e., heat travels in the direction of the current through a solution of this salt. Analogous results are obtained with zinc chloride and copper sulphate; but with nickel sulphate the resistance-ratio does not measurably alter, so that with this salt the effect is much more feeble.

—C. R. A. W.

*On the Electric Transference of Heat.* L. Houllévié. Comptes rend. 1893, **117**, 516—518.

It follows from certain experiments by Thomson, Remsen, Rowland, and others that the difference of potential between a conducting body and iron depends upon whether the latter is neutral or magnetic. Assuming this, let us consider a circuit formed by iron and another conductor which is not magnetic, e.g., copper, and let us suppose that the two iron-copper junctions are infinitely far apart. The electromotive force of the system is necessarily zero. But if we place a magnet near one of the junctions, its difference of potential is modified by the magnetism of the iron, while at the other junction its value remains constant. A current should therefore be set up; but this is impossible, because there is no permanent absorption of energy. It is therefore necessary that the variation of the electromotive force produced at one of the junctions in consequence of the magnetisation should be compensated in the rest of the circuit. This compensation must occur in the magnetic parts and it is natural to assume that it is of the nature of a continuous variation of potential between the sections which are unequally magnetised. The author has endeavoured to verify this result experimentally. The essential part of his apparatus consists of a strip of soft iron 1 cm. broad, 0.6 mm. thick, and 50 cm. long, bent into two parallel branches along which the current is passed, while a magnet is used to produce the difference of potential sought for. To detect the effect he employs a thermo-electric pile formed of four bismuth-copper elements arranged in horse-shoe shape. The thermo-electric pile is connected up to a Thomson galvanometer, the sensitiveness of which is such that an E.M.F. of one-millionth of a Daniell cell introduced into its circuit produces upon its scale a deflection of seven divisions.

If a current is passed along the iron strip a considerable permanent deflection (about 50 divisions) is at once produced; this arises principally from the difference of the heating effects (due to the term  $CR^2$ ) at the two poles of the thermo-electric pile. When the direction of the current along the iron strip is reversed, a small deflection (about one division) is observed. This is instantaneous and evidently arises from a very slight (but unavoidable) modification of the magnetic field in the neighbourhood of the

galvanometer produced by the reversal of the current. The galvanometer, however, presently exhibits a slow deviation, quite distinct from the preceding, and amounting in a few minutes to about three divisions, after which the needle remains at rest in its new position. The author is of opinion that this slow deflection reveals the effect expected. There is always an evolution of heat as the current passes from neutral regions to magnetised regions, and an absorption of heat in the contrary case. Hence there exists between two sections unequally magnetised (and in consequence of the magnetisation) a difference of potential in favour of the section which is less strongly magnetised.

—D. E. J.

*The Relations between the Optical Rotatory Powers of Cinchonidine and its Salts, and the Influence of Solvents on Rotation.* Carl A. Schuster. Monatsh. f. Chemie, **14**, 1893, 573—595.

THE author has prepared cinchonidine hydriodide and hydrobromide, salts not hitherto described, and has examined the rotatory powers of solutions of these salts, as also of cinchonidine hydrochloride, acetate, sulphate, and nitrate, and of the free alkaloid itself, using (a) absolute alcohol, (b) 50 per cent. spirit, and (c) water, as solvents. The rotation is always to the left, and is relatively greater in amount in a more dilute solution than in a stronger one; excepting with the sulphate in 50 per cent. spirit, where the opposite is the case; and with the acetate in absolute alcohol, where the specific rotatory power at first rises and then falls with increasing dilution, possibly through dissociation of the acid and base.

Excepting in the case of the sulphate, the rotation is least in absolute alcohol, and greatest in 50 per cent. spirit, intermediate values being obtained in aqueous solution (same degree of concentration throughout). With the sulphate the rotation is greatest in 50 per cent. spirit as before, but less in aqueous than in absolute alcoholic solution.—C. R. A. W.

## New Books.

DICTIONARY OF THE ACTIVE PRINCIPLES OF PLANTS; ALKALOIDS; BITTER PRINCIPLES; GLUCOSIDES: THEIR SOURCES, NATURE, AND CHEMICAL CHARACTERISTICS, WITH TABULAR SUMMARY, CLASSIFICATION OF REACTIONS, AND FULL BOTANICAL AND GENERAL INDEXES. By CHARLES E. SOHN, F.I.C., F.C.S. London: Baillière, Tindall, and Cox, 20 and 21, King William Street, Strand. 1894. 10s. 6d.

THIS work is an oblong-shaped 8vo. volume, strongly bound in dark green cloth, and containing preface, which points to the need of such a work alike for the analyst, professor, research chemist, student, and manufacturer, and to the fact that descriptions are given in the text of some 600 alkaloids, bitter principles, and glucosides, an introduction with explanatory notes, as to the method of using the dictionary, and subject matter, largely tabular, extending over 167 pages. The work closes with two indexes, the last of Substances, Reagents, &c., and the one preceding it, of Botanical terms. A list of Addenda follows the latter index.

Part I. of the work itself is in dictionary form, and herein are grouped together the constituents either of one plant or of a number of botanically or chemically allied plants. In this section the botanical details, general, physical, and chemical details, solubility and reactions, and colour tests, are given.

Part II. is a tabular summary for the purpose of contrasting one compound with another for analytical and other purposes.

Part III. A classification of reactions for the special use of analysts, showing what compounds are known to respond to a given test.

TABELLARISCHE Uebersicht über die künstlichen organischen Farbstoffe und ihre Anwendung in Färberei und Zeugdruck. Von Dr. Adolf Leinze. Dritte Lieferung. Berlin: Verlag von Julius Springer. 1893. London: H. Grevel and Co., 33, King Street, Covent Garden. 6s.

The third part of this tabular review of the progress of the application of the artificial colouring matters to dyeing and calico-printing, has just appeared. The record commences with "Brilliant yellow," and concludes with "Congo brown." It involves the novel expedient of adopting a tabular column consisting of dyed and printed specimens, the other columns of the table containing printed descriptive matter. Below each specimen is given the percentage of colouring matter used in the dye-bath, to produce the tint exhibited, adding at the same time the condition of the dye-bath after the process as to exhaustion or non-exhaustion. In an adjoining column are given data referring to the modes of using the dye; its durability and fastness, solubility, and modes of recognition by test. Another column contains the trade name, with letters signifying the maker's name, and the adjoining column gives the index number in Schultz and Julius' tables, where all information and literature of the colouring matter, can be found, and the final column contains the number of the colour referred to, in the present tables under consideration.

## Prizes.

PROGRAMME OF PRIZES offered by the SOCIÉTÉ INDUSTRIELLE DE MULHOUSE, 1894. Pamphlet, 68 pages. 1893.

THE Industrial Society of Mulhouse offers five foundations and a very large number of medals for competition in 1894. The founders receive a medal along with a sum of money varying between 400 and 5,000 francs. As a rule the medals are of bronze or silver, and in a few instances the medallists are also presented with a sum of money. The conditions which must be observed by intending competitors are as follows:—

1. Foreigners as well as natives are admitted to the competition. The members of the administrative council and the committees of the industrial society are alone excluded.

2. Memoirs, drawings, models, and samples must have the author's motto attached, and should be forwarded, postage paid, before the 15th of February 1894, to the President of the Industrial Society of Mulhouse, along with a sealed letter containing the author's exact name and address.

3. The Society only makes itself acquainted with the names of the successful competitors; at the same time it reserves the right of opening the sealed letter in cases where any additional or indispensable information is necessary.

4. Each competitor, by the mere fact of entering the competition, acknowledges the decision of the Society to be final.

5. Where necessary the Society will send deputies to the works in order to examine the plant and processes.

6. If a problem is not completely solved the Society may award, for the sake of encouragement, a larger or smaller fraction of the prize offered.

7. If several competitors are nearly equal, the prize will be divided between them, equally or unequally, according to the relative values of the solutions.

8. Each competitor retains the right of patenting an invention, but the Society reserves the right of publishing, wholly or in part, the memoirs which have been addressed to it.

9. According to the legislation in most countries, a discovery, invention, or application is not considered new when, in that country, and previous to the date of application for a patent, it has received such publicity that it has already been in use. In consequence, authors are

recommended to obtain letters patent for their discoveries before entering the competition, as the Society declines all responsibility in the matter. In case a prize is awarded for an invention which has not been patented, the author may demand that the publication of his memoir be deferred.

10. The Society does not return memoirs which have been sent in for competition, but the authors may retain copies. Drawings and models which accompany memoirs are retained until a suitable opportunity for returning them presents itself.

11. The relative values of the medals which are awarded by the Society are distinguished as follows:—

Medals of honour.  
Silver medals.  
Bronze medals.

12. The Society reserves the right to award prizes for any work which may be deemed worthy, even although the problem is not included under any of the sections mentioned in the programme.

There are 64 prizes offered in the section of chemical industry, 37 in mechanical industry, 7 in natural history and agriculture, 8 in commerce, 22 in statistical and geographical history, 9 in public health, 4 in fine arts, and 4 in various departments; altogether 160 prizes, including the foundations.

The section relating to chemical industry is divided into 14 sub-sections, in each of which several problems are presented for solution. The sub-sections are:—theoretical work, mordants, colours, fixation of colours, arcometry, drugs, bleaching, printing, decomposition of mordants, and processes of desiccation, steaming, engraving, actinometry, electricity, general.

In the mechanical section there are nine sub-divisions: construction of buildings, steam boilers, motors, and transmission, spinning, weaving, bleaching, printing, dyeing and dressing, distribution of water, electricity, gas-lighting, accidents from machinery (prevention of).

For the special problems which are set and other details connected with them, the programme, which can be obtained, on application from the General Secretary of the Industrial Society, must be consulted.—J. S.

THE FRANKLIN INSTITUTE REWARDS for MERITORIOUS DISCOVERIES AND INVENTIONS. Pamphlet, 3 pages.

THE Franklin Institute of the State of Pennsylvania, U.S.A., may grant, or recommend the grant of, the following medals for discoveries and inventions which contribute to the promotion of the arts and manufactures.

The *Elliot-Cresson Medal* (Gold) may be granted for some discovery in the arts and sciences, or for the invention or improvement of some useful machine, or for some new process, or combination of materials in manufactures, or for ingenuity, skill, or perfection, in workmanship.

The *John Scott Legacy, Premium, and Medal* (20 dollars and a bronze medal) is devoted to rewarding ingenious men and women who make useful inventions. This prize is awarded by the Board of Directors of City Trusts, but the nomination is made by the above institute.

The *Edward Longstreth Medal of Merit* (silver) may be awarded for a useful invention, an important discovery, and for meritorious work in, or contributions to, science or the industrial arts.

The secretary of the institute furnishes full particulars to those who wish their inventions and discoveries investigated with a view to obtaining the above medals.—J. S.

## GUTTA PERCHA.

The Société d'Encouragement pour l'Industrie Nationale is offering a prize of 120*l.* to anyone who will devise a substance capable of completely replacing gutta-percha in one at least of its principal uses, or for a work contributing to the increased production and better exploitation of this gum.

## Trade Report.

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

(From the Board of Trade Journal.)

#### UNITED STATES.

##### Customs Decisions.

The following decisions respecting the construction to be given to Acts of Congress relating to the classification of articles in the Customs tariff, and the application of the Customs laws of the United States, have recently been given by the Custom authorities in that country.

- (1.) Crystallising dishes made of very thin Bohemian glass and used for crystallising salts, chiefly in laboratories;
- (2.) Glass beakers or dishes with glass covers, used exclusively for growing bacilli germs therein; and
- (3.) Glass mortars and pestles, such as are chiefly used in laboratories, are dutiable at 45 per cent. *ad val.* under paragraph 107.

#### CHILI.

##### Exemption from Duty of Cyanide of Potassium.

Mr. J. G. Kennedy, Her Majesty's Minister at Santiago, in a despatch to the Foreign Office, dated the 2nd November, encloses copy with translation of a decree published in the *Official Gazette* declaring that cyanide of potassium and other similar substances may be imported duty free. A Bill, granting the above exemptions, was quickly passed through Congress and was framed on the petition of the representative of the Capell Gold Extracting Company, as an encouragement to gold mining in Chili.

### EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

#### NEW PATENT LAW IN AUSTRIA-HUNGARY.

Mr. P. von Schöller, Her Majesty's Consul-General at Vienna, in a report to the Foreign Office, dated the 29th December, transmits the following summary of alterations recently made in the patent law of Austria-Hungary:—

A new patent law has been promulgated, coming into force on 1st day of January 1894.

In accordance with the stipulations of this new law the joint grant of patents in both sections of the monarchy is done away with, and in future new patents will be granted both in Austria and in Hungary independently.

Patents hitherto granted remain in force.

Commencing January 1st next, patents obtained in Austria are valid only in the provinces represented in the Austrian "Reichsrath."

For the granting of patents there need only be paid the patent tax, without any fee for registration.

The patent tax amounts to 21 florins for each year during the first five years, rising gradually to 105 florins in the 15th year.

Patents obtained in Austria enjoy the like priority in Hungary if the application for the grant of the patent there is made there within 90 days from the date of obtaining the said patent for Austria. This stipulation refers also *vice versa* to patents granted in Hungary.

The period of validity of the patent must be the same in both sections of the monarchy.

Patents from foreign applicants must, commencing January 1st, 1894, be applied for in Austria independently, the same as will be the case with home applicants.

Everything relating to special rights granted to foreigners in consequence of State treaties is in no way modified.—*Board of Trade Journal.*

### GENERAL TRADE NOTES.

#### GERMAN PRODUCTION OF ZINC.

The following note with reference to the production of zinc in Germany is contained in a recent issue of *Kuhlou's*:—

Germany produces more zinc than any other country in the world; the exportation of zinc is accordingly very heavy, amounting to 28,000 tons, against an importation of not quite 9,000 tons. The main zinc-production district is in Upper Silesia, where the metal is made from calamine, and also more and more from zinc blende, by distillation. The smelting of zinc blende has led here, as in other places, to the establishment of very perfect arrangements for utilising and preventing the deleterious effects of the sulphurous acid generated in the roasting of the ore. In Germany, in 1890, 139,000 tons of zinc were smelted, in which work 6,271 men were employed. The value of the output was 62,000,000 marks (15,500,000 dols. approx.), of which 64 per cent. was produced in Upper Silesia, 20 per cent. in the Arnburg district, and the remainder in the Government districts of Düsseldorf and Aachen (Aix-la-Chapelle), and the kingdom of Saxony. The ore in the western parts of the country is in the main zinc blende.

The smelting of zinc is made very difficult, in most localities, by the presence of other metals in the ore. However, by a careful preparation of the ores and purification of the zinc obtained, this difficulty has been overcome. By the use of regenerative gas, heating furnaces, and well-adapted condensers the cost of production has been reduced and loss of metal prevented. With the smelting of zinc a small production of cadmium is connected.

#### DEPRESSION IN THE GERMAN CHEMICAL INDUSTRY.

The *Moniteur Officiel du Commerce* for the 7th December quotes an article from the *Kölnische Zeitung* in confirmation of the bad state of the chemical products industry in Germany and other countries, and in view of the very embarrassed condition of affairs in the principal industrial countries, no other result, it is stated, could very well be expected.

Firstly, as regards America, business in general, but in particular the textile industry, has suffered by reason of the uncertainty as regards the action of the legislature. The export figures published by the different American consulates in Germany show a great diminution; such is particularly the case for the chemical products industry, as may be seen from recent reports by the American consuls at Frankfurt-on-the-Maine and Mannheim. The large factories of chemical products have, as is well known, as their chief customers the weaving works and, more particularly, the dye works and printing works, &c., and when these are affected the chemical products industry sees its principal market closed, and there is no doubt this is precisely what is happening now.

What has been said about America might be urged with stronger reason about England; the great export industry which, in order to impart the latest improvements to manufactured products of all imaginable kinds, uses large quantities of German chemical products, has been considerably affected by the monetary condition of India, by the situation of America, and, in an equal degree, by the serious strikes that have taken place in the coal trade. With respect to Russia, the door is closed, and that country is at present supplied by the large factories of Bâle. The Swiss factories also produce all the patented specialities of the large German companies.

By reason of a general depression of business and a commercial crisis such as exist at present, the chemical industry, as one of the most important industries of the Empire of Germany, must suffer. The reason of such a temporary stagnation in the export of products is extremely difficult to discover, and it is impossible to express an opinion on the subject of the influence exercised by the present state of affairs on the annual yields of the large joint-stock companies. These large establishments have, in fact, many specialities which secure them good returns;

they are well managed in every way and provided with sufficient capital, so that a temporary depression in trade does not produce essential changes in the producing power of these establishments. By reason of the critical condition of business and the difficulty in finding markets, competition has become keener in connection with a reduction in prices. Some factories of chemical products have much difficulty in keeping in full work; they seek to attain this end by offering their goods at a very cheap rate. Prices for most of the principal goods are extremely depressed. The consumption of pharmaceutical products has also somewhat fallen off.

#### LUCIFER MATCH WORKS.

##### *Report of the Committee of Inquiry, October 1893.*

The committee have been unable to trace any cases of necrosis since the special rules were established, beyond the two which have occurred in the factory of Messrs. Bryant and May, yet they are of opinion that danger from that disease exists to all workers where white or yellow phosphorus is used. The committee approve the special rules already in force, but suggest some alterations and addition, chiefly in regard to better ventilation and periodical medical examinations.

Persons are not to be employed in galleries above departments where fumes may rise; and the adoption of means, whereby matches becoming ignited in the boxing departments may be rapidly extinguished, is recommended.

The proposed new rules are then set forth.

#### MANUFACTURE OF CHINA AND EARTHENWARE.

##### *Report of Committee appointed to Inquire into the Injurious Conditions of the Manufacture, and the Proposed Remedies, July 1893.*

The committee were instructed "to make inquiry into the conditions under which the manufacture of pottery is carried on, with the object of diminishing any proved ill-effects in the health of the workpeople engaged therein."

The central town of "the Potteries," Stoke-on-Trent, was selected as the centre of the inquiry, four-fifths of those employed in this industry being employed in and around this centre.

In regard to the medical aspect of the question, it is considered beyond dispute that the potter's occupation is injurious to health and life. The ill-effects are referable to two chief causes—dust and lead poisoning. The former pervades all operations of the industry, while the latter prevails in the glazing and colouring processes after the ware leaves the hands of the potter properly so called. Firing the ware is also injurious, but to a less degree.

1. *Of the Dust of Potters' Clay and of Flint.*—This is noxious by inhalation into the lungs, acting as an irritant, setting up inflammation, and gradually consolidating the organ.

Statistics of life are then given, and the average age reached by potters is given as 48, as against 53 attained by non-potters. Fifty per cent. die of chest complaints, and 21 per cent. of pulmonary consumption. In the case of *pressers*, as distinguished from other potters, 54 per cent. die of chest diseases, and 29 per cent. of pulmonary consumption. The business of *pressers* is more dusty than that of other potters, and that of potters is more dusty than that of those following other avocations; hence the inference is that the inhalation of mineral dust is largely responsible for the mortality. Those exposed to flint dust, as compared with those inhaling clay dust, are very few in number. Those most exposed are the "china scourers,"—all females—who brush off the flint powder which adheres to china after its removal from the oven. Other sufferers are "china placers," "flint sifters," and the few who "flint" the ware before it is "placed." The scourers break down for the most part within a few years.

In all employments where dust is the active agent in inducing disease, the problem is how best to prevent its production, and when produced how to keep it from the workpeople. Its production may be stopped by cleanliness in work, avoiding the scattering of clay, arrangements for

removing waste, cleansing workshops, keeping shops cool so as to prevent the clay drying too rapidly, and by sprinkling the floors with water. Inhalation is best prevented by efficient ventilation and ample working space. Appliances for ventilation and exhaust draughts of air are needed for potters and flint-powder workers, and manufacturers are recommended to employ engineers to solve the problem according to the necessities of each occupation. All these improvements are without avail unless the workpeople co-operate. Hitherto such co-operation has been very imperfectly given.

*Lead Poisoning.*—The committee consider much of the mortality from lead poisoning to be avoidable, and that no rules can entirely obviate its effects, because so much depends upon the worker's personal care and cleanliness. There are several ways in which the poison can be introduced into the system. It is sufficiently proved that dust charged with lead finds its way usually through the stomach, and also, in all probability, through the respiratory passages. When in the form of moist glazes it also enters through the skin. It undoubtedly gains access into the body by—(a) eating food with unwashed hands or partaking of it in the rooms where lead is dealt with; (b) neglect of cleanliness of clothing; (c) allowing glaze and colours to drop about, become dry, and so form dust that gets inhaled; (d) holding the pencil used in painting in the mouth, as is often done by majolica painters; (e) rubbing the eyes with dirty hands; (f) near sightedness, causing the workers to be too close to their work; (g) some constitutional idiosyncrasy producing excessive susceptibility to the poison of lead. At the same time experience proves that many persons using lead glazes and colours work for a long series of years without manifest injury to their health. These are the careful ones. On the contrary many break down quickly; this is especially the case with young women and lads, with whom the influence of sex and age is a factor to be remembered, apart from that of careless habits. Another of some import is that of being ill-fed and debilitated. It is easy to tell at a glance between those likely to resist the action of the poison or to succumb to it by noting their personal cleanliness, their clothes, workplaces, and other surroundings.

Those employed in mixing glazes and colours, if taking ordinary precautions, seldom suffer. The sole use of "fritted" or fused lead has been advocated on the supposition that it is innocuous, and below will be found the results of the committee's investigations under this head.

Statistics prepared by Mr. Hordley, the assistant secretary of the North Staffordshire Infirmary, are then quoted, showing the comparative number of indoor patients treated in that institution in a series of years for lead poisoning among potters. Mr. E. J. Hammersley, secretary of the North Staffordshire Provident Society, has also prepared a table showing the ratio of lead cases among the members of the North Staffordshire Provident Society. In each case the tables show a striking diminution of the number of deaths from plumbism within recent years. It is, however, fair to note that the diminution of plumbism of late years is partly due to the reduced production of majolica ware during the last five years, and also to the enforcement of the order of the Secretary of State of December 1882, prohibiting the taking of meals in majolica painting shops. The higher rate of wages obtainable by dipping-house labour acts as an inducement to women and young persons to engage in it, and also to continue to work even when aware of its ill-effects upon their health; and it becomes a subject for serious consideration whether a periodical examination should not be insisted upon in the instance of all workers with lead, to be made by the certifying surgeons visiting the factories.

*Firing.*—The injurious conditions connected with the firing of the ware are flint dust, lead poison, heat from the furnaces, and the gases and dust evolved. Flint dust belongs to china placing only, the articles within the "saggars" being packed in it. Workers in this department also sift the powdered flint. The leading feature connected with firing is the heat, producing rheumatic affections, exhaustion, and bronchial attacks, and the ill-effects of the occupation



are augmented often by the carelessness of the workers. Further details as to this carelessness, combined with prejudice, in the matter of ventilation of workshops, are then cited.

#### *General Remarks and Suggestions.*

The committee does not think that there has been much improvement in the health of potters, except as to lead poisoning, in the course of the last 20 years. A general improvement in sanitary conditions has been counteracted by increased density of population and the consequent building over of the cottage gardens where the worker possessed a healthy source of interest outside his calling. Workers also live further from their work, and ride to it instead of walking; and the absence of exercise and healthy recreation produce resort to indoor entertainments, where the air is as vitiated as it is in the workshop.

The following suggestions are made:—(1) That no "ground laying" should be done in a room where other occupations are carried on; (2) flint-sifting should be so carried on that the dust is extracted by a fan; (3) shops in basements or below the surface of the ground should be forbidden; (4) lathe-treading should be disallowed for females under 16; (5) "wedging" of clay should not be done by boys under 13 or girls under 16; (6) no married woman should be employed where lead is used: it provokes miscarriage; (7) ware should not be kept on shelves above the heads of workers; and (8) hot closets in pressers' shops should have double doors. Special rules are then laid down, the enforcement of which, in the opinion of the committee, will greatly diminish the injurious effects of the various processes. These rules do not differ materially from those already in force.

The committee think power should be given to condemn such factories and parts of factories as are unfit for work.

With reference to the question of modifying manufacturing processes, the committee have considered the problem of modifying the method of glazing earthenware so as to dispense with the use of lead. At present the raw glaze in which the ware is dipped is a mixture in water of flint, stone, a "fritt" compound of borax and other ingredients, and white lead or red lead. White lead is known to be dangerous from its easy solubility, and two suggestions have been made with a view to meet the evil. One is to use leadless glazes only, which is incapable of universal application. The other is to fuzel all the lead required with the borax and other ingredients into a "fritt" or coarse glass, and to use lead only in that insoluble form. Some firms are now fritting all their lead, and claim it as an improvement; others regard it as impracticable. Mr. A. P. Laurie was therefore deputed to test the solubility of lead fritts in hydrochloric acid (a constituent of the gastric juice) and in bile. Bile was soon proved to be without action, but a solution containing 0.3 per cent. of HCl at 100° F. dissolved a considerable quantity of lead. Comparative experiments revealed that these fritts were better than raw lead, but differed in solubility according to composition. Mr. Laurie's final conclusion is that if the percentage of raw lead and the percentage of borax in a fritt do not amount, when added together, to more than 40, the fritt may be regarded as harmless; but if they amount to more than 40 it is probably too soluble to be safely used. The committee recommends manufacturers to continue their experiments in fritted lead, both with a view to obtaining a satisfactory glaze and to rendering the lead therein as insoluble as possible. In conclusion, the committee urges the desirability of providing mess-rooms for the workers, who, under the new rules, are prohibited from having meals in the work-rooms, and of instituting a periodical medical examination of workers in processes where lead is used or poisonous dust is generated.

#### REPORT OF THE CHEMICAL WORKS COMMITTEE OF INQUIRY, OCTOBER 1893.

The report of the committee appointed by the Home Secretary to inquire into the dangers to life, limb, and health attending employment in chemical works has just been issued. The scope of the inquiry included the conditions of labour in chemical works, the dangers to life

and health of the workpeople employed therein, and proposed remedies for these dangers. The exact questions which the committee was appointed to consider were:—

"(1.) How far the manufactures, as at present carried on in chemical works, injuriously affect the health of the workpeople; and how far the injurious effects depend upon the age and sex of the operatives.

"(2.) What means can be adopted to abate the injurious effects of the manufacture.

"(3.) What special regulations should be adopted to protect vats and other dangerous places and utensils used in the manufacture."

With regard to alkali works, the bleaching powder department is stated to be the most trying of all to those employed in chemical works, owing to the exposure to chlorine gas, under the system which generally prevails at present. In the "Weldon" chambers, which are most commonly used, a thick layer of lime, 4 in. to 6 in., is spread on the floor. The chambers are then closed, and strong chlorine gas is turned on, which is absorbed by the lime. At the end of about four days the gas is turned off, the free gas in the chamber is either drawn off by an exhaust or absorbed by a lime distributor, and the doors are opened. The men, about two hours after, enter to pack the powder. As soon as the powder is disturbed by the shovel it gives off chlorine gas, and no man could work in the chamber without some form of respirator. The packers, in order to work in the chambers, wear a respirator, commonly called a "muzzle." This consists of about 30 folds of flannel, damped and tied tightly over the mouth, with the nostrils free and resting on it. The men are obliged to inhale through the muzzle and exhale by means of the nostrils, otherwise they would be "gassed." The exertion of breathing through the thick folds of flannel shows itself by the red and puffed state of the men's faces, and profuse perspiration on coming out of the chambers, which they are obliged to do at intervals during their work. None but strong, healthy men could stand the work. In another process of making the bleaching powder the "Deacon" chambers are used. Though exposed to some gas, the packers at the Deacon chambers can use a very much smaller "muzzle," which is therefore not nearly so trying. In addition to these, mechanical means have been tried for the manufacture of bleaching powder which would obviate all exposure to the gas. The Hassen-clever process, by mechanical means (this Journal, 1889, 194), is said to be now successfully used in Germany. The committee express a strong hope that the old process may shortly be abolished for some mechanical process. Meanwhile they have suggested certain special rules to obviate in some degree the escape of gas for the benefit of those working in and around the chambers.

The manufacture of bichromate of potash or soda is practically in the hands of three Scottish firms. The committee found that almost all the men working where dust was prevalent had either perforation of the septum of the nose or had lost the septum altogether. Many of those employed suffer also from what are technically called "chrome holes" on hands and arms. These are caused by dust or liquor acting on broken skin, or by the handling of crystals. The committee strongly recommend the use of respirators where any dust arises and the use of gloves where crystals have to be handled. They are of opinion that the due observance of cleanliness on the part of the operatives would largely conduce to the prevention of the evils referred to.

Attention is called to the long hours of work in some departments of alkali works, and the adoption of the eight-hour shift is strongly advised. At the works of Messrs. Brunner, Mond, and Co., this system has been in force for some time, and has resulted in a great improvement in the condition of the health of the men employed. In 1889 before the eight-hour shifts were introduced, 10.12 per cent. of the employes were in the doctor's hands; in 1891 only 5.1 per cent. had to be attended. The committee say:—

"Messrs. Brunner, Mond, and Co. add that the cost of wages paid per ton of alkali produced by them is now no more than it was four years ago, in spite of the fact that

the men employed on shift work are paid the same wages for eight hours as they were formerly paid for twelve. This improvement they attribute partly to the improvements in the apparatus used, effected at considerable cost, and partly to the increased efficiency of the men, due to their better health and spirits."

As regards the question as to how far the injurious effects depend upon the age and sex of the operatives, the committee find that no females are employed in alkali works, and but few boys under 18 years, except in cooperages, plumbers' shops, box-making, &c. Moreover, their inquiries do not lead them to suppose that exceptional mortality, apart from accidents, exists in them. In conclusion, they believe that great good will ensue from the adoption of the proposed rules; and they strongly recommend that these be issued as soon as possible, looking at the unfortunate omission in the Factory Act of 1891, whereby all obligation to fence dangerous pots, pans, &c. was rescinded.

The two medical members of the committee submit a separate report dealing with the medical aspects of these questions considered more fully than is done in the general report.

A special set of rules for persons employed in caustic soda departments has been drawn up, and is recommended for adoption, as well as another set for bichromate works.

They are set forth below, and will be issued to the manufacturers, who will be allowed 21 days from the date of issue in which to make objections.

#### FACTORY AND WORKSHOP ACTS, 1878 TO 1891.

##### SPECIAL RULES.

##### *Chemical Works.*

I. In future every uncovered pot, pan, or other structure containing liquid of a dangerous character, shall be so constructed to be at least 3 ft. in height above the ground or platform. Those already in existence which are less than 3 ft. in height, or in cases where it is proved to the satisfaction of an inspector that a height of 3 ft. is impracticable, shall be securely fenced.

II. There shall be a clear space round such pots, pans, or other structures, or where any junction exists a barrier shall be so placed as to prevent passage.

III. Caustic pots shall be of such construction that there shall be no footing on the top or sides of the brickwork, and dome-shaped lids shall be used where possible.

IV. No unfenced planks or gangways shall be placed across pots, pans, or other structures containing liquid of a dangerous character. This rule shall not apply to black-ash vats where the vats themselves are otherwise securely fenced.

V. Suitable respirators shall be provided for the use of the workers in places where poisonous gases or injurious dust may be inhaled.

VI. The lighting of all dangerous places shall be made thoroughly efficient.

VII. Every place where caustic soda or caustic potash is manufactured shall be supplied with syringes or wash bottles, which shall be enclosed in covered boxes fixed in convenient places, in the proportion of one to every four caustic pots. They shall be of suitable form and size, and be kept full of clean water. Similar appliances shall be provided wherever, in the opinion of an inspector, they may be desirable.

VIII. Overalls, kept in a cleanly state, shall be provided for all workers in any room where chlorate of potash or other chlorate is ground. In every such room a bath shall be kept ready for immediate use.

In every chlorate mill, tallow or other solid lubricant shall be used instead of oil.

IX. Respirators charged with moist oxide of iron or other suitable substance shall be kept in accessible places ready for use in cases of emergency arising from sulphuretted hydrogen or other poisonous gases.

X. In salt-cake departments suitable measures shall be adopted by maintaining a proper draught and by other means to obviate the escape of low-level gases.

XI. Bleaching powder chambers, after the use thereof as far as may be practicable, been drawn off or absorbed by fresh lime, shall, before being opened, be tested by the standard recognised under the Alkali Act. Such tests shall be duly entered in a register kept for the purpose, which shall be produced to an inspector whenever called for.

All chambers shall be ventilated, as far as possible, when packing is being carried on, by means of open doors on opposite sides and openings in the roof so as to allow of a free current of air.

XII. In cases where the co-operation of the workers is required for carrying out the foregoing rules, and where such co-operation is not given, the workers shall be held liable in accordance with the Factory and Workshop Act, 1891, section 9, which runs as follows:—"If any person who is bound to observe any special rules established for any factory or workshop under this Act, acts in contravention of, or fails to comply with, any such special rule, he shall be liable on summary conviction to a fine not exceeding two pounds."

R. E. SPRAGUE ORAM,  
H.M. Chief Inspector of Factories.

#### FACTORY AND WORKSHOP ACTS, 1878 TO 1891.

##### SPECIAL RULES (CHEMICAL WORKS).

##### *Bichromate Works.*

I. In future every uncovered pot, pan, or other structure containing liquid of a dangerous character, shall be so constructed as to be at least 3 ft. in height above the ground or platform. Those already in existence, which are less than 3 ft. in height, or in cases where it is proved to the satisfaction of an inspector that a height of 3 ft. is impracticable, shall be securely fenced.

II. There shall be a clear space round such pots, pans, or other structures, or where any junction exists a barrier shall be so placed as to prevent passage.

III. No unfenced planks or gangways shall be placed across pots, pans, or other structures containing liquid of a dangerous character.

IV. Respirators suitable for protection of nostrils and mouth shall be provided where injurious dust or noxious fumes may be inhaled.

V. The lighting of all dangerous places shall be made thoroughly efficient.

VI. Inasmuch as dust is the principal cause of the various evil results to workers in chromium compounds, all due means shall be taken to limit in every way the formation of dust.

VII. Gloves of some waterproof material shall be provided for the use of workers who handle the crystals.

VIII. Sufficient lavatory accommodation, with hot and cold water, soap, nail brushes, and towels, shall be provided.

IX. In cases where the co-operation of the workers is required for carrying out the foregoing rules, and where such co-operation is not given, the workers shall be held liable in accordance with the Factory and Workshop Act 1891, section 9, which runs as follows:—"If any person who is bound to observe any special rules established for any factory or workshop under this Act, acts in contravention of, or fails to comply with, any such special rule, he shall be liable on summary conviction to a fine not exceeding two pounds."

R. E. SPRAGUE ORAM,  
H.M. Chief Inspector of Factories.

#### PROPOSED NEW SPECIAL RULES FOR CHEMICAL WORKS.

A meeting of the Chemical Section of the London Chamber of Commerce, was held at the office of the Chamber, in Eastcheap, on Friday, January 12, to consider the new special rules for chemical works proposed by the Home Office. Mr. Charles Wightman, Chairman of the Section, presided.

The CHAIRMAN said that he considered the rule demanding the covering of all vessels unworkable if strictly interpreted. It was not practicable to cover all vessels in which minor

chemical operations were carried on. He suggested that a resolution should be proposed agreeing to a recommendation of the sub-committee, that the authorities should be requested to alter Rule 1, so as to read:—

*"Every uncovered pot, pan, or other structure, containing liquid of a dangerous character, shall be so constructed as to be at least three feet in height above the ground or platform, or shall be securely fenced to the satisfaction of the inspector."*

Instead of as at present:—

*"Every uncovered pot, pan, or other structure, containing liquid of a dangerous character, shall be so constructed as to be at least three feet in height above the ground or platform. Those already in existence which are less than three feet in height, or in cases where it is proved to the satisfaction of an inspector that a height of three feet is impracticable, shall be securely fenced."*

This suggestion was unanimously carried, as was also one prefixing the word "uncovered" to the word "pots" in the second and fourth rules. A letter was then ordered to be written to the Home Office, expressing the Section's high appreciation of the rules, trusting that they would fulfil the objects aimed at, and suggesting the "very minor alterations" already described.—*Chemist and Druggist*.

#### THE PROPOSED NEW TARIFF LAW.

Since publishing the abstract of the new tariff Bill prepared by the Ways and Means Committee (this Journal, Dec. 1893, p. 1069), a few changes have been made by the committee. Those affecting the mineral industries are as follows:—

Magnesian firebrick, 1 dol. per ton. Pottery classification changed and rates made 35 and 40 per cent. Ferromanganese, from 22½ per cent. to 10 per cent.

Common black plate, from 35 per cent. *ad valorem* to a specific duty of nine tenths of a cent. Tin plate from an *ad valorem* duty of 40 per cent. to a specific duty of 1½ cents per pound, taking effect October 1st. Wire, from 30 to 35 per cent. Lead sheets, &c. made specific at 1½ cents per pound. Petroleum, crude or refined, is made free if the country producing it admits ours free.

The committee has also decided to recommend that the new law take effect June 1st, instead March 1st, as originally proposed.—*Engineering and Mining Journal*.

#### THE LEGALITY OF LABORATORY STILL.

The following case, which is of considerable interest, was heard recently at Paisley:—Zechariah John Heys, Zechariah Henry Heys, and Zechariah George Heys, trading under the firm of Zechariah Heys and Sons, makers of dyes, South Arthurlie, Barrhead, were charged at the instance of Mr. Peter Ryder, of the Inland Revenue Office, Paisley, with having contravened the second section of the Acts 9 and 10 Victoria, cap. 90, in so far as on 29th September in their premises at South Arthurlie, they, not being then and there persons known as regular makers of stills, nor being licensed distillers, rectifiers, or compounders of spirits, or vinegar makers, did keep and use a still without having taken out or having in force such license as was required under the said Act, whereby they were liable in a penalty of 50l. Respondents pleaded not guilty, and were defended by Mr. William Becket, writer, Glasgow, while Mr. Shearer appeared for the Inland Revenue authorities. Evidence was led, in the course of which it was stated that the still was used in the laboratory for concentrated methylated spirits so as to produce absolute alcohol, which was afterwards used for testing oils in order to determine the fatty matters in them. The still in question, it was also pointed out, was practically a toy, useful only for experiments in a laboratory. Mr. Shearer, on behalf of the Inland Revenue, said the prosecution was not for the purification of the methylated spirits, but for keeping the still without permission from the necessary authorities. The Bench held the charge proven, and imposed a modified penalty of 17.1s.—*Chemical Trade Journal*.

#### AN AUSTRALIAN COAL DISCOVERY.

An important discovery of coal, which is at the same time an instance of results confirming scientific observations, is noted in New South Wales, where coal of excellent quality has been struck under Port Jackson, which is the harbour of Sydney, at a depth of 2,929 ft. This is the second bore that has been put down in the same locality. The first reached a total depth of 3,095 ft., and at 2,901 ft. a "burnt" seam of coal about 8 ft. in thickness was pierced; but as it was known that some of the coal seams had been disturbed, and as the bore had proved the existence of a thick seam of coal, it was determined to sink a second in further search of the valuable deposit in the same locality, and the result has been, as stated, the discovery of a seam of good bituminous coal 10 ft. in thickness. The bore was made by an American diamond drill. The core of coal is 8 ft. long and 4 in. in diameter; the remainder of the coal was in dust driven up by the action of the water and will be analysed. The seam is known as the Bulli seam. The depth as determined in advance by Professor David was within 30 ft. of the actual depth, though the professor had nothing to guide him within 20 miles of the present bore, which is situated at Cremore Point on the north shore opposite, and about three miles from the principal wharfs. This coincidence must be considered largely as "luck," but at the same time it is a proof of the great regularity of the measures. Probably the condition of the coal as reported in the first bore-hole was due to careless boring grinding up the cores. The nearest worked coal-mine is some 40 miles south of Sydney by rail, and Newcastle, which is the principal coal port, is 70 miles north of Sydney. The bore was put down by the Sydney and Port Packing Coal Company, through the agency of the Government diamond drill, the engineer in charge being Mr. Hooper. The company received assistance from the prospecting fund voted by Parliament.—*Ibid*.

#### NATIVE ANTIMONY.

According to W. L. Watts, in the Eleventh Report of the State Mineralogist of California, which has just been published, a ledge of quartzite and porphyritic rock containing native antimony has been discovered on Erskine Creek, four miles south of Hot Springs, in Kern County. Two tons of metal were taken from a shaft 30 ft. deep and 1½ tons from a cut 8 ft. deep. The native antimony was found in nodular masses, coated with oxide of antimony and clay, which varied in weight from 1 oz. to 300 lb. The two openings from which ore was obtained were about 1,000 ft. apart, on the same ledge. There are many veins carrying sulphide of antimony in the neighbourhood of this ledge, particularly in a southerly direction therefrom, and numerous claims have been located, but are at present awaiting capital for development.—*Ibid*.

#### A NEW SOUTH WALES ANTIMONY MINE.

The Eleanora mine was discovered 13 years ago, and has been worked with varying fortunes until recently, when it has been placed on a substantial footing under charge of Mr. George Smith. At present, says the "Australian Mining Standard," the chief workings are near the main shaft, above the 400-ft. level north and south. Here the vein is on an average 7 ft. wide, carrying 11 dw. to 15 dw. of free gold per ton, and about 15 per cent. of antimony. After being hauled up the shaft the ore is screened; the coarse is hand-sorted and the fines are treated by a May's jigger. The antimony portion of the ore goes direct to the furnace, the remainder to the battery. At the south end of the mine a new main hauling shaft 12 × 4 ft. is being sunk to meet the 400-ft. level south from the No. 1 shaft. Good progress is being made with the work, and an adit is also being driven to drain the mine.

The crushing plant consists of a 25-head battery, which is hand-fed. When the ore has been crushed it passes over large baize tables on to three concentrators. The baize is washed every hour into bins. The material thus saved goes into two large amalgamating barrels, and after most of the gold has been caught, it again passes on to two small

separators. It is only six months since the concentrating plant was erected, yet 12,000 dols. worth of concentrates have been saved and sold to the Wallaroo Company. The tailings carry about 4 dwt. of gold, but these are all being saved for future treatment. As showing the tough nature of the ore, it may be mentioned that the 25-head of stamps only get through 140 tons per week, working full time.

The smelting plant consists of five reverberatory furnaces, four for treating the ore crude and one for reducing the oxide of antimony to white metal. The average production of antimony is over 10 tons per week. This is bagged and sold in London, fetching on an average 21*l.* per ton for crude, and 36*l.* for white metal. The average yield of gold is about 78 oz. per week, not including 3½ tons of concentrates of 9 oz. per ton. Mr. Smith estimates that there are 40,000 tons of tailings on the ground, containing 14,000 oz. of gold, and he is now working out a scheme for their treatment. When the electric power company commences operations it is the intention of the management of the Eleanora Company to erect a new 60-head battery with the best concentrating plant obtainable.—*Ibid.*

#### TEA-SEED OIL.

A writer in the *Indische Mercur* points out that *Camellia oleifera*, Abel, a plant closely allied to the tea-shrub yielding the leaves of commerce, is largely cultivated in China for the sake of the pale bland oil prepared from its seeds. These seeds contain a considerable proportion (10 to 44 per cent.) of oil of saponine, a toxic element, and their oil, unless refined, is, therefore, unsuited for eating. The pressed cakes, containing the bulk of the saponine, are used as a hair-wash, a fish-poison, and an insecticide. The seeds of the Japanese camellia (*C. japonica*) yield an oil which excels as a lubricant. Of tea oil proper (*i.e.*, oil from the seeds of *Camellia theifera*, Griff.), two varieties are recognised, *viz.*, the Chinese and the Assam oil. Chinese tea-seed oil is of the consistency of olive oil, pale yellow, and inodorous. The oil has a specific gravity of 0.917 to 0.927 at 15° C., is insoluble in alcohol, and congeals at -5° C. (?). It is edible, very suitable for soap manufacture, and forms a useful lubricant. Oil of Assam tea seed grown in Java has been investigated recently by Mr. L. van Itallie, city pharmacist, of Rotterdam. The oil is present in the husked seeds to the extent of about 20 per cent., and can be extracted by petroleum and ether. It has an acrid taste, a pale yellow colour, very thin consistency, a specific gravity of 0.920 at 15°, and congeals at -12°. Its iodine number (Hübl) is 88, and its saponification number 194. It contains 91.5 per cent. of fatty acids soluble in water. The chief constituents of the oil were palmitic acid (about 19 per cent.), liquefiable fatty acids (oleic and linoleic acids, about 72 per cent.), glycerin (about 8 per cent.), with traces of volatile fatty acids, lecithin, and phytosterin.

It is not at all unlikely that there may be a future for tea-seed oil in the European markets, but in that case it will be necessary to supply a better class of seed than that which was shown at the auctions. It is doubtful whether it would not be the better plan to send over the oil pressed in Ceylon or India. It appears that some of the merchants to whom samples of the tea-seed have been sent are of opinion that the oil obtained from it would find a ready sale in quantity, in London, at 20*l.* to 22*l.* per ton as a safe quotation. Planters are advised to crush 5 or 10 tons of seed and send the oil over to London, on trial, in packages not exceeding 10 cwt. each.—*Chemist and Druggist.*

#### THE MANUFACTURE OF ALUMINIUM.

U.S. Consular Reports, December 1893, 460.

On June 26, 1893, the Consuls at Birmingham, Bremen, Brussels, and St. Gall were instructed to make investigations and report upon the manufacture of aluminium in their respective districts.

Under date of July 25 the Consul at Bremen reported that the aluminium factory at Hamelingen, near Bremen, the only factory of the kind in his district, "ceased

operations some time ago," and now confines itself to the production of magnesium. The process at this factory for the manufacture of aluminium was too expensive to enable it to compete with factories employing the electric system.

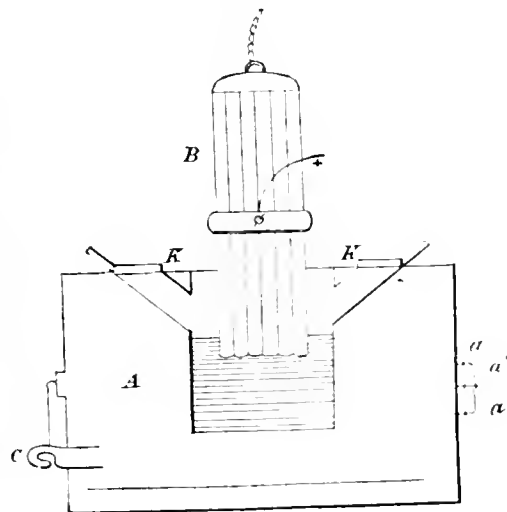
The Consul at Brussels reports that aluminium is not manufactured in Belgium.

In 1888 there was organised in Europe a company or corporation called the Aluminium Industrie-Aetien-Gesellschaft (Aluminium Manufacturing Company). Its authorised capital was 2,000,000 dols., of which 800,000 dols. was paid up. Its purpose was the production of aluminium in large quantities and at low cost to the consumer. The caution of Schaffhausen, Switzerland, was induced by this company to grant it the privilege of drawing from the Rhine above the falls (known as the Rhine Falls), at the town of Neuchâtel, 20 cubic metres of water per second. In all the power thus obtained is equal to 1,000 horse-power. The method employed at Neuchâtel is the electric process of M. Héroult, a French inventor, who is a shareholder in the Neuchâtel company. This process is partly disclosed and can be described; but it also is partly undisclosed and kept a strict secret by the inventor.

What is disclosed of the process is substantially as follows:—Water from the Rhine sufficient to operate two turbines, or wheels, of 600 horse-power each, and a third of 300 horse-power, is conducted through large iron pipes. By means of the two wheels first named two large dynamos are set in motion, which generate an electric current of 14,000 amperes and 30 volts (1,500,000 Watts). This current is run over copper ropes to the electrodes of the apparatus in which the aluminium is produced, a sketch of which is given below. Loose smelted argil (clay), without any other mixture, is then brought into contact with the current at the electrodes. The positive electrode is of carbon (coal plates) and the negative of copper.

At the positive the oxygen of the argil combusts with the carbon and forms carbon oxide; at the negative the copper melted takes up from the argil the aluminium.

A is a large crucible. At *a, a', a'* copper pins admit the electric current to the crucible. B is the positive electrode, consisting of coal plates, joined together at the top by a frame and arm. A chain for raising and lowering the electrode is attached to this arm by a ring. A (the crucible) is covered with graphite plates (K) in which are holes for the admission of material to the crucible. These holes are closed by covers which can be removed to permit the escape of gases. At the beginning of an operation pieces of copper (which become the negative electrode) are put in the crucible. These are then caused to melt by lowering the positive electrode B and consequent production of a heavy current. The clay is next put in the crucible, and quickly melts. The aluminium (more or less alloyed) is drawn off through the aperture C.



By this process all the aluminium alloys can be produced, excepting such as involve metals which volatilize in the melting process.

The different trade qualities of the aluminium made by the Neuhausen company are as follows:—

Quality.	Aluminium.	Silicon.	Iron.
	Per Cent.	Per Cent.	Per Cent.
0.....	99.79	0.06	0.04
I.....	99.61	0.18	0.21
II.....	99.33	0.53	0.14
III.....	99.25	0.56	0.19
IV.....	99.14	0.58	0.28
V.....	96.79	1.84	1.37
VI.....	94.32	3.25	2.43
VII.....	92.84	3.82	3.34

Aluminium of qualities 0 and I can be forged in a hot or cold state; but aluminium of quality II can only be forged with great difficulty, as with 2 per cent. of silicon the metal becomes brittle.

Besides aluminium alloys, the Neuhausen concerns produces (as appears from the above table) practically pure aluminium. The details of the process by which this is done are not obtainable. They are the undisclosed part, of which mention has been made, of the method employed at Neuhausen. That the company in question is able to produce aluminium (both alloyed and pure) in such large quantities as it does and at such little cost is doubtless due in part to the exceptionally favourable arrangement by which its operating power is secured to it. It is due in part also to the experience and inventive genius of M. Héroult.

According to the last annual report of the directors of the Neuhausen company (that for 1892), the company had sold of their products—

Year.	Amount.	
	Francs.	Dols.
1890.....	493,000	98,600
1891.....	1,035,000	207,000
1892.....	2,100,000	420,000

Sales, it is stated, are no longer made by kilograms, but by carloads. It is also stated that in 1892 the dividend to the shareholders was 8 per cent.

But there have arisen in connection with the operation of the Neuhausen company some unexpected difficulties. Shortly after the factory was started the vineyards and vegetation in general of the country about the factory site began to wither. The neighbouring property-owners at once brought actions; but before they came to trial the factory-owners bought at high prices all the property affected. The factory was then enlarged, which served to spread the ill-effects to a territory beyond that which had been purchased.

Certain hotel-keepers then complained and asked that their property be bought also. On July 11th, 1893, at an extra session of the Regierungsrath (the cantonal legislature), before which the whole matter seems to have come, it was decided that the landowners complaining did so with just cause, and that the Neuhausen establishment must be closed, unless means could be devised to absorb or neutralise the gases to which it gave rise and which were destroying vegetation.

An expert—Professor Meister—has been set at work to consider the question of absorption. He states that he has been heretofore employed as an expert in this same line; that he knows the dangerous nature of the gases complained

of, hydrofluoric acid being the principal gas to which the harm done is to be attributed. He states further that, owing to the wide extent of territory involved, complete and effectual absorption will be both very difficult and expensive. He thinks, however, that with plenty of money the task can be achieved.

Meanwhile the matter rests. Should the Neuhausen company be unable, either from lack of financial or other means, to neutralise the injurious gases incident to the operation of their plant, it would seem that they must cease manufacturing.

A branch factory which was opened near that of Neuhausen, but in the canton of Zurich, has been subjected to litigation and is now closed. Whether the closing was because of the litigation or from other causes is not positively known.

#### NEW PROCESS FOR MAKING CITRIC ACID.

##### *U.S. Consular Reports, December 1893, 469.*

Dr. Carl Wehmer, a Hanoverian botanist, is said to have recently discovered that sugar solutions exposed to the action of certain microscopic fungi, the spores of which float in the atmosphere, become transformed into citric acid precisely identical with that extracted from the lemon.

The first experiments made to prepare artificially in this way citric acid are said to have given excellent results, 11 kilos. of sugar producing 6 kilos. of crystallised citric acid (this Journal, see p. ).

The new process has already been patented in several countries, including Italy; and at the factory of Thann, the distinguished chemist Scheurer-Kestner is now carrying on experiments with a view to applying the process on a large scale. Everything tends to show that this new process will assume great development and will make it possible to supply the trade with citric acid at a much lower cost than that actually ruling, and will in all probability supersede in a few years the present method of producing lemon juice and citrate of lime.

The article from which I quote closes by saying: "We make haste to notify our readers, so as to put lemon-growers on their guard, and to prevent new investments being made in this branch of agriculture, which must receive a great blow from this new method of preparing citric acid, and thereby sustain heavy losses."

To show what is meant by "heavy losses," I would refer to my report dated January 8, 1889. In the year 1887, from Messina alone, 4,438 pipes of 130 gallons of lemon juice (used to fix colours in calico printing), and valued at 635,834 dols., were exported. A large quantity of crystallised citric acid was also exported.

Unmerchandiseable lemons are turned to great account, in Sicily more particularly, by extracting the essence from the peel and by converting the juice into concentrated lemon juice. Should this resource now be taken from the Sicilian lemon-grower, he will indeed sustain a heavy loss.

While Florida and California lemon-growers will not be affected by this new discovery, should it ever prove all that is claimed for it, because their industry is still in its infancy, the question appears of sufficient interest to arrest attention.

#### NEW TABLE OILS IN GERMANY.

##### *U.S. Consular Reports, December 1883, 422.*

On account of the great expense and difficulty in procuring pure olive oil for table purposes, there have been many attempts made in Germany to produce from other substances that the olive an oil which, having all the qualities that recommend the olive oil, could be sold at a lower price.

In southern Germany for some years past oil has been produced from the beech-nut. It has given great satisfaction, but has not come into general use because the production has been small, and the oil has never been pushed on the market. One reason why more has not been done in the production of this beech-nut oil has been the great scarcity of the nut in certain years.

The beech-nut contains but 22·77 per cent. of oil, but when the nuts are plentiful, the ease with which they can be gathered, the fact that there is absolutely no other expense except the pressing, and the good prices that have been received for the oil, have made the production of the oil very profitable.

It is only of late that the seeds of the linden tree have been used for the production of oil. According to the report of Dr. C. Müller to the German Botanical Society, this oil has a number of excellent qualities, which would appear to make it certain that the linden seed will hereafter be considered one of the principal sources for obtaining table oil. The linden tree is a regular bearer, so that a large quantity of seed may be counted upon each autumn. The percentage of oil in the linden seed is given at 58. It is maintained that the oil has a peculiarly fine flavour—free from all bitter or aromatic taste—and that it has the appearance of olive oil. It belongs also to the oils which do not evaporate.

Oil made from linden seed will never become rancid. It has no tendency to oxygenate. It will stand a great degree of cold without freezing. Dr. Müller has exposed it to 3° F. below zero without being able to notice any change.

#### THE GRANTING OF PATENTS IN RUSSIA.

*Chem Ind.* 16, 472.

The Russian Board of Trade publishes the following declaration as to the manner of obtaining a patent in Russia, and as to the amount of the patent duties:—

Any person wishing to take out a patent can do so without the intermediation of an agent by presenting a petition to the Board of Trade. The petition to contain the name of the invention, the time for which the patent is required, as also the name, position, and address of the applicant. In the petition there must be given a clear and exact description of the invention, pointing out the novel points in the same (parts of the apparatus or a novel combination of known parts). Applications for patents for

engines, apparatus, or other articles have to be accompanied by a clear drawing, the individual parts lettered so as to correspond to the letters in the subject matter. In all cases where it is impossible to obtain a clear idea of the invention without a drawing, such drawing must be annexed. A petition is to be provided with two stamps each of 80 kopeks value, and, in addition, with a number of similar stamps corresponding to the number of the pages in the petition. At the same time the amount of the patent duty is to be paid in, either at the Board of Trade or at the nearest Government pay office for registration; the receipt for such payment must accompany the petition. The patent duty amounts to 90 roubles for three years, 150 roubles for five years, and 150 roubles for 10 years. When the petition is accepted the applicant receives a certificate to this effect, and the fact of the granting of the patent is published in certain specified newspapers.—H. A.

#### REPORT ON THE POSITION OF THE INDIA-RUBBER INDUSTRY IN 1892.

*E. Clouth, Chem. Ind.* 1893, 16, 193.

Owing to the steady rise in the price of Para-rubber, and to the high prices of cotton and linen yarns and fabrics on the one hand, and the downward tendency of the prices of the finished india-rubber articles on the other, the position of the india-rubber industry was no better in 1892 than in the previous year. Only in a few cases, such as the manufacture of bicycle tyres, and of driving belts from Balata rubber, which for this purpose appears to be especially suitable, were orders at remunerative prices forthcoming. The objectionable use of inferior raw material, the increase in the consumption of substitutes, and the working up of old, already vulcanised rubber (recovered rubber) were the inevitable consequence of this state of affairs, and led to a considerable deterioration in quality of the articles, against which consumers, to their own detriment, are still very indifferent.—C. O. W.

#### NITRATE OF SODA.

*Shipments, Consumption, Stocks, and Prices, from 1886 to 1893.*

		1886.	1888.	1890.	1892.	1893.						
Shipments from South American ports to all parts for the six months ending 31st December.	Tons	274,000	396,000	692,000	373,000	550,000						
Do. for the 12 months ending 31st December.....	"	438,000	761,000	1,028,000	781,000	930,000						
Afloat for Europe on 31st December.....	"	181,000	334,000	514,000	215,000	358,000						
Stocks in United Kingdom ports:—												
—		1886.	1887.	1888.	1889.	1890.	1891.	1892.	1893.			
Liverpool...	Tons	10,700	2,000	3,500	11,000	10,000	7,000	9,000	2,000			
London.....	"	5,300	3,250	1,800	4,100	3,250	3,900	3,500	3,300			
Out ports...	"	17,000	10,750	9,200	14,900	10,750	12,100	7,500	3,800			
						33,000	14,500	21,000	20,000	9,100		
Stocks in Continental ports on 31st December .....						Tons	66,000	63,500	172,000	185,000	162,000	
Consumption in United Kingdom for the six months ending 31st December.						"	24,000	27,000	28,000	33,000	29,000	
Do. in Continent do. do.....						"	112,000	187,000	171,000	192,000	231,000	
Do. in United Kingdom for the 12 months do.....						"	105,000	102,000	119,000	117,000	160,000	
Do. in Continent do. do.....						"	320,000	527,000	666,000	685,000	682,000	
Do. in United States do. do.....						"	60,000	65,000	104,000	100,000	104,000	
Do. in the World do. do.....						"	186,000	694,000	889,000	902,000	846,000	
Visible supply on 31st December (including the quantity afloat, for—												
Europe and Stocks in United Kingdom and Continent .....							"	280,000	412,000	710,000	423,000	460,000
London Spot price on 31st December .....							per Cwt.	9s.	11s.	7s. 7½d.	9s. 1½d.	9s.

—W. MONTGOMERY & CO.'S CIRCULAR.



# THE ECONOMIC POSITION OF THE OPEN-HEARTH STEEL FURNACE.

H. H. Campbell. Paper Read before the English Congress at Chicago. *Engineering and Mining Journal*, December 23, 1893.

THE open-hearth furnace is a regenerative gas furnace in which pig iron, wrought iron, steel, or similar iron products are exposed to the direct action of the flame and converted into steel. As an outlet for the scrap ends and miscellaneous scrap from the steel mills of the country, the open-hearth furnace fills a position of general economic importance. More than this, it has been found peculiarly fitted for producing certain grades of structural and other steels, and furnaces have been operated where scrap was scarce and where it was necessary to use pig iron alone as the regular charge. Thus the process has been raised from a subsidiary to an independent position. In the field of structural work, as in other branches, the Bessemer converter is always a colossal rival. In the United States Bessemer linings are almost universally acid, and therefore in the following detailed comparison of the two processes it will be unnecessary to consider the metallurgical position of the basic converter or the applicability of its product to the stringent demands of modern engineering.

1. In making steel with the phosphorous from 0.06 to 0.10 per cent. the converter can turn out a product at less cost than the open hearth. For many purposes it answers well enough. In comparing it with open-hearth metal of exactly the same contents of carbon, silicon, manganese, phosphorus, sulphur, and copper, no marked difference can be detected by the ordinary methods of testing; but many engineers, after long experience with both metals under various trials and stresses, think that the Bessemer metal suffers a larger number of inexplicable breakages. Experiments on punched test-pieces indicate that this arises from its greater liability to crystallise under shearing stresses.

2. To make Bessemer steel with low phosphorus requires very careful selection of the raw material. If cupolas are used the pig iron will absorb both sulphur and phosphorus from the coke, and the percentage of these elements will be increased by the waste in the converter. The pig iron must therefore contain much less than the maximum of impurity allowed in the steel. It must also contain the requisite amount of silicon for successful blowing, and the scrap used in the vessel should be as pure as the iron. These conditions are not impossible or impracticable, but they enhance the cost of the product considerably and prevent the employment of pure Bessemer steel in competition with common open-hearth metal.

3. No solid fuel is used in the open-hearth furnace, and therefore phosphorus cannot be absorbed from it by the metal, while sulphur will not be unless the coal be unusually sulphurous. There is also more leeway in the selection of stock. In the acid-lined furnace, low- or high-silicon iron can be used, as well as washed pig, charcoal blooms, or basic scrap, while the smaller amount of oxidisable elements reduces the increase in the percentage of phosphorus and sulphur due to waste. In the basic furnaces the ability to remove both these objectionable impurities renders the question of stock a matter of secondary importance.

4. The conditions of Bessemer practice preclude a system of testing during the operation. The vessel may be turned down for a time, but a skull is likely to form on the lining where the metal lies, and this skull, if dissolved by a subsequent hotter charge, may appreciably affect the composition after a test has been taken. Moreover, the chilling of the charge during the testing is a matter of vital importance in the practical working of the mill. The fact must also be noted that the economy of the Bessemer process depends upon continuous and rapid running; any system of interrupted work is bound to raise the cost of the product.

5. The open hearth admits a system of testing without any correlated disadvantages. Such testing is necessarily crude, since the bath is continually changing under the action of the flame, and the results must be obtained quickly. The tests must also be taken before the addition of the recarburiser, and hence will be only relative. After

the addition of the recarburiser, the rapid oxidation of the manganese renders delays out of the question, but in spite of the limited opportunities the tests that can be made are of great value in controlling and regulating the composition of the charge.

6. Uniformity and homogeneity, therefore, are two of the most important factors in the comparison of the merits of the Bessemer and the open-hearth product, but unfortunately no conclusive testimony can be given to the skeptical or even the careful mind. No one conversant with the facts doubts that Bessemer heats can be made which are as homogeneous throughout as any open-hearth charge that was ever melted. No one doubts that in good practice the proportion of Bessemer heats which are not homogeneous is a small percentage of the whole number. But the question is not as to the homogeneity of ninety-nine heats, it is about the quality of the hundredth. And it is not one single test of this hundredth charge that is required but a large number of tests taken from all parts of the cast. One piece of steel differing radically from the rest wipes away all favourable arguments drawn from any number of other tests indicating homogeneity. The terms homogeneity and uniformity are often confounded. The first should mean the likeness of all parts of the same heat, and the second the likeness of one heat to another. It may be said that the second term includes the first, but this would imply a stretch of meaning, as by common acceptance it may be said that certain products are uniformly irregular. What is required in steel products is a combination of uniformity and homogeneity, and obviously no series of records, however long, can demonstrate the existence of this necessary combination, unless all heats have been thoroughly tested and have been found homogeneous.

In the case of low-carbon steels, the open-hearth possesses slightly better facilities for securing homogeneity and uniformity than the Bessemer. For high-carbon steels the conditions of manufacture make the hearth far superior. The metal in the converter is always blown until nearly all the carbon is eliminated, since the stopping of the blow at any definite intermediate point has proved impracticable. All the carbon content of the steel, therefore, must be added in the recarburiser, and absolutely perfect homogeneity can only be secured by absolutely perfect mixing. In the open hearth, on the other hand, the high-carbon steels are made by interrupting the process at the desired stage. It is plain that no mixing is required, so far as carbon is concerned, since about the same quantity of recarburiser will be used for a given manganese, whether high or low steel is being made.

The manganese in the recarburiser distributes itself very evenly under proper conditions. Some variations in physical properties will be caused by differences in finishing temperatures, and some by the fact that the test bars, not being machined, are not exactly round, and hence their area may not be accurately calculated, but the data will sufficiently indicate that the variations are not of practical importance.

The foregoing comparison of the open hearth and the converter may not be a convincing argument against Bessemer metal. It may justify engineers in using the cheap article in many structures, but it will also sustain the more cautious members of the profession who refuse to incur a known or probable risk. In adhering to the safest course, engineers are continually calling for a metal with lower phosphorous. The limit has been 0.10 per cent.; it is now 0.08; soon it will be 0.06; it should be 0.04. Every step in this direction means more work for the open hearth, and it means also the development of the basic furnace.

The stock required in acid work for a pure product is, and always must be, more expensive than for the common grades. The basic-lined furnace renders possible the elimination of the impurities. Like every new device, it has its censors. One of the commonest arguments against it is that good steel cannot be made from bad stock. This statement must be carefully qualified in speaking of the acid process, since the finest open-hearth and crucible-steels may be, and usually are, made from washed pig iron, puddled iron or charcoal blooms, all produced by the application of



a basic process to a more or less phosphoric iron. So far as the assertion is applied to the basic open-hearth process, it is unquestionably wrong; the first piece of good steel made from phosphoric iron relegated this doctrine to the realm of ignorant prejudice. Some engineers prefer not to employ basic metal rather than incur the least risk of unsatisfactory material from a new process. Against such reasoning little can be said, since it has already been admitted that Bessemer and open-hearth metals of the same composition may be unlike; but it is well to consider that a bad heat of steel made by the basic process may be due to ignorance and inexperience in new methods, while such an excuse is out of place when applied to the Bessemer practice, which has been developed for a generation.

### BOARD OF TRADE RETURNS.

#### SUMMARY OF IMPORTS.

Articles.	Year ending December 31st.	
	1892.	1893.
	£	£
Metals.....	21,003,537	20,629,317
Chemicals and dyestuffs.....	6,694,841	6,353,643
Oils.....	7,076,035	7,409,841
Raw materials for non-textile industries.	41,989,612	40,976,930
Total value of all imports ....	423,892,178	405,067,690

#### SUMMARY OF EXPORTS.

Articles.	Year ending December 31st.	
	1892.	1893.
	£	£
Metals (other than machinery) ....	33,043,916	30,806,267
Chemicals and medicines .....	8,584,893	8,695,234
Miscellaneous articles.....	29,615,141	29,347,940
Total value of all exports.....	227,077,053	218,496,246

#### IMPORTS OF OILS FOR YEAR ENDING 31ST DECEMBER.

Articles.	Quantities.		Values.	
	1892.	1893.	1892.	1893.
			£	£
Cocoa-nut..... Cwt.	164,569	155,347	191,380	202,885
Olive .....	20,476	17,206	762,516	652,102
Palm .....	1,058,580	1,169,443	1,169,490	1,458,639
Petroleum .....	130,186,085	155,125,987	2,446,906	2,548,577
Seed .....	22,033	30,616	555,832	732,637
Train, &c..... Tuns	21,121	19,924	415,181	389,848
Turpentine .....	516,597	453,250	590,933	492,236
Other articles .. Value £	..	..	944,224	932,917
Total value of oils...	..	..	7,076,462	7,409,841

#### IMPORTS OF METALS FOR YEAR ENDING 31ST DECEMBER.

Articles.	Quantities.		Values.	
	1892.	1893.	1892.	1893.
			£	£
Copper:—				
Ore..... Tons	91,919	80,798	585,573	485,992
Regulus .....	134,168	118,710	3,222,106	2,822,200
Unwrought ....	35,062	42,018	1,655,912	1,914,312
Iron:—				
Ore.....	3,780,503	4,065,863	2,716,820	2,792,028
Bolt, bar, &c....	75,915	65,820	692,250	593,033
Steel, unwrought..	6,483	8,935	62,486	90,498
Lead, pig and sheet ..	182,782	191,174	1,976,436	1,855,215
Pyrites .....	604,411	612,818	1,063,506	1,065,407
Quicksilver..... Lb.	4,274,274	3,039,338	396,184	341,186
Silver ore..... Value £	..	..	3,032,169	3,076,822
Tin .....	589,365	671,054	2,743,814	2,892,107
Zinc .....	52,793	56,926	1,102,591	1,067,397
Other articles ... Value £	..	..	1,779,998	1,688,140
Total value of metals	..	..	21,669,684	20,629,317

#### IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR YEAR ENDING 31ST DECEMBER.

Articles.	Quantities.		Values.	
	1892.	1893.	1892.	1893.
			£	£
Bark, Peruvian .. Cwt.	100,401	76,356	230,557	148,962
Bristles..... Lb.	3,001,385	3,466,224	439,356	518,757
Caoutchouc..... Cwt.	272,163	293,198	2,982,412	3,328,572
Gum:—				
Arabic.....	68,602	65,619	191,617	169,402
Lac, &c.....	114,833	100,463	445,584	516,016
Gutta-percha ....	45,497	40,490	519,511	303,596
Hides, raw:—				
Dry..... Cwt.	368,191	357,118	946,354	932,043
Wet .....	541,286	589,245	1,138,903	1,248,844
Ivory .....	11,505	16,018	559,083	477,001
Manure:—				
Guano..... Tons	27,874	18,311	189,433	94,721
Bones.....	63,008	44,979	286,452	198,783
Nitrate of soda...	119,561	86,767	1,020,192	806,197
Phosphate of lime ..	314,130	323,257	665,689	694,437
Paraffin..... Cwt.	552,572	768,131	749,704	823,500
Linen rags..... Tons	23,032	26,751	244,065	199,155
Esparto.....	212,967	185,450	1,029,901	870,131
Pulp of wood ....	190,946	215,584	981,025	1,180,310
Rosin..... Cwt.	1,681,393	1,570,173	384,050	394,252
Tallow and stearin ..	1,375,679	1,559,292	1,747,968	2,163,509
Tar..... Barrels	131,886	167,216	80,412	60,921

IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR YEAR ENDING 31ST DECEMBER—*cont.*

Articles.	Quantities.		Values.	
	1892.	1893.	1892.	1893.
Wood:—			£	£
Hewn ..... Loads	2,469,139	2,127,891	1,885,850	4,048,697
Sawn ..... "	5,090,798	4,762,752	11,171,920	10,269,960
Staves ..... "	136,063	131,708	593,539	512,567
Mahogany ..... Tons	56,315	61,719	501,213	559,670
Other articles....Value £	..	..	9,968,912	10,566,630
Total value .....	..	..	41,923,702	49,976,930

Besides the above, drugs to the value of \$17,357, were imported as against \$32,815, in 1892.

## IMPORTS OF CHEMICALS AND DYE STUFFS FOR YEAR ENDING 31ST DECEMBER.

Articles.	Quantities.		Values.	
	1892.	1893.	1892.	1893.
			£	£
Alkali..... Cwt.	56,245	87,703	41,516	79,104
Bark (tanners', &c.) "	380,337	318,617	158,105	121,528
Brimstone..... "	521,589	515,115	147,781	124,797
Chemicals..... Value £	..	..	1,502,294	1,359,729
Cochineal ..... Cwt.	5,456	4,471	33,763	27,226
Cutch and gambier Tons	25,200	24,913	518,395	530,626
Dyes:—				
Aniline ..... Value £	..	..	198,712	188,386
Alizarin ..... "	..	..	335,001	294,046
Other ..... "	..	..	8,529	22,443
Indigo ..... Cwt.	71,344	67,949	1,356,595	1,390,948
Nitrate of potash . "	308,355	242,568	267,931	213,874
Valonia ..... Tons	33,173	34,815	459,429	449,196
Other articles... Value £	..	..	1,622,488	1,551,710
Total value of chemicals	..	..	6,689,539	6,353,643

## EXPORTS OF DRUGS AND CHEMICALS FOR YEAR ENDING 31ST DECEMBER.

Articles.	Quantities.		Values.	
	1892.	1893.	1892.	1893.
			£	£
Alkali..... Cwt.	5,885,609	5,835,664	2,119,440	1,858,157
Bleaching materials "	1,489,700	1,327,669	598,649	551,107
Chemical manures. Tons	319,134	354,106	2,137,811	2,312,067
Medicines..... Value £	..	..	1,013,231	948,753
Other articles... "	..	..	2,715,762	3,025,150
Total value .....	..	..	8,584,893	8,695,234

## EXPORTS OF METALS (OTHER THAN MACHINERY) FOR YEAR ENDING 31ST DECEMBER.

Articles.	Quantities.		Values.	
	1892.	1893.	1892.	1893.
			£	£
Brass..... Cwt.	108,215	115,683	454,537	454,985
Copper:—				
Unwrought..... "	815,822	566,916	2,061,444	1,367,953
Wrought..... "	324,332	340,067	972,997	972,521
Mixed metal.... "	291,582	201,499	729,088	682,777
Hardware..... Value £	..	..	2,194,726	2,048,042
Implements..... "	..	..	1,262,049	1,224,781
Iron and steel.... Tons	2,739,279	2,857,743	21,765,768	20,614,155
Lead ..... "	58,162	48,935	708,510	553,275
Plated wares... Value £	..	..	321,493	306,549
Telegraph wires "	..	..	909,917	1,065,767
Tin ..... Cwt.	112,939	134,800	544,067	606,318
Zinc ..... "	196,211	194,654	178,976	159,994
Other articles.. Value £	..	..	940,344	810,650
Total value .....	..	..	33,043,916	30,866,267

## EXPORTS OF MISCELLANEOUS ARTICLES FOR YEAR ENDING 31ST DECEMBER.

Articles.	Quantities.		Values.	
	1892.	1893.	1892.	1893.
			£	£
Gunpowder..... Lb.	7,935,800	7,682,400	203,327	194,403
Military stores.. Value £	..	..	1,032,535	1,012,247
Candles..... Lb.	18,375,500	19,046,800	344,298	352,918
Caoutchouc.... Value £	..	..	1,215,307	1,186,953
Cement..... Tons	492,615	437,849	902,910	744,926
Products of coal Value £	..	..	1,332,273	1,275,238
Earthenware ... "	..	..	1,904,712	1,823,481
Stoneware..... "	..	..	152,329	163,063
Glass:—				
Plate..... Sq. Ft.	2,158,076	1,684,097	116,467	86,012
Flint..... Cwt.	93,739	83,647	225,528	216,174
Bottles..... "	753,742	639,239	360,343	305,767
Other kinds.... "	222,088	194,314	182,967	158,119
Leather:—				
Unwrought.... "	131,452	145,160	1,245,210	1,371,658
Wrought..... Value £	..	..	332,687	322,331
Seed oil..... Tons	67,232	66,137	1,322,718	1,437,196
Floorcloth ..... Sq. Yds.	16,442,200	17,541,000	712,837	709,690
Painters' materials Val. £	..	..	1,525,152	1,434,661
Paper ..... Cwt.	931,751	909,993	1,579,694	1,474,734
Bags..... Tons	57,618	52,244	401,889	361,662
Soap..... Cwt.	540,799	695,455	586,124	646,102
Total value .....	..	..	29,615,141	29,347,940

## Monthly Patent List.

\* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### APPLICATIONS.

24,406. W. P. Thompson.—From H. L. Fuge, Germany. Improvements in pressure regulators or reducers for gases, liquids, or the like. Complete Specification. December 19.

24,526. H. Reiser. Process and apparatus for cleansing filtering material. Complete Specification. December 20.

24,599. W. Crawford and J. Crawford. Improvements in vacuum apparatus for concentrating vegetable or other substances. December 21.

24,761. The Manchester Oxygen (Brin's Patent) Company, Limited, and W. M. Jackson. Improvements in means for utilising the pressure in cylinders or reservoirs containing compressed gases, and in the application and use of such gases. December 23.

25,011. B. E. R. Newlands. Improvements in apparatus for conveying substances and subjecting them to the action of gases or vapours or air currents, applicable also for drying, heating, cooling, or sifting substances, and for analogous uses. December 29.

1894.

24. J. B. Hilliard. Preventing gases from intermingling while passing through liquids. January 1.

58. A. W. T. Hüllsner and F. W. P. Röhrig. Improvements relating to the process and means for clarifying and filtering liquids. January 1.

70. G. Frère and M. Meslans. Improvements in the method of and apparatus of determining the density of gases. Complete Specification. Filed January 1. Date applied for 30th May 1893, being date of application in France.

232. J. Shenton. Improvements in the combustion chambers of puddling, mill, chemical, and other furnaces. January 4.

358. J. B. Allott. Improvements in drying and evaporating at low temperatures, and apparatus therefor. January 6.

575. A. L. G. Dehne. Improvements in filter presses. January 10.

659. L. Rusden and R. Eeles. Improvements in apparatus for the application of liquid fuel to furnaces. January 11.

697. H. M. Carroll and G. Hill. Improvements in stoking mechanism for gas retorts. January 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.\*

1893.

1780. H. J. E. Jensen. Improvements in and relating to filtering apparatus. January 10.

4076. J. J. Meldrum and T. F. Meldrum. Improvements in or connected with gas producers. December 30.

4112. E. L. Pease. An improvement in the construction of gas washers and ammonia stills. December 30.

5847. C. Sachs. Improvements in and relating to freezing or refrigerating apparatus. December 30.

15,484. E. A. Fehling and A. Steinbart. Improvements in and relating to pyrometers. December 27.

19,750. J. H. W. Ortmann and C. W. C. Herbst. An improved apparatus for sterilising or disinfecting liquids. December 27.

21,858. G. E. O. Lange. Improvements in apparatus for cooling milk and other fluids. December 27.

22,327. J. Bradbury. See Class VII.

### II.—FUEL, GAS, AND LIGHT.

#### APPLICATIONS.

24,112. T. de Zebrowski. Improved process for the solidification of petroleum and apparatus therefor. December 19.

24,422. E. E. Dulier. An improvement in apparatus for destroying smoke. December 19.

24,431. R. Fegan. Improvements in the manufacture of coal briquettes. December 19.

24,432. R. Fegan. Improvements in the manufacture of briquettes from coal-dust or small coal. December 19.

24,524. S. Trivick, C. J. Head and A. Head. New or improved method of treating coke to render same more highly combustible. December 20.

24,603. R. F. English. An improvement in coal briquettes. December 21.

24,632. A. Farinetti and F. F. E. Heyse. A new or improved process for utilising asbestos for vaporising liquids and for distilling and producing gas from mineral oils. December 21.

24,826. J. Blum. Process of and apparatus for the conversion of carbonaceous matter into permanent gas practically free from nitrogen, for heating and illuminating purposes. December 27.

24,856. J. Young. Improvements in obtaining cyanides and ammonia direct from the air or other gases containing nitrogen. December 27.

24,955. B. J. B. Mills.—From La Compagnie Internationale pour l'Exploitation des procédés A. Seigle, France. See Class XII.

1894.

68. F. W. Clark. Improvements relating to the manufacture of gas for illuminating, heating, and other purposes, and to apparatus therefor. January 1.

81. F. Kempster. An apparatus for the utilisation of mineral oils for heating purposes. Complete Specification. January 2.

120. W. B. Fitch. Improvements in candles. January 2.

185. A. McLean. An improved mode of treating peat. January 3.

281. J. Murrie. An improved fuel economiser. January 5.

297. W. Steers. Improvements in fluid fuel injectors. January 5.

534. I. Carr. Improvements in or connected with the manufacture of illuminating gas. January 10.

647. T. Archer. An improved manufacture of fuel. January 12.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

4022. F. Siemens. A process and apparatus for converting hydrocarbons into combustible gas. December 27.

21,707. E. Andreoli. Production of ozone and luminosity by electrical means. December 30.

22,159. C. Bolle.—From J. L. S. Joly. Improvement in the manufacture of candles. December 27.

22,396. R. Langhans. Improvements in the manufacture of incandescent bodies for incandescent gas lighting. December 27.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

## APPLICATION.

1894.

736. J. H. W. Stringfellow. Improvements in the treatment of tar, and in obtaining useful products therefrom. January 12.

## COMPLETE SPECIFICATIONS ACCEPTED.

1892.

21,343. W. J. Wisse and A. Schneller. New or improved treatment of hydro-carbons (or of analogous substances composed mainly of hydrogen and carbon) to decompose them and obtain various products. December 20.

1893.

5284. J. Y. Johnson.—From F. von Heyden. Improvements in the manufacture of guaiacol. January 17.

## IV.—COLOURING MATTERS AND DYES.

## APPLICATIONS.

24,365. P. R. J. Willis.—From W. J. Matheson, United States. A new process of making solid and friable colouring matters from logwood extract and other dye-wood extracts. December 18.

24,543. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. The manufacture of colouring matters and of intermediate products related to the rhodamine series. December 19.

24,713. S. Pitt.—From L. Cassella and Co., Germany. The production of  $\alpha_1$ ,  $\alpha_4$  dioxynaphthalene,  $\alpha_2$   $\beta_1$  disulpho acid, and azo dyestuffs deriving therefrom. December 22.

24,714. S. Pitt.—From L. Cassella and Co., Germany. The production of azo dye-stuffs derived from  $\alpha_1$ ,  $\alpha_4$  azimidonaphthalene. December 22.

24,715. S. Pitt.—From L. Cassella and Co., Germany. The production of dyestuffs from di-azo safranines and amidonaphtholes. December 22.

24,802. J. C. L. Durand, D. E. Inguenin, and A. J. J. d'Andiran. Manufacture of new colouring matters. Filed December 23. Complete Specification. Date applied for July 4, 1893, being date of application in France.

24,870. C. Dreyfus. The manufacture and production of new colouring matters. December 27.

24,888. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of diaceto compounds or analogous disubstituted acid, derivatives of amines, or substituted amines. December 27.

25,012. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. See Class VI.

25,074. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of sulpho acids of the naphthalene series derived from naphthalene  $\beta_1$   $\beta_3$  disulpho acid or naphthalene  $\beta_2$   $\beta_3$  disulpho acid. December 30.

1894.

77. D. M. Murrow and A. Taylor. Improvements in inks, dyes, and the like. January 2.

406. O. Imray.—From The Farbwerke vormals Meister, Lucius, und Brüning, Germany. Improvements in the production of sulphurous aromatic bases and of their sulphonic acids. January 8.

407. O. Imray.—From The Farbwerke vormals Meister, Lucius, und Brüning, Germany. See Class VI.

515. J. Y. Johnson.—From Kalle and Co., Germany. Improvements in and relating to the manufacture of azo dye-stuffs. January 9.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

3495. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of alkylated hydroxynaphthalene sulphonic acids and colouring matters therefrom. December 27.

4612. S. Pitt. From L. Cassella and Co. The production on the fibre by the aid of *p*-amido-diphenylamine of dyestuffs fast to light and washing. January 10.

4613. S. Pitt. from L. Cassella and Co. Improvements in the production of diamidonaphthalene sulpho-acid and amido-naphthol sulpho-acid, and colouring matters therefrom. January 10.

5039. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation. Improved manufacture of colouring matters. January 10.

5316. J. Hickisson. Improvements in the manufacture of coloured marking inks and marking ink pencils. January 17.

5320. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. Improvements in the manufacture of piperazine and its salts. January 17.

5407. S. Pitt. From L. Cassella and Co. The production of new disazo colouring matters. January 17.

7014. H. Kysow. A liquid blue for laundry purposes. January 10.

## V.—TEXTILES, COTTON, WOOL, SILK, Etc.

## APPLICATIONS.

24,638. Comte H. de Chardonnet. Improvements in the manufacture of artificial silk. December 21.

24,758. W. Barraclough and B. Barraclough. An improved textile fabric. December 23.

24,769. J. H. Openshaw. Improvements in the manufacture of cloths known as cotton cords. December 23.

24,904. P. P. Craven. Improved process and machinery for glazing, polishing, and finishing textile fabrics. December 27.

24,978. E. N. Molesworth-Hepworth. Improvements in spinning cotton and other fibrous substances, and in apparatus therefor. December 29.

1894.

72. J. Lowman. New or improved machinery or apparatus for facing or surfacing cotton or other textile fabrics. January 1.

810. A. F. B. Gomess. An improvement in the treatment of textile vegetable fibres. January 13.

#### COMPLETE SPECIFICATION ACCEPTED.

1892.

18,358. A. F. B. Gomess. A new or improved process for the treatment of textile vegetable fibres, and in apparatus connected therewith. January 17.

### VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

#### APPLICATIONS.

25,012. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The production of azo-colours upon fibres. December 29.

1894.

98. A. H. Leggiff. Improvements in machines or apparatus for applying bronze or other powders to the surface of paper, cardboard, cloth, and such like. January 2.

266. G. Wilkie. Improvements in and relating to appliances for mordanting yarn and thread. January 5.

407. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Production of azo-colours on the fibre by coupling in vacuo. January 8.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

977. J. M. Collins. Improvements in machines for dyeing and scouring yarn. January 17.

2009. L. Lang. Improvements in or relating to calico printing. December 30.

2153. O. Ostersetzer. Improvements in printing and dyeing on fabrics. January 17.

3660. H. Ashwell. Improvements in or relating to vats or like vessels for dyeing or treating textile fabrics or garments. January 10.

4612. S. Pitt.—From L. Cassella and Co., Germany. See Class IV.

18,232. O. P. Amend. Improvements in the process of dyeing. December 30.

18,517. J. G. Haslam. Improvements in yarn dyeing machines. January 10.

20,257. E. Labhardt. Apparatus for dyeing and washing loose and spun wool, cotton, and such like material. January 17.

### VII.—ACIDS, ALKALIS, AND SALTS.

#### APPLICATIONS.

24,475. J. R. Wyld and N. Glendinning. Improvements in the treatment of hydrochloric acid for the production of chlorine. December 20.

24,563. M. N. D'Andria. Improvements in and connected with the manufacture of magnesium hydrate. December 21.

24,739. A. J. Boulton.—From S. C. Peuchen and P. Clarke, Canada. Improvements in the concentration or vaporisation of sulphuric and acetic acids and in apparatus therefor. Complete Specification. December 23.

24,856. J. Young. See Class II.

25,023. H. Gaskell, jun., V. C. Driffield, A. Carey, and F. W. Wright. Improvements in and connected with the manufacture of bleaching powder. December 29.

25,024. H. Gaskell, jun., and A. Carey. Improvements in or relating to the manufacture of chlorine. December 29.

1894.

60. I. Werlein. An improved method or process of hardening aluminous materials and the product obtained thereby. January 1.

231. H. R. Angel and W. Smethurst. Making carbonic acid gas of a purer and more economic nature than heretofore by the ordinary processes. January 4.

456. P. C. Choate. Improvements in the art of preparing solutions carrying salts of zinc. Complete Specification. January 9.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

4342. M. N. d'Andria. See Class XIII.

4920. T. Parker and A. E. Robinson. Improvements in the utilisation of chlorides of sodium and potassium for the obtaining of useful products. January 17.

13,722. C. Kellner. Improvements in the manufacture of ammonia, sodium hydroxide, and chlorine, and in apparatus therefor. December 27.

13,928. A. Muller. A method of and apparatus for effecting the absorption of carbonic acid.

22,327. J. Bradbury. Improvements in apparatus and furnaces for concentrating sulphuric acid and other liquids. January 17.

### VIII.—GLASS, POTTERY, AND EARTHENWARE.

#### APPLICATION.

24,682. The Worcester Royal Porcelain Co., Ltd., E. P. Evans, A. Rushton, and H. Hawker. Improvements in the manufacture of pottery or earthenware. December 22.

#### COMPLETE SPECIFICATION ACCEPTED.

1893.

19,699. W. W. R. Nicholls and R. C. Daley.—From The Lansalson China Clay Co. An improved system and apparatus for removing impurities from and refining china clay. January 17.

# IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

## APPLICATIONS.

24,343. M. Koenen. Improvements in the manufacture of concrete blocks, bodies, or structures, and in apparatus therefor. December 18.

24,360. H. Jassoy. An improved method of constructing pavement stones, pavements, landings, and the like. December 18.

24,424. O. Hetterich. An improved process for making wood incombustible. December 19.

24,659. A. Gilechrist. Rollers for indenting cement, granolithic, concrete, or other pavement cements in a semi liquid state. December 22.

24,813. F. F. Scruby and W. H. K. Bowley. A fireproof wall and ceiling covering or decoration. December 23.

25,070. A. J. Boulton.—From D. Belloc and E. Bénard, France. Improvements in the manufacture of cement. December 30.

1894.

15. J. Dougan, J. McMillan, and J. L. Crawford. Improvements in materials and methods for erecting buildings. January 1.

16. J. Walker and R. Peach. An improved cement. January 1.

475. G. W. A. Stein. Improved method of manufacturing hydraulic cement. Complete Specification. Filed, January 9.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

4588. S. McDougall and R. Parry. Improvements in or relating to street pavements, floors, platforms, steps, or the like. December 30.

4737. L. Nathan. Improvements in the manufacture of artificial marble. January 10.

5132. J. Hill. Improvements in the utilisation in the manufacture of Portland cement, of precipitated carbonate of lime forming waste products from chemical works, and in apparatus for employment therein. January 17.

22,782. H. Behrens. Improvements in the manufacture of moulded blocks, slabs, and other articles for use as building materials from cement or other similar substance or composition, and in moulds for producing the same. January 17.

# X.—METALLURGY, MINING, Etc.

## APPLICATIONS.

24,685. W. Wright. Improvement in metallic alloys. December 22.

24,704. D. W. Sugg. Improvements in the manufacture of alloys. December 22.

24,767. W. Jenks. Improvements in furnaces for annealing iron and steel sheets, and in other furnaces employed in the manufacture or treatment of iron and steel. December 23.

24,796. W. Hayward, sen. A new or improved process for recovering tin from tin waste, tinned iron, and steel, and enamel from waste enamelled iron and steel, and furnaces and appliances for the said process and for other purposes. December 23.

24,900. E. Weinberg, R. Carriek, and J. G. Johanson. Improvements in the elimination of the oxides of zinc, iron, and copper from cobalt-oxide, and apparatus therefor. December 27.

24,939. H. L. Sulman. Improvements in the treatment of gold ores. December 30.

25,033. W. Evans. Improvements in or connected with puddling furnaces. December 29.

25,071. A. Trug. Method and apparatus for extracting gold and silver from their ores by the combined action of amalgamation and centrifugal force. Complete Specification. December 30.

1894.

132. T. G. Hunter. An improved process of removing tin from tin scrap or tin-coated metal. Complete Specification. January 2.

328. J. Tinn. An improvement relating to apparatus for galvanising sheet iron and steel. January 6.

364. R. Pearson. An improvement in the process of extracting gold from ores. January 6.

427. G. Robson. Treatment of finely-divided substances, such as crushed ores, slime, tailings, and the like, for the separation and recovery of metals and compounds therefrom, and apparatus therefor. January 8.

437. R. Heathfield and W. S. Rawson. Coating metallic surfaces with zinc and alloys of zinc. January 8.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

572. B. D. Martin and N. Martin. Improvements in the process or method of casting chilled rolls. January 10.

2377. A. Allen, A. Bashforth, and H. Allen. A reheating furnace for steel and iron puddling, and other purposes. January 17.

4001. H. W. Hemingway. Improvements in the manufacture of oxide of iron and in means applicable for use in such manufacture. December 27.

4328. A. Longsdon.—From L. Grambow. A new or improved process for improving the quality of steel armour plates. December 27.

5327. T. M. Ash, H. W. Gill, and L. Green. An improved process for coating or plating non-metallic articles with metal. January 10.

10,096. E. de Pass.—From H. Thofehn. Improvements in the manufacture of metallic oxides and compounds. December 30.

16,180. L. V. J. Lynen. Process and apparatus for the extraction of zinc. January 10.

16,857. A. Raze. Process for avoiding or considerably diminishing the choking and contraction of the mouth of the retort in the manufacture of the "Thomas" steel. December 27.

22,461. J. Cox. A new or improved metallic alloy. December 27.

22,828. R. Haddan.—From B. Jones. Improvements in and relating to cores, moulds, and mould-facings for the manufacture of steel and other metallic castings. December 30.

23,431. C. Raleigh. Improved means applicable for use in extracting gold and silver from auriferous and argentiferous solutions. January 10.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### APPLICATIONS.

24,295. H. F. Hoveler. Improvements in elements for secondary and other electric batteries, and in the manufacture of same. December 18.

24,344. J. A. Kingdon. Improvements in dynamo electric machines. December 18.

24,353. C. K. Mills.—From C. J. Barbier, France. A new or improved preparation of active material for electric accumulators. December 18.

24,389. A. Lehmann. Improvements in electrode plates for secondary batteries. December 18.

24,417. J. B. Torres. Improvements in electric or galvanic batteries. December 19.

24,442. C. K. Mills.—From C. J. Barbier, France. A new or improved electrical accumulator plate. December 19.

24,871. H. T. Johnson. Improvements in dry batteries. Complete Specification. December 27.

1894.

482. H. C. L. Holden. Improvements in secondary batteries. January 8.

494. W. P. Thompson.—From C. L. Coffin, United States. Improvements in forges or furnaces for heating or working metals electrically. Complete Specification. January 9.

496. W. P. Thompson.—From C. L. Coffin, United States. Improvements in apparatus for heating or welding metals electrically. Complete Specification. January 9.

646. W. Walker, jun., F. R. Wilkins, and J. Lones. Improvements in primary voltaic batteries. January 11.

669. C. J. Wollaston. Improvements in electric batteries. January 11.

670. A. Philip. Improvements in the manufacture of electrodes and use of the same. January 11.

835. J. Hargreaves and T. Bird. Improvements in the construction of electrolytic apparatus, and in the application thereof in oxidising, chlorinating, and analogous processes. January 13.

836. J. Hargreaves and T. Bird. Improved means for obtaining metallic bases and their compounds by electrolysis. January 13.

### COMPLETE SPECIFICATIONS ACCEPTED.

1892.

18,039. J. Hargreaves and T. Bird. Improvements in electrolytic cells and diaphragms. January 17.

23,101b. C. A. J. H. Schroeder and H. E. R. Schroeder. New or improved galvanic batteries. December 27.

23,773. R. Niewerth. Improvements in electric smelting, and in furnaces therefor. December 27.

1893.

660. H. F. Kirkpatrick-Picard and E. Thame. An improved method of manufacture of elements or plates for secondary batteries. January 10.

2241. C. A. J. H. Schroeder and H. E. R. Schroeder. High-tension battery. December 27.

3734. A. Hough and O. March. Improvements in secondary battery plates or elements. December 27.

3743. E. H. Liveing. Improvements in the preparation of carbon electrodes for electrolytic operations. December 27.

3744. E. H. Liveing. Improvements in the construction of carbon electrodes. December 27.

4691. The Lithanode and General Electric Co., Ltd., and J. T. Niblett. Improvements in the manufacture and production of elements for secondary batteries. January 17.

6605. E. T. Parker. Improvements in diaphragms for use in electrolytic processes. January 17.

22,311. E. P. Usher. Improvements relating to storage batteries. December 30.

22,312. E. P. Usher. Improvements in storage batteries. December 30.

23,436. H. H. Lake. From The Union Chemical Co. Improvements in apparatus for electrolytically producing soda and chlorine. January 17.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

### APPLICATIONS.

24,461. T. Cordewener and A. de Kunwald. See Class XVIII.—A.

24,556. J. V. Ruymbeke. Process of distilling glycerin, and of treating the residuum of such distillation. December 20.

24,724. V. Contant. A new washing powder or compound. Filed December 22. Date applied for June 28, 1893, being date of application in France.

24,955. B. J. B. Mills. From La Compagnie Internationale pour l'Exploitation des procédés A. Seigle, France. Improvements in the composition of oils for feeding vapour-burning lamps. December 28.

24,956. L. Hatschek. Improvements in the manufacture of disinfectant soap. December 28.

25,001. H. W. Taylor. An improved soap. December 29.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

3469. J. S. Fairfax.—From W. D. Field. Improvements in or relating to compounds of "blown" or aerated oils with pyroxyline. December 30.

5107. H. D. Morgan. Soap cooling frames. January 17.

20,413. H. J. Bull. An improved method or process and apparatus for deodorising oils and fats. January 17.

23,387. W. T. Forbes. Improvements in the process and apparatus for extracting oil and other solvent matters from fibrous plants and other materials. January 10.

23,398. A. J. Boulton.—From A. Gross. Improved manufacture of detergent for washing and cleansing dirty linen. January 10.

## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

### APPLICATIONS.

24,356. H. H. Lake.—From A. C. Girard and E. A. G. Street, France. An improved manufacture of writing pencils. December 18.

24,485. E. Bayley. An improved liquid enamel or glass for laundry use. December 20.

24,695. C. U. King and R. V. Jellicoe. Improvements in materials or compounds adapted for use in varnishes as substitutes for india-rubber, gutta-percha, and like substances, and for other purposes, and the process for their production. December 22.



1894.

71. W. H. Hyatt. Improvements in the manufacture of paint. January 1.

166. O. Imray.—From J. Sachs, C. E. Meier, and M. Gerstendorfer, United States. Improvements in the manufacture of pigments. Complete Specification. January 3.

## COMPLETE SPECIFICATION ACCEPTED.

1893.

2883. A. Macdonald. Improvements in the treatment of galena for the production of lead, lead sulphate, and lead oxide. January 17.

4342. M. N. d'Andria. Improvements in and relating to the manufacture of paints, sulphuric and sulphurous acid from proto-sulphate of Iron. December 30.

4870. A. P. Laurie. Improvements in the preparation of oxide of zinc pigments. January 17.

5316. J. Hiekisson. See Class IV.

5349. M. N. d'Andria. Improvements in the manufacture of red oxide paints. January 17.

9825. E. Edwards.—From J. W. Overton. An improved paint. December 27.

16,703. J. V. Walton. Improvements in apparatus employed in the manufacture of white lead. December 30.

17,142. L. Grote. Improvements in the manufacture of artificial stone preservative paint, and other analogous materials. January 10.

22,294. E. Waller. Improvements in the manufacture of white lead. December 27.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

## APPLICATIONS.

24,463. W. Zahn. Improvements in the art or process of tawing hides or skins. Complete Specification. December 19.

24,468. C. B. Dawney. An improvement in process for treating hides and skins for leather. December 20.

1894.

230. J. McNeill. An improved glutinous composition, applicable to leather, gutta-percha, and cotton belts. January 4.

## COMPLETE SPECIFICATION ACCEPTED.

1893.

4274. W. Becker. An improved process to make leather soft, durable, and waterproof. December 30.

## XV.—AGRICULTURE AND MANURES.

## APPLICATIONS.

24,465. A. R. O. Pieper. Improvements relating to the manufacture of artificial manure. December 19.

1894.

792. M. C. Gunster. Improvements in the manufacture of manure and fertiliser. January 13.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

## APPLICATIONS.

24,718. S. Touyarou. Improvements in the manufacture of sugar and in machinery therefor. December 22.

24,742. E. Shaw. Improved method of and apparatus for boiling sugar and glucose either separately or mixed. December 23.

1894.

136. H. H. Lake.—From P. H. V. Weyde and O. Lugo, United States. Improvements in the manufacture of sugar. Complete Specification. January 2.

## COMPLETE SPECIFICATION ACCEPTED.

1893.

5617. H. H. Lake.—From W. Angele. Improvements relating to the manufacture of soluble starch. December 27.

## XVII.—BREWING, WINES, SPIRITS, Etc.

## APPLICATIONS.

24,548. R. Genge. Improvements in the preparation of malt and in apparatus therefor. December 20.

24,708. F. W. Golby.—From P. Condamin, France. A process for the crystallisation of liqueurs. December 22.

24,880. O. Imray.—From L. Genes, France. Improvements in the manufacture of yeast and alcohol. December 27.

25,052. S. H. Sheldrake. An improved process for the preparation of ale and stout. December 30.

1894.

54. C. E. Ramspott. An improved process for the production of a non-alcoholic beverage. January 1.

507. T. Chandler. A new or improved malt product and process for manufacturing same. January 9.

## COMPLETE SPECIFICATION ACCEPTED.

1893.

228. A. Blake. An improvement relating to the process of malting grain. January 10.

# XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

## APPLICATIONS.

### A.—Chemistry of Foods.

24,461. T. Cordewener and A. de Kunwald. Improvements in and in apparatus for the manufacture of margarine and alimentary fat. Complete Specification. December 19.

24,970. M. Watson. Improvements in cereal leguminous or other vegetable foods. December 28.

### B.—Sanitary Chemistry.

25,040. H. B. Sharp and J. B. Petrie. Improvements in the preparation of compositions adapted for use in the treatment of sewage and foul waters. December 30.

### C.—Disinfectants.

24,531. S. Pitt.—From the Chemische Fabrik auf Actien vorm. E. Schering, Germany. An improved antiseptic disinfectant or deodorant. December 20.

24,709. S. F. Kattenhoy. Apparatus for the production of ozone from phosphorons for disinfecting purposes. Complete Specification. December 22.

24,956. L. Hatscheck. See Class XII.

1894.

147. C. G. Moor. An improved disinfectant. January 3.

## COMPLETE SPECIFICATIONS ACCEPTED.

### A.—Chemistry of Foods.

1893.

4486. W. Clark. Improvements in fluid meats and the process of manufacturing the same. December 30.

4487. W. Clark. Improvements in food preparations and in their manufacture. December 30.

12,617. H. W. Hart. Improved method of treating coffee berries for the extraction and production of their food and health (dietetic) giving constituents. January 17.

21,448. W. B. Walters. A mixing or churning apparatus for use in making butter and condensed milk. December 27.

22,287. M. Kirchberger.—From Ban Heng Hin and Co. Improvements in the treatment of pepper. December 27.

## XIX.—PAPER, PASTEBOARD, Etc.

### APPLICATIONS.

24,503. J. Y. Johnson.—From P. Sohège, France. Improvements in the manufacture of cellulose pulp. December 20.

24,521. C. Beadle. A process for treating paper so as to provide against falsification of documents. December 20.

24,652. W. M. C. Callender. The manufacture of a new material designed to serve as a substitute for bone or celluloid. Complete Specification. December 21.

24,669. J. Craig. Improvements in machinery or apparatus for manufacturing paper. December 22.

1894.

109. C. F. Cross. Treatment of wood for the manufacture of cellulose and other products. January 8.

824. W. White. An improved method of preparing pigment paper for photographic printing. Complete Specification. January 13.

## COMPLETE SPECIFICATIONS ACCEPTED.

1892.

23,603. W. Ibbotson. Coating or covering paper with aluminium and its alloys. December 27.

1893.

22,415. D. Westad. Improvements in machines for making millboard or paste-board from wood-pulp. December 27.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

### APPLICATIONS.

24,459. S. Pitt.—From The Chemische Fabrik auf Actien vorm. E. Schering, Germany. Improvements in the manufacture of laevulose. December 19.

24,604. S. W. Wilkinson. Improvements in or applicable to aromatic hydrocarbons and their manufacture. December 21.

1894.

189. J. F. Challen. Orangine. January 3.

681. F. W. Warrick. An improvement in capsules of ferrous carbonate and ferrous phosphate. Complete Specification. January 12.

## COMPLETE SPECIFICATIONS ACCEPTED.

1892.

23,449. H. Byk. Improvements in the method of obtaining phenols and naphthols, commonly called "salols," from salicylic acid, &c. December 27.

1893.

4191. O. Imray.—From The Society of Chemical Industry in Basle. Manufacture of new pharmaceutical compounds. December 30.

12,617. H. W. Hart. See Class XVIII.—(A).

## XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.

### APPLICATIONS.

24,487. A. Burchett. Improvements in photography. December 20.

24,963. J. Packham. Improvements in producing photographic prints. December 28.

25,002. M. Andresen. Improvements relating to the development of photographic pictures. December 29.

1894.

824. W. White. See Class XIX.

## XXII.—EXPLOSIVES, MATCHES, Etc.

## APPLICATIONS.

24,425. H. Boyd. A new explosive. December 19.

1894.

601. H. V. Keeson. Improvements in percussion fuses for projectiles. January 11.

## COMPLETE SPECIFICATION ACCEPTED.

1893.

22,384. R. C. Schupphaus. Improvements in methods of securing the chemical stability of nitro-compounds. December 27.



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## NOTICES.

### ANNUAL GENERAL MEETING.

The Annual General Meeting will be held in Edinburgh in the month of July next. Full particulars will appear in a subsequent issue.

### BANKERS' ORDERS.

For the convenience of Members, the Treasurer has arranged with the Bankers of the Society that they shall collect subscriptions from Bankers in town and country; and Members, who have not already done so, are invited to fill up and sign the Banker's Order enclosed with the December number of the Journal, which should then be sent to the Honorary Treasurer, Mr. E. Rider Cook, East London Soap Works, Bow, E.

### PATENT LAW REVISION.

Discussions upon the proposed revision of the Patent Law have been held in all Sections of the Society.

Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 41 of the bye-laws, the Society has the right of priority of publication for three months of all such papers. Infringement of this bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. EYRE and SPOTTISWOODE, the Society's printers and publishers, to whom all communications respecting them should be addressed.

#### CANDIDATES FOR ELECTION AT COUNCIL MEETING. 23rd FEBRUARY 1894.

Ansbacher, Louis A., P.O. Box 1934, New York, U.S.A., colour manufacturer.

Buchan, Alex., 93, Malmesbury Road, Canning Town, E., chemist.

Coker, Ernest G., H.M. Patent Office, Chancery Lane, W.C., examiner of patents.

Cowburn, W. H., Hope House, Bignor Street, Cheetham Hill, Manchester, chemical merchant.

Dufton, Arthur, Technical College, Bradford, Yorks, teacher of chemistry and dyeing.

Fletcher, Ernest L., Grappeuhall, near Warrington, gas engineer.

Hadley, Walter S., Millersdale, Sutton, St. Helens, Lancashire, plate-glass maker.

Haynes, David O., P.O. Box 583, Detroit, Mich., U.S.A., proprietor of "Pharmaceutical Era."

Hepburn, J. G., Priory Works, Dartford, Kent, leather manufacturer.

Hills, Harold F., 149, Bow Road, London E., analytical chemist.

Jones, M. W., 50, Mayfield Road, Whalley Range, Manchester, chemist.

Knight, W. A., Sexey's Trade School, Bruton, Somerset, head master.

Krause, Dr. Albert H., 132, Woodland Avenue, Cleveland, Ohio, U.S.A., chemist, Grasselli Chemical Company.

Langen, H. G., Maschinenfabrik, Greveubroich, Germany, chemical engineer.

Leitch, Jno. W., Millsbridge Chemical Works, near Huddersfield, aniline dye manufacturer.

Loewenstein, Harry H., c/o The Lowe Bros. Co., Dayton, Ohio, U.S.A., chemist.

Louis, Henry, 20, Lancaster Road, Belsize Park, N.W., mining and metallurgical engineer.

McMullan, Chas., 20, Corn Market, Belfast, Ireland, soap works manager.

Maxwell, John, Solway Chemical Works, Sillioth, Cumberland, chemical mazure manufacturer.

Nuttall, Harry, c/o John A. Bremner and Co., Albert Street, St. Mary's, Manchester, oil manufacturer.

Picard, Hugh F. K., 59, Abbey Road, St. John's Wood, N.W., metallurgist.

Santer, Herbert, Albion Brewery, Caledonian Road, N., brewer.

Schidrowitz, Philip, 24, Chichester Street, Chester, research chemist.

Sohn, Chas. E., 25, Beverley Road, Anerley, S.E., analyst.

Waldman, Louis J., P.O. Box 162, Albany, N.Y., U.S.A., aniline dye manufacturer.

Watmough, Benj., 4, Ferneliffe Road, Bramley, near Leeds, analytical chemist.

Watson, Jas., Caldwell, South Shields, assistant manager of alkali works.

White, W. Gilchrist, Lamb-Roe, Whalley, Lancashire, calico printer's chemist.

Wotherspoon, Peter, 14, Prospect Terrace, Jarrow-on-Tyne, chemist.

#### CHANGES OF ADDRESS.

Abbott, John, 1/o London; 6, Chichester Terrace, Brighton.

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Gibson, Dr. J., 1/o Harrington Gardens; 20, George Square, Edinburgh.

Harmon, L. E., 1/o New York; Clover Mills, Buffalo, N.Y., U.S.A.

Isaac, J. F. V., 1/o Putney; 114, Marine Parade, Glasgow.

Ivatts, Harold E., Journals to 203, Earlham Grove, Forest Gate, E.

Loewenthal, Dr. R., 1/o Schwerin; Steglitzerstrasse, Berlin, W.

Luthy, Otto, 1/o Barberton; 2336, Fairmount Avenue, Philadelphia, Pa., U.S.A.

McFarlane, R. F., 1/o Servia; 27, Cardigan Terrace, Heaton, Newcastle-on-Tyne.

Manu, Harold H., 1/o York; 5, Rue Rollin, Rue Monge, Paris.

Manning, Isaac H., 1/o Baltimore; Chapel Hill, N. Car., U.S.A.

Miller, Dr. A. K.; Journals to 111, Plymouth Grove, Manchester.

Miller, Geo., 1/o Widnes; Church Cottage, Halewood, Lancashire.

Nichols, J. A., 1/o Hurstfield; Stanley Mount, New Mills, Stockport.

Pedler, J. R., 1/o Dulwich; 47, Tregunter Road, South Kensington, S.W.

Phipson, Dr. T. L.; Journals to 8, Hotham Villas Road, Putney, S.W.

Pope, W. J., 1/o Hartismere Road; 16, Barclay Road, Waltham Green, S.W.

Preston, E. S., 1/o Plymouth; Easton, Weybridge, Surrey.

Quinn, J. Cardwell, 1/o Upper Thames Street; 503, Romford Road, Forest Gate, E.

Rayner, J. A. E., 1/o Ambleside; Grove House, Waver-tree, Liverpool.

Richardson, Clifford; Journals to Central Power Station, Washington, D.C., U.S.A.

Saniter, E. H.; Journals to 11, New Market Street, Wigan.

Towers, J. W., 1/o Widnes; Brantwood, Allerton, near Liverpool.

Tulloch, J., 1/o Jarrow; 11, Newton Terrace, Hebburn-on-Tyne.

Turgensen, Dr. Rolof, 1/o Offenbach; Smetanka 6, Weinberge, Prague, Austria.

Zinkeisen, Dr. W., 1/o Glasgow; 9, Campden Grove, Kensington, W.

#### RESIGNATIONS CANCELLED.

Leeds, Prof. Albert R., Stevens Institute of Technology, Hoboken, N.J., U.S.A.

Whitaker, Alf., Waltham, Horsforth, Leeds.

#### ERRATA IN LIST.

Larsen, J., should be spelt Larson, J.; address as before.

Rouillard, R. A. (wrongly spelt Romillard), 5, Dundonald Road, Wimbledon.

## Deaths.

Smith, Thos., Heriot Hill House, Edinburgh.

Solvay, Alf., 25, Rue du Prince Albert, Brussels. At Nice.

## London Section.

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### SESSION 1893-94.

#### REMAINING MEETINGS.

First Monday in each Month at 8 p.m.

March 5th:—

Admiral J. H. Selwyn. "The Zymean Metallurgy."  
Mr. F. Bale. "The Commercial Production of Chlorine by the Ammonia-Soda Process."  
Mr. F. H. Leeds, F.I.C., F.C.S. "Notes on Lithographic Varnish."

April 2nd:—

Mr. J. W. Lovibond. "Colour as a means of Quantitative Estimation."  
Mr. W. C. Young, F.I.C., F.C.S. "The Natural Diminution of the Dissolved Organic Matter in the Water of Rivers."  
Mr. H. Louis, A.R.S.M., F.I.C., F.G.S., &c. "Notes on an Improved Specific Gravity Bottle."

N.B.—Election of Officers and Five Members to the Local Committee.

Nominations must be signed by Ten Members and sent to the Hon. Local Secretary not later than Tuesday, March 20th. Members can only sign one nomination. (See By-Laws.)

April 16th (extra meeting).—Mr. H. de Mosenthal. "Gold Extraction Process."

May 7th:—

Mr. Claude Vautin. "The Commercial Electrolysis of Fused Salts."  
Messrs. W. H. Stanger, Memb. Inst. C.E., and Bertram Blount, F.I.C., F.C.S. "Testing of Hydraulic Cements."  
Mr. Oscar Guttmann, Assoc. M. Inst. C.E., F.I.C. "Manufacture of Smokeless Powder."

June 4th:—

Mr. W. G. Macmillan:  
(a.) "Experiments on the Strength of Leather."  
(b.) "Note on the Colour of Brass."  
Dr. P. Dvorkovitch. "Distillation of Peat."

Notes of the Meetings appear regularly in the Scientific Press.

Meeting held Monday, February 5th, 1894.

MR. WM. THORP IN THE CHAIR.

## THEORY OF DYEING. PART I. "INGRAIN COLOURS."

BY W. P. DREAPER, F.I.C., F.C.S.

**General Introduction.**—It was not until comparatively recent times, that chemists attempted to study and explain the very varied phenomena which make up what is termed "the practice of dyeing." In fact, until a large amount of close work had been done in physical and organic chemistry, the problem was an almost insurmountable one, and could not be attacked with much chance of success. Unfortunately, until late years, the part played by the fibre on which the colour is dyed and introduced, was to a

great extent ignored. Probably most investigators sided with those who favoured a more or less mechanical theory, being influenced, no doubt, by the more easily investigated problem of the part played by mordants, and other questions, which it is hardly necessary to go into on the present occasion. The introduction of "aniline" colours, and the tremendous impetus given to research in this direction from the year 1856, naturally kept research chemists who were interested in the matter, working at the chemistry of the dyes themselves.

However, the question gradually resolved itself into "Is the process of dyeing a purely chemical or mechanical one?" This question, in spite of a certain amount of work that has been done on the subject, has not yet been finally answered.

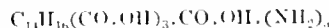
In taking into account any theory of dyeing, whether chemical or mechanical, there is one thing that must be considered. As the fibres are constituted, one must allow of two processes in the dyeing. Firstly, a process by which the dye is brought into contact with the general mass of the fibre, and then, in the case of a chemical theory, the reaction which takes place between the fibre and the dye. In all processes going under the name of dyeing, it is necessary to present the dye to the fibre in a suitable form. In simple dyeing this is nearly always brought about by dissolving the former in some convenient solvent, and then immersing the fibre in it, under certain conditions of temperature, concentration, &c.

The phenomenon of solid solution was first noticed by Van t'Hoff in 1890 (*Zeits. f. Phys. Chem.* 5, 322), and it was, I believe, first suggested by Otto N. Witt, in 1891 (*Färb. Zeit.* 1890-91), as an explanation of the process of dyeing. Certainly all the conditions necessary to this theory are present, and great attention has been lately paid to it.

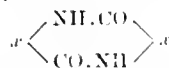
On the other hand, Leo Vignon (*Comptes Rend.* 110, 286, 112, 623), E. Knecht (*J. Soc. D. and C.* 1889, 77), and others, have insisted, and brought forward results which tend to prove, that the process of dyeing is a purely chemical one. Knecht's results show that when silk, or wool, is dyed with basic colouring matters, the dye is decomposed, the base combining with the fibre, the acid remaining in solution. He says, in conclusion, "This affords an almost undeniable proof that, in those cases at least, the dyeing is not a mechanical absorption, but a strictly chemical one." He also noticed that the wool would only take up a certain maximum amount of colour.

According to this general view, silk and wool are probably amido-acids, which have the power of combining with either an acid or basic compound. Vignon, experimenting on cellulose, found that by introducing an amido group into the cellulose molecule (by treating with strong ammonia at a high temperature, and under pressure), the fibre then readily took up acid dyes. This result is interesting, and, although only what might be expected, shows that the above chemical theory may apply to silk or wool, as both of them contain nitrogen.

Richardson (this Journal, 1893, 426), in an interesting article, tends to show that the "mass formula"—



represents the silk molecule, and that the following graphic formula probably represents fibroin:—



$x$  representing a hydrocarbon residue. However, it will be generally acknowledged that a great deal more work will have to be done on the subject before the question is finally settled. A method of dyeing has lately been patented, based on this knowledge, azo dyes being directly produced by diazotising silk fibre and combining with suitable phenols and amines. I have prepared one or two samples in this manner, and, with apologies to the patentee, I now produce them for your inspection.

It is almost unnecessary to state that cellulose will not act in this manner, and from its constitution a theory which will satisfy the requirements of silk and wool dyeing will not be applicable to cotton, as will be seen from the

following formula of cellulose, as recently given by Messrs. Cross and Bevan (Chem. Soc. Jour, 1893, 837),  $C_6H_7O(OH)_4$  in its simplest terms.

*Introduction of Dye to Fibre.*—Osmosis probably plays an important part in the process of dyeing, and it might be possible to institute experiments, comparing the osmotic pressure of dyes, through inert membranes, with the results obtained with fibres under similar conditions. It is perhaps necessary to point out here that dyes are nothing but organic salts exhibiting specific optical effects which will not influence, or distinguish them from similar colourless organic bodies in their action towards fibres.

One can only imagine that the dyes are introduced to the interior of the fibre by osmotic action. Under these circumstances it is probable that the rate of dyeing is more due to this action than to any other, chemical or mechanical.

The following general laws will therefore govern the rate of dyeing:—

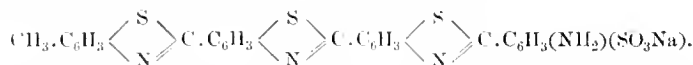
(1)\* The pressure (osmotic) is proportional to the concentration of the solution, or proportional to the volume in which a definite volume of the substance is contained.

(2.) The pressure increases for constant volume proportionately to the absolute temperature.

(3.) Quantities of substances (dissolved) which are in the ratio of the atomic molecular weights of the substances, exert equal pressure at equal temperatures.

Diffusion being caused by osmotic pressure (W. Norst, Zeits. f. Phys. Chem. 2, 613), and the rate of diffusion increasing with temperature (Graham), the practical results in dyeing seem to fall in well with the above facts.

Fick's law "that the quantity of salt, which diffuses through a given area is proportional to the difference between the concentration of the two areas, infinitely near to one another," has been since verified and found to be true for gelatin or agar-agar solutions. Animal membranes were found to alter the process, which took place, and the results were, roughly, half those obtained with the purely osmotic phenomena. The flow of the dissolved



If this dye be diazotised and combined with phenols, amines, or other suitable compounds, azo dyes of various colours are produced. As Mr. Green has fully pointed out in this Journal (Vol. VII., 179), these colours can be produced on the fibre. Under these conditions the colours are remarkably resistant to the action of soap solution, in this way resembling the so-called adjective colours, such as alizarin-red on an alum mordant; although one cannot in any way compare them to the latter class of dyes, for they are completely soluble in alkaline or soap solution. Speaking generally, they are soluble in both acid and alkalis when phenols are used as developers, and only in alkalis when these are replaced by amines; neither can these colouring matters be compared with the so-called "substantive colours," which dye on to cotton without mordant. Although some of these are very resistant to the action of soap when dyed on that fibre, yet they are comparatively speaking "loose" when dyed on silk.

Some other explanation to that generally given for the process of dyeing will have to be devised to explain their general behaviour to reagents, the colours being soluble in alkaline solution, and yet fast against the action of soap. On preparing some of these azo dyes outside the fibre, and dyeing the same direct, the colours were not resistant to the action of the same reagents. With a view to studying this question more fully, the following experiments were instituted. These were all performed on silk, and without confirmation must not be taken to apply to other fibres. It is necessary to consider these individually, for even in physical properties, such as shade, and fastness, against light and reagents generally, the results obtained on different fibres are not identical.

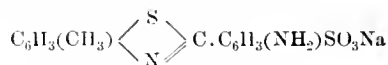
substance was hindered, but not stopped. An osmotic rise in pressure took place, but did not reach its true value (Zeits. f. Phys. Chem. 3, 316).

This is probably a similar state of things to that obtained in dyeing, although it is not denied that minor factors may modify the direct results.

E. J. Mills and T. Hamilton have shown (this Journal, Vol. VIII., 263) "that under certain circumstances the proportion of two mixed colours dyed on wool is the same as that in which they existed in the vat before dyeing." This result also corroborates the above idea, falling in with law (3).

Cross and Bevan (this Journal, XII., 104), although dealing with the more inert cellulose and ligno-cellulose fibres, come to the conclusion that there is a direct reaction between ferric ferricyanide and ligno-cellulose, similar results being obtained with gelatin.

*Ingrain Colours.*—With the object of, if possible, helping to clear up some of the questions in connection with this subject, the following work has been undertaken. I propose this evening to take up the so-called "ingrain colours," or rather to glance at one or two of the questions involved in the dyeing of them; for I find that the complete study of this subject cannot be entertained in the short space devoted to this paper, involving, as it would do, hundreds of experiments. I also saw that I should have to confine my experiments within comparatively narrow limits, only ascertaining that the results held good on a more liberal scale. It was therefore decided that these experiments should be confined to the study of the colouring matters obtained from *p*-toluidine, which are sent out under the name of "Primuline." Mr. A. G. Green kindly informs me that this dye contains 10 per cent. of dry sodium carbonate, 20–30 per cent. of dehydrothiotoluidine sodium sulphonate—



and the rest amidobenzenyl-amido-thio-cresol sodium sulphonate—

*Experimental Results.*—In all the following experiments the following standard method of dyeing was adopted. Six samples of silk, weighing 1 grm. each, were boiled in 400 cc. of distilled water with 1 per cent. of primuline. At the end of a quarter of an hour 2 grms. of sodium chloride were added, and the solution kept at the boil for 10 minutes; and then 1 cc. of 30 per cent. acetic acid added, and the dyeing continued for a further 35 minutes. By this means the fibre was evenly dyed.

The standard soap solution used in all cases was a 0.4 per cent. solution of olive-oil soap, containing 1 per cent. of free KHO, the standard soda solution being a 0.4 per cent. solution of dry sodium carbonate. The temperature was kept at 95°C., 500 cc. of boiling-out liquor being used for every grm. of silk.

No. 1. The action of soap solution on primuline itself was first studied, with the following results:—

No.	Time of Boiling.	Loss of Colour.
	Mins.	Per Cent.
1	5	0.10
2	10	0.20
3	15	0.30
4	30	0.30
5	60	0.60

In order that the loss of colour might be noted with as much accuracy as possible, independent samples were dyed with varying amounts of colour, and the shades obtained used for comparison with the above. This was the general

\* Only holds good for indifferent substances.



method adopted in this paper, although many of the results were obtained by colorimetric methods. The latter method was considered to be the more accurate when small quantities of the dye were removed.

No. 2.  $x.N_2.C_6H_4.OH$ .—Six samples were dyed as above, with 1 per cent. primuline, diazotised, and combined with phenol in alkaline solution, and the resultant shades were boiled out in standard soap solution, with the following results:—

No.	Time.	"Ingrain" Loss in Colour.	"Direct" Loss in Colour.
	Mins.	Per Cent.	Per Cent.
1	5	0.03	0.10
2	15	0.09	0.50
3	30	0.15	0.57
4	45	0.18	0.65
5	60	0.20	0.74

The results in this and similar cases have been entered under the column "Ingrain," and all through the paper results obtained in this way, by diazotising on the fibre, will be thus designated.

Six samples were dyed "direct," with an amount of the phenol azo dye, (prepared outside the fibre) equivalent to 1 per cent. of primuline, in standard dye liquor. After dyeing for one hour, all the colour had gone on to the fibre, and the samples were then passed through soap solution under standard conditions; the above results were obtained. The two sets of results are also plotted as curves, and the difference in colour, if any, is noted in the table devoted to that subject.

No. 3.  $x.N_2.C_6H_4.NH_2$ .—Six samples were dyed with 1 per cent. primuline, and, after diazotising, combined with aniline in acid solution, and the resulting shades boiled out in standard soap. As with all azo dyes prepared with amines from primuline, it was found necessary to dissolve the dye in slight excess of NaHO for the "direct dyeing"

set of samples, for the colouring matter is insoluble in acid solutions. Care had to be taken in dyeing, not to acidulate the dye liquor until nearly all the colouring matter was fixed on the fibre. Otherwise the treatment was identical with that adopted in the case of the phenol compound. The results are given in the tables.

No. 4.  $x.N_2.C_6H_4.N_2.C_6H_4.NH_2$ .—Another case that will require special mention, is that of the "azotriple" colouring matter, obtained by rediazotising the above amino-azo colour, and recombining with the same base. The direct dyeing was conducted in the usual way, with dye prepared by rediazotisation, and combining with aniline, and like the previous dye was insoluble in acids.

No. 5.  $x.N_2.C_6H_3(COOH.OH)$  (alkali yellow or cotton yellow R).—A yellow dye is put on the market of the above composition, prepared by combining the diazotised primuline with ortho-oxybenzoic (salicylic) acid.

No. 6. The results obtained by acting on the diazotised primuline with ammonia are also added to the list. The composition of this yellow dye is unknown, but the ingrain colour is very fast against soap. It is known in commerce under the name of "mimosa."

No. 7. Atlas Red R.  $x.N_2.C_6H_3(NH_2)_2$ , is prepared by diazotising primuline and combining with *m*-tolylene diamine. It gives a scarlet on silk. Diazotised and combined with  $\beta$ -naphthol a brown dye is produced. The results obtained with this colour will be found in the general table.

The results obtained with other developers and dyes are tabulated as follows, and the different sets of curves are also given in the different diagrams.

*Action of other Reagents.*—In order to find out whether the above general results were obtained with other reagents besides soap, the action of sodium carbonate solution on the different dyes was studied. An example is given. The aniline azo dye was used. A 0.4 per cent. solution of dry sodium carbonate was employed, the ratio of solution to silk being kept the same as in the previous experiments with soap.

*Microscopical Examination of Dyed Fibre.*—No difference, as far as evenness of colour was concerned, could be noticed between the fibre dyed "ingrain" and "direct."

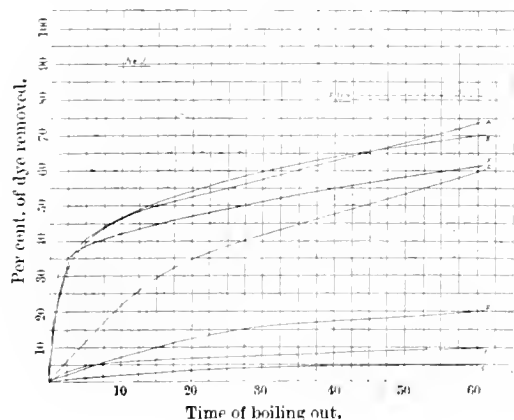
Dye.	Developer.	5 Minutes.	10 Minutes.	15 Minutes.	30 Minutes.	45 Minutes.	60 Minutes.	Remarks.
Primuline	C <sub>6</sub> H <sub>5</sub> OH .....	..	0.05	0.10	0.15	0.18	0.20	.. .. Ingrain.
"	" .....	0.40	..	0.50	0.57	0.65	0.74	.. .. Direct.
"	C <sub>6</sub> H <sub>4</sub> (OH) <sub>2</sub> (1.3) .....	0.03	..	0.05	0.09	0.15	0.17	.. .. Ingrain.
"	" .....	0.45	..	0.50	0.58	0.65	0.75	.. .. Direct.
"	βC <sub>10</sub> H <sub>7</sub> .OH .....	0.02	..	0.05	0.19	0.13	0.15	.. .. Ingrain.
"	" .....	0.30	..	0.40	0.50	0.55	0.63	.. .. Direct.
"	NH <sub>4</sub> .OH .....	0.02	..	0.04	0.05	0.06	0.08	.. .. Ingrain.
"	" .....	0.24	..	0.36	0.42	0.46	0.50	.. .. Direct.
"	C <sub>6</sub> H <sub>4</sub> .COOH.OH(1.2)	0.03	0.04	0.05	0.07	0.09	0.12	.. .. Ingrain.
"	" .....	0.48	..	0.60	0.68	0.71	0.75	.. .. Direct.
"	C <sub>6</sub> H <sub>5</sub> .NH <sub>2</sub> .....	0.04	..	0.05	0.06	0.09	0.10	Azo dye,..... Ingrain.
"	" .....	0.40	..	0.50	0.60	0.65	0.70	" .....
"	C <sub>6</sub> H <sub>5</sub> .NH <sub>2</sub> .....	0.01	..	0.03	0.04	0.05	0.07	" (Na <sub>2</sub> CO <sub>3</sub> ) Ingrain.
"	" .....	0.18	..	0.20	0.25	0.28	0.30	" .. Direct.
"	C <sub>6</sub> H <sub>5</sub> .NH <sub>2</sub> .....	0.01	..	0.02	0.03	0.04	0.05	) Azo triple { Ingrain. dye. { Direct.
"	" .....	0.38	..	0.45	0.52	0.58	0.61	
"	C <sub>6</sub> H <sub>4</sub> .(NH <sub>2</sub> ) <sub>2</sub> 1.3.....	0.01	..	0.025	0.035	0.05	0.052	.. .. Ingrain.
"	" .....	0.40	..	0.45	0.55	0.65	0.80	.. .. Direct.
"	βC <sub>10</sub> H <sub>7</sub> .NH <sub>2</sub> .....	0.10	..	0.15	0.20	0.25	0.27	.. .. Ingrain.
"	" .....	0.50	..	0.60	0.65	0.70	0.79	.. .. Direct.
"	" .....	0.10	0.20	0.30	0.39	..	0.50	.. .. Direct.
Atlas Red R	βC <sub>10</sub> H <sub>7</sub> .OH .....	0.10	..	0.14	0.20	0.25	0.27	.. .. Ingrain.
	" .....	0.50	..	0.55	0.62	0.71	0.76	.. .. Direct.

It will be seen from the above table that the results vary very much with different developers. Perhaps this will be more clearly seen in the set of curves on the diagrams. Before alluding to this matter, however, I wish to point out that the colours obtained by ingrain dyeing are not identical with those from the direct method, as the following table will show:—

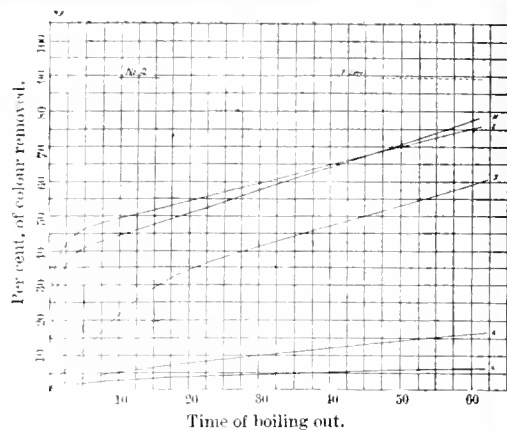
Dye.	Developer.	Colour Obtained.	Method of Dyeing.
Primuline	$C_6H_5.OH$ .....	Yellow.	Ingrain.
"	" .....	Do., slightly darker.	Direct.
"	$C_6H_5.NH_2$ .....	Yellow (brown shade).	Ingrain.
"	" .....	Do., slightly darker.	Direct.
"	$C_6H_4(OH)_2.3$ .....	Orange.	Ingrain.
"	" .....	Do., redder shade.	Direct.
"	$C_6H_4(NH_2)_2.3$ .....	Red-brown.	Ingrain.
"	" .....	Do., redder shade.	Direct.
"	$C_6H_4.OH.CO_2H$ ..	Yellow.	Ingrain.
"	" .....	Do., slightly duller.	Direct.
"	$C_{10}H_7.OH$ .....	Red.	Ingrain.
"	" .....	Do., red (blue shade).	Direct.
"	$C_{10}H_7.NH_2$ .....	Scarlet.	Ingrain.
"	" .....	Do., Red (blue shade).	Direct.
"	$NH_4.OH$ .....	Sulphur yellow.	Ingrain.
"	" .....	Do.	Direct.
"	$C_6H_5.NH_2$ .....	Yellow-brown.*	Ingrain.
"	" .....	Do. (brownier).*	Direct.

\* Azo triple dye.

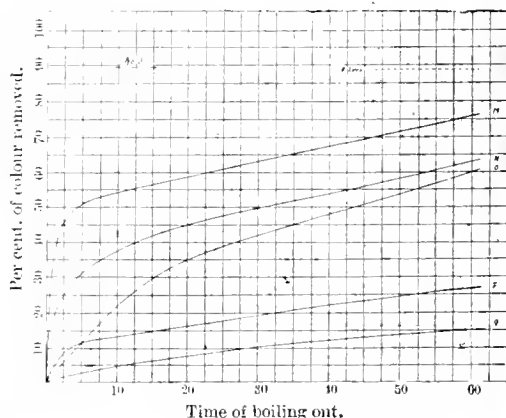
This would in itself point to the fact that the compounds produced are not identical, or at least tend to show that such is the case, and adds strength to the general argument.



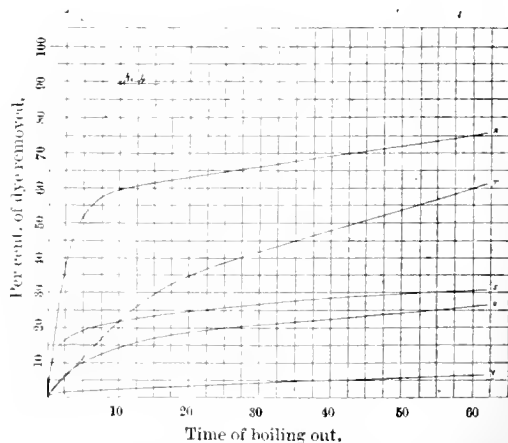
- A. and D. Prim. and  $C_6H_5.OH$ .  
 B. and E. Prim. and  $C_6H_5.NH_2$   
 C. Primuline.  
 F. and G.  $C_6H_4.NH_2$  (azo triple).



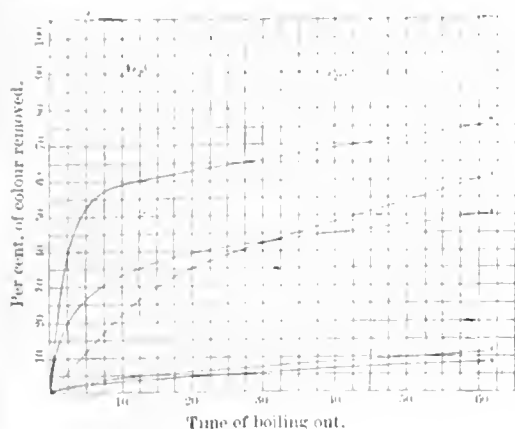
- I. and K. Prim. and  $C_6H_4(OH)_2.3$ .  
 H. and L. Prim. and  $C_6H_4(NH_2)_2.3$ .  
 J. Primuline.



- M. and P. Atlas Red R, and  $C_{10}H_7.OH.3$ .  
 N. and Q. Prim. and  $C_{10}H_7.OH.3$ .  
 O. Primuline.



- R. and U. Prim. and  $C_{10}H_7.NH_2.3$ .  
 S. and V. Prim. and  $C_6H_5.NH_2(Na_2CO_3)$ .  
 T. Primuline.



W. and Z. Prim. and  $C_6H_4.COOH.OH.1'2$ .

X. and 2.  $NH_4OH$  and Prim.

Y. Primuline.

Dye.	Developer.	"Ingrain" $x$ .	"Direct" $y$ .	$\frac{x}{y}$	Remarks.
Primuline	$C_6H_5.OH$ .....	0.20	0.74	$\frac{1}{3.7}$	
"	$C_6H_4(OH)_2(1'3)$ .....	0.17	0.75	$\frac{1}{4.4}$	
"	$C_6H_4.OH.COOH(1'2)$ .....	0.12	0.75	$\frac{1}{6.2}$	
"	$C_{10}H_7.OH\beta$ .....	0.15	0.63	$\frac{1}{4.2}$	
"	$NH_4.OH$ .....	0.08	0.50	$\frac{1}{6.2}$	
"	$C_6H_5.NH_2$ .....	0.10	0.70	$\frac{1}{7.0}$	Azo dye.
"	$C_6H_5.NH_2$ .....	0.05	0.61	$\frac{1}{12.0}$	Azo triple dye.
"	$C_6H_5.NH_2$ .....	0.07	0.52	$\frac{1}{7.4}$	$Na_2CO_3$
"	$C_6H_4.(NH_2)_2(1'3)$ .....	0.052	0.80	$\frac{1}{15.4}$	
"	$C_{10}H_7.NH_2\beta$ .....	0.27	0.79	$\frac{1}{2.9}$	
"	$C_{10}H_7.OH\beta$ .....	0.27	0.76	$\frac{1}{2.8}$	

Most striking, too, is the result obtained with the aniline azo triple dye, its resistance ratio being as 1:1.7 when compared with the azo dye itself.

The results with Atlas Red R and  $\beta$ -naphthol do not exhibit that fastness which should correspond with the azo triple dye from primuline and aniline; but it must be remembered that the dye is of a phenolic nature, and in this way perhaps falls in with the previous remarks. Thus, without any evidence to the contrary, we may take it for granted that the relative fastness depends more on the phenolic or basic nature of the dye than on the internal structure of the dye molecule. These remarks of course only apply to primuline dyes, but will probably be found to be true for all similar bodies.

Some azo dyes cannot be developed on silk. Take, for instance, the dye produced from  $\beta$ -naphthol sulphonie acid ("R salt") and diazotised primuline.

According to the general law of osmosis as previously mentioned, this difference can hardly be due to difference

*Observations on Curc.* It is not proposed in this paper to discuss the question from a mathematical point of view, but only in a general way.

It will at once be seen that the results obtained are strikingly at variance with any simple theory of solid solution, for in that case the curves from the direct dyeing method should coincide with the ingrain dyed curves. This is not the case, and it will be further noticed that the direct dyeing results show that the dyes are more easily removed than the original primuline itself, while the ingrain dyes are very resistant to the action of soaps and alkali, and therefore must be taken to be in combination, in some way or other, with the fibre itself. This will be better seen by the following table, where the results in each case after one hour's boiling are tabulated and their ratio shown.

It will be noticed that the alcoholic or phenolic azo dyes do not resist the action of the soap to anything like the extent that the amine compounds do. Take, for instance, the figures for resorcinol and *m*-phenylene-diamine respectively—4.4 and 15.4. These general results will be noticed all down the column except in the case of  $\beta$ -naphthylamine, which does not give a very resistant ingrain dye when compared with the other amine derivatives. It might be argued from these general results that fibron shows a more acid than basic reaction, but this point is simply noticed in passing.

in diffusion of the developers, as has been suggested, for quantities of substances (dissolved) which are in the ratio of the atomic molecular weights of the substances, exert equal pressure at equal temperatures." I have come to the conclusion that the phenomenon is due rather to a compound being formed by the diazo compound with the silk itself, which the "R salt" is unable to decompose.

The resistance of primuline itself (as seen by the curve) against soap is, as a matter of fact, greater than the resistances of many of the azo dyes themselves, and this partly corroborates this suggestion.

When this silk is dyed in the first place with primuline, we may take it for granted that a combination takes place between the primuline base and the fibron; then on diazotising, the primuline is diazotised, and perhaps partly combines with the silk again, and then the developer, if powerful enough, will form the dye. It is hardly possible that this truly represents the reaction, but it may help to give an idea as to what might take place when the colour is

produced in the fibre,--one has a soluble dye on the one hand and a fibre on the other. There must either be a chemical combination or simply a mechanical action, such as the withdrawing of alkaloids from aqueous solution by ether.

If we accept the latter statement as representing the true state of affairs, we are face to face with the fact, fully established in this paper, that when the dye is presented to the fibre under different circumstances, totally different results are obtained. I can see nothing for it but to fall back on the chemical theory and believe that this with modifications, supplemented by a theory of osmosis, will most truly represent the action that goes on when azo dyes, such as primuline, are dyed "ingrain."

**False Zero.**—It was necessary to allow for the amount of dye that would still remain on the fibre when an equilibrium was established between the dye solution and the silk. In order to estimate this, 1-grm. samples were dyed in 500 cc. of 0.4 per cent. soap solution containing 1 per cent. of the dye in question. The dyeing was continued for an hour; the silk was then removed from the dye-bath, thoroughly washed, and passed through acetic acid. After drying the percentage of dye on the fibre was estimated by comparison. Of course the ratio of the quantity of solution to dye would greatly influence the result, and with a much larger quantity of the former would more nearly approach the true zero (100 per cent.).

The results obtained by dyeing as above were as follows:—

Dye.	Per Cent. of Dye taken up by Fibre.	—
Primuline and $C_6H_5.OH$ .....	0.18	Diag. 1.
" " $C_6H_5NH_2$ .....	0.19	"
" " $C_6H_5(NH_2)_2$ .....	0.11	Diaz. 2.
" " $\beta C_{10}H_7.OH$ .....	0.12	Diaz. 3.
" " $C_6H_5.COOH.OH$	0.11	Diaz. 5.

In conclusion, I would point out, that the above results tend to connect up a link in the chain of a mechanico-chemical theory of dyeing—that is, a theory that depends primarily on a diffusion process obeying a modified form of the general laws of osmosis, supplemented in some cases, if not all, by a chemical, or series of chemical, reactions between the fibre and the dye. Whether this can be brought under the term "solid solution," even in its widest terms, is extremely doubtful, for one must then acknowledge that the "solubility" (?) of the dye dyed "direct" is greatly altered when dyed "ingrain."

#### DISCUSSION.

Mr. A. G. GREEN said they were much indebted to Mr. Dreaper for the careful quantitative study of the dyeing affinities of the ingrain colours, because it was only by the quantitative study of matters of this kind that any satisfactory theory of dyeing could be arrived at. The fact that the fastness of the ingrain colours formed upon fibres was immensely greater than the fastness of those same colours when made separately and then applied to the fibre, he had noticed many years ago. He had been rather inclined to favour a mechanical explanation. When an azo colour was built up in a fibre, one might suppose that the pores of the fibre were only sufficiently large to accommodate the primuline molecules or aggregate of molecules, and were tolerably completely filled up; then if you converted the primuline into an azo colour the molecules were greatly increased in size, and consequently had a greater difficulty in getting out when the cotton was boiled in a soap solution. It was quite possible, however, that the question was a chemical one. Mr. Dreaper's experiments only applied to silk, but exactly the same thing was found in the case of cotton. Many of these colours when made separately had a certain affinity for cotton fibre; for instance, that derived

from metaphenylene diamine; whilst in other cases, *e.g.*, the colour from betanaphthol, the affinity was very small. If the same colour were applied to cotton "direct" and "ingrain," it was always found that the ingrain colour resisted the action of boiling soap to a far greater extent than the colour applied direct. There was a good deal of evidence to show that primuline on the fibre was really in a state of chemical combination; for instance, take the sensitiveness of diazotised primuline on the fibre to light. If diazo-primuline were made separately and then applied to the fibre, it was very stable to light and was hardly affected at all. If you took two pieces of cotton, one dyed with primuline, and then diazotised the other, to which the same quantity of finely-divided diazotised primuline had been mechanically applied, the first would be completely decomposed on exposure to sunshine in a few minutes, whereas the other would remain completely unchanged for some hours, showing there was a considerable difference in chemical properties in the diazotised primuline when formed on the fibre, and when formed in a separate condition. The behaviour of dehydrothiolumidine sulphonic acid, the compound from which primuline was derived, was very curious. It differed from primuline in having no affinity for cotton, and was colourless, whereas primuline was yellow. But it would dye silk in the same way as primuline, that is to say, the silk would take it up, although without being coloured; this colourless silk could be developed just as if dyed with primuline, giving very similar ingrain colours. This was the only case which he had yet met with of a "colourless dye," if he might use the expression; that is, of a colourless substance possessing dyeing affinity. He had tried the sulphonic acids of a great number of phenols and amines, but had never found any possessing an affinity for animal or vegetable fibres.

Mr. F. EVERSHED asked how Mr. Dreaper estimated the quantity of colour removed from the fibres, and how long it was necessary to diazotise these silks. Also in some cases where the silk was dyed with primuline and afterwards developed, might not some of the colour attributed to the primuline be due to what one might call the silk colour, *e.g.*, that formed from silk itself?

The CHAIRMAN said that of the dyeing of fabrics he knew little or nothing, but he should like to ask Mr. Dreaper if there was anything to show whether ingrain colours had their additional fastness by reason of a combination between the material of the fibre and the colour, or whether it was simply that the colouring matter was formed inside the fibre, and therefore enclosed. Many substances apparently could only with great difficulty be washed out from a fibrous matter such as cotton fibre, simply because they require a very long time. The soluble substances thus enclosed only passed out by a process of dialysis, and that required time, whereas if they were external they were readily washed out.

Mr. DREAPER, in reply to Mr. Evershed, said that the pieces of silk after about a quarter of an hour showed a distinct yellow tinge. His idea was that primuline might fill up the pores of the fibre, and the azo dye formed from it might occupy more space and be held in the pores in the same way that alizarin dyes were supposed to be fixed on the fibre. He did not see how this could be the case, because the dye itself was soluble in alkali, and would at once be dissolved, and then it would only be a matter of exosmosis, or of the dye diffusing out of the fibre.

Mr. GREEN said the larger the molecules were, the faster the colour was. Mr. Dreaper's numbers for the azo triple base seemed to bear out his idea.

Mr. DREAPER suggested that might be because it was more basic, the aniline curve being faster than the phenol one. If the dye were not in combination with the fibre it would immediately go into solution when boiled with alkaline soap, and then more or less rapidly leave the fibre. He had noted the rate at which these developers diffused into the

fibre; they did not take longer than five minutes, judging from the rate at which the colours developed. It was the soap or sodium carbonate which diffused into the fibre, that would govern the rate of loss in colour, supposing that the dye was mechanically held in the fibre. He questioned whether, in the case of silk, it could be said to contain pores; it was more like a solid rod; it was not like cotton at all. There were no internal cavities. It was not likely that the silk colour formed on the fibre would materially interfere with the primuline result, for the length of time that the silk was in the nitrous acid would not be sufficient to diazotise the fibre. The Chairman had also put a question whether the ingrain colour was in combination or was simply inside the fibre. As far as he had gone, a microscopical examination showed no difference in the fibre; and from Knecht's, Vignon's, and other workers' results, one would imagine the colour must be in combination with the fibre. Undoubtedly if one dyed silk with a basic salt, and had the base going on to the fibre and the acid left in solution, it must be, as Knecht said, a chemical reaction, because the dye salt itself was split up. Primuline was much the same kind of body, and one might get the primuline base combining with the fibrous and sulphonic acid remaining in solution.

Mr. GREEN said that could not be the case; primuline was a sulphonic acid of the primuline base, not a sulphate. Moreover, as regards the possible greater basicity of the azo triple base, he thought that would not be so, since the proportion of the amido-group to the rest of the group was actually smaller. With regard to solution on boiling with alkaline soap, he would remind the author of the paper that the dyestuff was enclosed in a fibre, through which it had to diffuse. In the case of the developer, beta-naphthol, the molecular weight was 143 only, whereas the primuline molecule was 500 or 600. The molecular weight of primuline azo-beta-naphthol would be somewhere about 700. He thought silk might have internal cavities, though small ones.

Mr. DREAPER said the primuline curve on the diagram showed that that body had greater affinity for the fibre than many of the azo dyes derived from it. If dyed "direct" it was simply a matter of diffusion. According to the molecular weight, it would diffuse more readily than the azo triple bases. If the primuline were not in combination in some way with the fibre, by the general law of osmosis the primuline should diffuse out of the fibre into the liquor, and be boiled off to a greater extent than the azo triple dye dyed "direct." This was not the case; in fact, the primuline was much more resistant than the azo triple dye.

Mr. GREEN thought Mr. Dreaper's explanation could not hold true, because it would not apply to cotton, and he had observed exactly the same results working qualitatively in that case as had been described. The fastness of primuline on cotton was intermediate between the fastness of the ingrain colour made from it and the same colour made separately and then applied. He was not prepared to explain it. It appeared to him very difficult to find a chemical theory of dyeing which would include the dyeing of cotton.

Mr. DREAPER said cotton was not an amido compound, and must be taken separately when considering the general theory of dyeing.

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Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

### SESSION 1894.

March 7th:—

Mr. F. H. Tate. "Methods for the Rectification of Nitric Acid."

Mr. Max Muspratt. "The Quantitative Estimation of Cadmium."

April 14th.—Mr. J. Henry Brown. "The Treatment of Caproic Iron Pyrites as carried on at Portuguese Mines."

Meeting held Wednesday, February 7th, 1894.

MR. PERCY BATESON IN THE CHAIR.

## THE MANUFACTURE OF STRAW CELLULOSE.

BY JAMES BEVERIDGE.

THE manufacture of cellulose or paper pulp from the various kinds of straw is so well known to experienced paper-makers that it is probably difficult to mention anything specially new in dealing with such a subject. This statement may also be made in regard to other fibrous plants besides straw, and it may be deemed unnecessary by some that one should occupy the time of our Society on such a trite subject. The manufacture as a whole is, however, unquestionably a difficult one, and there are certain problems associated with it which are of great importance to pulp and paper-makers, and certainly of more than passing interest to the technical chemist.

Straw has been used for paper-making from very early times. It is said that the Chinese manufactured paper from rice-straw before the Christian era. This art originating with them gradually extended to Europe through Spain, Germany, France, Holland, and finally to England. The rapid development of the industrial resources of Europe at the beginning of this century, and our ever increasing social requirements demanding the extension of our paper industry, told heavily on the supply of rags, and gave a great impetus to the manufacture of paper pulp from fibrous plants. The scarcity of rags to meet these requirements not unnaturally caused paper makers to turn their attention to straw, because of its universal distribution and abundant supply. The invention, too, of a method of manufacturing a comparatively cheap and inexhaustible supply of alkaline soda products from common salt by Leblanc practically placed the chemical treatment of straw—as indeed all other fibrous plants—on a sound manufacturing basis. Since that period and the rise of the alkali industry in England, the inventions of Weldon and Deacon relating to the manufacture of cheap chlorine have added

much to the development of the straw pulp industry. Indeed, it may be said with perfect truth that the success of this manufacture to-day fluctuates with the market value of soda and bleaching powder.

There are two kinds of pulp made from straw, viz. straw pulp proper, and what may be more correctly termed straw cellulose. The former is made by steeping the straw, or boiling it under pressure, with milk of lime until it is thoroughly softened, after which it is washed and ground with suitable machinery and manufactured into a cheap quality of wrapping paper. This method is employed extensively in France and other countries of the continent where there is a demand for such papers, but, so far as I am aware, is not used in England to any great extent, excepting probably in the manufacture of strawboards.

Although the subject of this paper has nothing to do with this kind of straw pulp, yet it may be mentioned incidentally that the chemical action of the milk of lime on the incrusting materials surrounding the straw fibre is not a vigorous one. These incrusting materials are not completely, nor indeed to a great extent, separated from the cellulose. The mineral matter remains in the product practically untouched, and if any less quantity than that corresponding to the percentage in the original straw operated upon exists in the prepared pulp, it is due rather to the washing after digesting than to any solvent action of the milk of lime. The milk of lime, under certain conditions, has a bleaching action on the straw. It neutralises the organic acids usually formed when fibrous plants are heated for any length of time in presence of water. The yield of pulp obtained by such a treatment amounts to from 75 to 85 per cent. The papers produced from this pulp are of a very poor quality, and wholly used for packing purposes. They are hard, brittle, and possess a low tensile strength.

The preparation of pure cellulose from straw is a very different manufacture, and involves a cycle of operations, each one of which requires the most careful supervision. The product is used for the production of papers of first-class quality, fine and medium writings, for example, and must be well prepared, and as free from dirt as it is possible to get it. It is therefore necessary that cleanliness be observed in every department, and that the straw itself and the water used for washing, &c. be subjected to a preliminary purification as necessity may require before they enter the mill. It will only be necessary for me to touch lightly upon a series of operations through which the straw passes, and in order to make the whole subject plain, I will subdivide it into the following divisions:—

I.—Straws. Their composition, nature, &c.

II.—Manufacturing operations—

(a.) Cleaning, cutting, and dusting.

(b.) Digesting the straw in caustic soda lye.

(c.) Washing, breaking, purifying, and bleaching.

III.—General data *re* (a) yield, (b) bleach, (c) loss of soda, &c.

IV.—Fuel and mechanical power required to produce a ton of straw cellulose.

#### 1.—Straws: their Nature and Composition.

The straws usually employed for the preparation of cellulose are those obtained from the cereals, oat, wheat, rye and barley. In some parts of the continent maize straw has also been used, but I understand with only moderate success, as it yields a somewhat coarse fibre. Next to wood, oat, wheat, rye, and barley straws are the most universally distributed fibrous plants, known as sources of paper pulp. In point of cheapness they also rank next to wood, which is recognised as the cheapest source of cellulose at present known in this country. The physical character and composition, especially with regard to their ash contents, vary enormously. This is not only true of the different kinds of straw, but also of the same variety, and seems to depend upon the district or country in which they are grown. Probably the soil has influences on the composition of the ash as well.

The fibres contained in these straws are loosely bound together by resinous and intercellular matter, which is easily dissolved by caustic soda, and subsequently separated by washing. Thus isolated they are soft, flocculent, and admirably adapted for use in the manufacture of high-class writing papers. They differ slightly from one another in their paper-making qualities; the fibres from barley straw, for example, differing from those of oat, wheat, and rye in length, breadth, and general physical character. This difference is very noticeable when the pulp from each of these straws is separately brought forward in the mill. It is not very difficult to classify the straws according to the nature of the fibre or cellulose they yield, although such a classification as I am about to attempt is true only locally. My experience being confined to Dutch straws, the following bears directly on them:—

*Barley straw* yields a short, very soft fibre of low felting power. The knots and husks are soft, and in consequence this kind of straw is easily digested.

*Oat straw* is usually somewhat harder; the knots and husks are more difficult to digest. The fibres it yields are comparatively long, soft, and of medium felting power.

*Wheat and rye straw* are, according to my experience, closely allied. They both yield long fibres of good felting power, and are the most valuable as sources of cellulose. Rye straw in particular yields cellulose of excellent quality, and as it usually contains a comparatively small quantity of mineral matter, it is soft and easily digested.

The classification of these particular straws grown in other countries would doubtless be different, because the amount of ash they contain controls to a large extent the facility with which they are converted into cellulose.

The composition of these straws, according to Müller, an oft quoted authority, also varies enormously. His analyses, which I reproduce, plainly shows this:—

COMPOSITION OF STRAWS (MÜLLER).

	Winter Rye.	Winter Wheat.	Summer Barley.	Winter Barley.	Oats.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Water.....	14.3	14.3	14.3	14.3	14.3
Organic constituents.....	82.5	80.2	79.7	80.2	80.7
Ash.....	3.2	5.5	..	5.5	5.0
Fat and wax.....	1.3	1.5	1.4	1.4	2.0
Nitrogenous bodies.....	1.5	2.0	3.0	2.0	2.5
Starch, gum, &c. ....	25.7	28.7	31.3	28.4	36.2
Cellulose.....	51.0	48.0	43.0	48.4	40.0
Per cent. of dry cellulose on dry straw.....	63.0	56.0	50.1	56.4	46.6
* Per cent. air dry cellulose on air dry straw .....	60.0	53.3	47.7	53.7	44.4

\* Air dry cellulose containing 10 per cent. of moisture. Air dry straw containing 14.3 per cent. of moisture.

The estimation of the cellulose in fibrous plants by the method recommended by Müller is a long, tedious process, and is difficult to perform with accuracy in the laboratory. In my opinion it is uncertain, and requires experience and the exercise of great care in order to obtain concordant results. Few paper or pulp makers will believe that the percentage of cellulose obtained by him in some of the above examples really represents the truth. Some of them, according to my experience and researches, are unquestionably high, and cannot be taken as representative of the class of straw to which they relate. Although it is impossible to assign a definite percentage of cellulose to any one class of straw, yet practical manufacturing yields are fairly constant and it is very probable that Müller has included other organic matters closely allied to cellulose in the percentages given under this heading in his table.

It is manifest that a process is required for quickly estimating the cellulose available for papermaking in fibrous plants, especially one that will be fairly accurate and yield results more surely indicating the yield it is possible to get

in manufacturing practice. It is well known that weak aqueous solutions of bisulphites of soda, lime or magnesia, at moderately high temperatures, do not dissolve cellulose, whereas solutions of caustic soda or potash do, and they can be said to dissolve cellulose at all. The method of estimating the cellulose in this way seems to me to yield much more satisfactory results than Müller's method, and I have used it for many years for ascertaining the amount of cellulose in a great variety of fibrous plants and for the use of paper stocks. By digesting a known weight of the plant, straw, or an aqueous solution of bisulphite of soda, lime or magnesia, and after washing, bleaching, and drying, taking an allowance for the mineral matter in the pulp, the percentage of cellulose may be readily calculated. This method yields results invariably much lower than Müller's figures, and very closely resembling manufacturing yields, and therefore I believe more truly represents the cellulose available for papermaking purposes. Some of the results obtained by this method are the following, viz.:—

#### COMPOSITION OF STRAWS.

(By the method of digesting in bi-sulphite of soda.)

	French Wheat.	Zeland Wheat.	100% Wheat 1 lb. 1000.	Barley Oat.	Barley Rye.	Barley Barley.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Water.....	11.8	8.2	12.5	11.2	7.6	11.9
Ash.....	7.2	10.0	7.5	7.5	1.8	7.1
Cellulose (unbleached).....	36.6	37.6	36.4	37.5	41.3	34.1
Organic matter other than cellulose.....	44.4	44.2	43.6	40.9	49.3	47.7
	100.0	100.0	100.0	100.0	100.0	100.0
Percent, dry cellulose on dry straw.....	41.5	40.9	41.6	42.9	44.7	38.4
*Per cent of air dry cellulose on air dry straw.....	40.6	41.8	39.4	41.7	45.9	37.0
Per cent, silica (SiO <sub>2</sub> ) in straw.....	1.10	0.24	5.17	2.55	0.92	2.06
Cwts. straw to produce 1 ton of air dry cellulose...	4.9	47.8	39.5	47.9	43.5	52.7
Cwts. of silica from 1 ton of air dry cellulose.....	2.15	2.98	2.54	1.12	0.40	1.68
Cwts. of 60 per cent. alkali (Na <sub>2</sub> O) required to form Na <sub>2</sub> SiO <sub>3</sub> with the total silica.	3.70	5.11	4.39	1.94	1.62	4.85

\* Air dry cellulose containing 10 per cent. of moisture. Air dry straw containing moisture as shown above.

It will be noticed in both of these tables of analyses that the percentage of oven dry cellulose in the oven dry straw has been calculated in each case for comparison, and that the descending order in which the straws indicated yield in my own table is as follows, viz.:—rye, oat, wheat (average), and finally barley. On the other hand, the order, according to Müller's table, is different: rye, wheat, barley, and oat. If the results from both tables be compared, the difference between the percentages given for each class of straw is so great that, as I have previously stated, the experienced pulp or paper-maker would decline to accept Müller's results as representing the truth. Of course, I am well aware that this depends entirely on what one calls "cellulose." If the organic intercellular matter, insoluble in ether, benzol, and ammoniacal water, which binds the individual fibres together in the plant can be considered as cellulose, then Müller's figures are no doubt correct. On the other hand, this intercellular organic matter appears to offer little resistance to the solvent action of weak caustic alkalis and bisulphites, and certainly from a paper-maker's point of view, cannot be considered as available for his use.

#### 11.—Manufacturing Operations.

(1.) *Cleaning.*—The straw as it is brought from the stacks or store house is first of all passed through a machine to thoroughly open it; or this may be done by hand. It is then delivered or placed upon tables made of wire gauze,  $\frac{1}{2}$ -in. meshes, alongside of which girls stand whose duty it is to remove all weeds and other plants likely to produce what is known as shieve, in the finished pulp. From these tables it is elevated either automatically or by hand to the cutter, which usually consists of an ordinary chaff-cutter used by farmers. The straw is fed continuously by an endless travelling belt through an orifice across the face of which, at right angles to the feed, a wheel revolves, having one or more knives attached to its spokes. The length of cut varies from  $\frac{1}{2}$  in. to  $1\frac{1}{4}$  in. From this the chaff falls on to an inclined jogging sieve of  $\frac{1}{2}$ -in. wire gauze and in travelling to the lower end the bulk of the sand, husks, and fruit grains are separated. Although the chaff at this stage is tolerably well cleaned, some manufacturers prefer to give it a final dusting, and to do this they employ a revolving sieve, through the centre of which a shaft revolves at a high



velocity. This shaft has pegs studded along its length at equal intervals, and so placed as to form a kind of archimedean screw. The sieve is cone shaped or slightly inclined, and as the chaff travels forward, all loose dust is separated, being carried through the meshes by the current of air induced by the revolving shaft. It is then stored in heaps ready for the pulp boilers.

The object in subjecting the straw to such a trenchant system of cleaning as above indicated is to remove all foreign weeds, husks, fruit grains, and sand. This is absolutely necessary for the production of high-class pulp, for it has been found by experience that these impurities produce more or less unbleachable particles, and much of the success attained in avoiding these imperfections depends on the completeness and care with which the straw is primarily cleaned before it enters the pulp boilers. The loss incurred by cleaning will depend upon the quality of the straw, but it should not exceed 5 per cent. of the total weight operated on.

(B.) *Digesting the Straw in Caustic Soda Lye.*—The boilers usually employed for doing this are of the rotary type—either cylindrical or spherical. Very seldom are upright stationary boilers used. The reason of this seems to lie in the necessity of keeping the mass within the boiler in a continual state of motion, so that each particle of straw will be equally acted upon by the caustic lye. This cannot very well be attained in stationary boilers, even with the usual vomiting arrangement, because the chaff, when softened by the lye, sinks down into such a dense solid mass as to interfere with the free circulation of the lye through it.

Of the two rotary boilers mentioned, the spherical type offers certain advantages over the other. It occupies relatively less floor space, is more easily filled and emptied, and presents less radiating surface per unit of capacity. These boilers are usually provided with two manholes, a blow-off cock, &c., and are heated by steam injected through the trunnion. Motion is given to them by means of worm gearing fixed to the standards supporting the boiler.

The strength and volume of the caustic lye employed necessarily vary in different mills, according to the kind of straw used and upon other conditions peculiar to the nature of the apparatus in use, e.g., the steam arrangements of the factory. Both are slightly modified when barley straw is used alone, but as in actual practice the best results are obtained by mixing the different kinds of straw and keeping the mixture as nearly as possible constant, any rule laid down with respect to the proportion of caustic lye required is adhered to from day to day. The actual quantity of caustic soda, reckoned as 60 per cent. alkali, also varies slightly with different kinds of straw, as well as on the temperature and speed with which the digesting operation is carried out. Where the temperature is high and the time given for the boiling is long, the caustic is reduced to a minimum.

The following figures, representing the "charge," &c., of a straw pulp boiler, is from actual practice:—

Weight of straw (mixture of oat and wheat)	4,480 lbs. (40 cwt.)
Gallons of caustic lye	1,610
Hours under steam pressure	4
Steam pressure above atmosphere	60 per sq. in.
Maximum temperature	307° Fahr.

#### CAUSTIC LYE.

Twaddell	10½
Total weight in pounds	16,945
Percentage by volume of Na <sub>2</sub> O	0.3249
" " " 60 per cent. alkali	0.5416
Total 60 per cent. caustic soda in pound	872
Pounds of 60 per cent. caustic alkali on 1 cwt. of straw = 21.8.	

The operation of digestion is very simple. The pulp boiler is filled as full as possible with the cut and cleaned chaff and the required volume of caustic lye run in. The manlid door is then put on and a small quantity of steam admitted, while the boiler is made to revolve for 15 minutes or so in order to soften the straw and cause it to subside to make room for more. When this is done the lid is again removed and the vacant space within the boiler filled up

with more straw. In this way the charge of straw per boiler is increased from 15 to 20 per cent. When the boiler is thus filled and the manlid fastened on, high pressure steam is injected into the charge through the trunnion, and the pressure gradually raised to the desired degree. The pressure varies in different mills, but as a rule it registers from 60 to 90 lbs. per square inch. This pressure is reached after about two hours steaming, and is further maintained for 4 or 4½ hours. In the meantime the boiler is kept continually revolving.

(C.) *Washing, Breaking, Purifying, and Bleaching the Straw.*—After the digesting operation has been completed, the whole contents of the boiler are emptied into a tank placed beneath it, where the crude cellulose is washed with hot water. It is necessary to accomplish this work with the least possible quantity of water in order to avoid undue dilution of the waste lye. The most efficient method is that of the application of the principle of displacement as carried out in the lixiviating of ball soda in the Lèblanc soda process. It is possible in manufacturing practice to remove 95 per cent. of the soda associated with the boiled straw cellulose by this system of washing with a dilution of about one-third, that is to say, the pulp can be washed in the tanks with a quantity of water, represented by one-third of the volume of black lye associated with the pulp. The weakest washings from these vats are run to waste, as it does not pay to evaporate them, even although the most refined system of evaporation be used for their concentration.

After the bulk of the black soda lye has been removed in the way described, the crude cellulose is allowed to drain, and then conveyed to an apparatus called a "breaking engine," in which it is broken up into pulp.

In this engine it is again washed to remove the last traces of soda; and a proportion of the intercellular matter surrounding the fibres as well as any dirt will pass away with the wash water. That part of the intercellular matter carried away is in a fine state of division, and if it be left in the pulp it is supposed to consume a large quantity of bleaching powder in the subsequent bleaching. As a matter of fact, however, if the straw be properly boiled in the first instance, the intercellular matter has little influence on the amount of bleach required to bleach unit weight of crude product. It is possible to wash straw pulp in the breaking engine to such an extent as to seriously affect the yield of pulp, and therefore the bulk of the washing can be most advantageously done in the tanks.

The pulp, although now broken up, so that the fibres are separated from one another, is not yet in a fit state of purity to be bleached. It invariably contains a few unboiled pieces of straw, or possibly some unboiled knots or hard weeds, and it is absolutely necessary to separate these to get a clean product. In order, therefore, to cleanse it, the contents of the breaking engine are emptied into a large tank, and pumped into special purifying apparatus. This apparatus consists of a long shoot, called a sand-trap, as a rule about 200 feet or more in length, about 18 inches wide, and 9 inches deep, containing at intervals of 18 inches or so, cross-boards about 4 inches high, and inclined towards the flow of the pulp. At the end of this sand-trap, a series of cast-iron boxes, called strainers, are placed. These strainers contain brass plates, having very fine slits cut in them, and it is through these slits that the individual fibres pass, whilst the rough, hard, unboiled pieces are retained. After the pulp passes through these slits, it is practically pure and clean. The width of slit of course regulates to a certain extent the degree of purification required.

The pulp before entering this apparatus is largely diluted with water to enable the particles of sand, dirt, and heavy weeds to subside as the mixture flows over the submerged cross-boards. Any impurities which pass the sand-trap are retained by the strainer plates.

It is now necessary to remove the large excess of water from the cellulose before bleaching, and this is done in a variety of ways, one of the best being by means of a machine called a "tambour." This is simply a skeleton cone made of hard wood and covered inside with fine brass wire gauze. The whole is mounted on a horizontal shaft running through its centre, and as the pulp flows into the narrow end, as it

revolves, the water passes through the wire gauze, whilst the pulp is discharged from the wide end, in a concentrated state, into a reservoir, from whence it is pumped into the "potcher."

The "potcher" is constructed on the same principle as the "breaking engines," but as the name implies, is used simply as a mixing machine. When full, a certain volume of a clear solution of bleaching powder is added, and the stirring continued for an hour or two, when the whole charge is run off into large concrete tanks, fitted with false bottoms of perforated tiles. In these tanks the mixture of pulp and bleach liquor is allowed to remain for 12 or 16 hours, by which time the hypochlorite will be exhausted, and the pulp of a white colour.

It is only necessary to allow the spent bleach liquor to drain away, by drawing a plug from its seating in the bottom of the tank, and then removing the cellulose to another tank, where it is mixed with clean water, and finally pumped on to the paper machine, by which it is converted into a thick sheet of paper, in which state it is sold.

The foregoing in effect embraces the routine of this manufacture. The apparatus, both in arrangement and detail, is modified in different mills. The operations are simple, and require no great skill to carry them on successfully, but the chemical problems involved in the economical production of straw cellulose are more difficult to deal with, and I now propose to touch upon the most important of them.

### III.—Yield, &c.

It has always been the aim of manufacturers to obtain the highest yield of cellulose from unit weight of straw, and to this end various modifications of the soda process have been suggested and worked on a manufacturing scale. A process with this intent was brought out some years ago by Leunig and worked by an English company, but the results were very indifferent. Caustic soda was used to remove the incrusting organic and mineral matters surrounding the fibres substantially in the manner above described, but instead of bleaching with hypochlorites chlorine gas was employed. This, as is generally known, is a costly and anything but an easy process to carry on economically and free from nuisance, if not actual danger, and it is not surprising that the Leunig process proved unsatisfactory from a commercial point of view.

From what I have previously stated it might be inferred that a sulphite process for the treatment of straw would prove of value, inasmuch as by it the maximum yield of pulp would be obtained for the reasons already given—namely, that cellulose is less soluble in solutions of bisulphites of lime, soda, or magnesia than those of caustic soda. Notwithstanding that this is substantially true there are yet serious defects attached to the quality of the product which render it practically worthless for use in the manufacture of papers of a high quality. This arises from the fact that the silica in the straw remains untouched by the bisulphite solutions, and appears in the cellulose in a very objectionable form. When such cellulose is converted into paper, the silica appears upon the surface of the sheet as small shining scales. It is also obvious that there is only one available method of cheaply removing this silica, which consists in

dissolving it out with a weak solution of caustic soda. My experiments in this direction have proved that this can be done, and that the cellulose produced by this dual treatment is of first-class quality. The yield, however, remained substantially the same as that obtained by digesting the straw in caustic soda alone. In this, as in the Leunig process, all organic substances readily soluble in weak caustic solutions are removed.

Some very interesting figures on the question of yield which I here reproduce were given in the *Papier Zeitung* some few years ago by Roth. I understand them to be the results obtained in actual manufacture, and as such are very instructive to the pulp and paper maker. They clearly show that the yield from unit weight of straw varies inversely with the quantity of caustic soda used for digesting, and that the amount of bleaching powder required to bleach the pulp varies directly with the yield. Assuming the temperature (or pressure), the time used for digestion, and the quality of the straw with respect to its percentage of cellulose to be kept practically constant, I have found by a long series of experimental trials that the above conclusions are strictly correct.

These manufacturing yields as given by Roth seem to indicate that it is possible to obtain 50 per cent. of bleached cellulose from straw, but whether the product corresponded in quality to what is found in the market as straw pulp seems to be very doubtful. The fact that 7 cwt. of bleaching powder were required to bleach one ton of it plainly indicates that it was of low quality. It is of course possible to treat straw with weak caustic solutions, to obtain a high yield, but this product cannot be looked upon as "straw pulp" of ordinary quality.

In practice, the actual yield seldom exceeds 40 per cent. In one pulp mill with which I have been connected, working a mixture of oat and wheat straw, with occasionally a small parcel of rye and barley, all grown in Holland, the yield of bleached cellulose containing 10 per cent. of moisture fluctuated over a long period between 40 per cent. and 41 per cent.

The bleaching powder required to bleach this air-dry pulp varied from 18 to 20 per cent. These results closely correspond to those given by Roth in the Austrian factory.

Lately the so-called "sulphate process" has been applied in a German factory, and as the digesting fluid consists largely of sulphide of sodium, which, as might be inferred from its properties, has a less solvent action on the cellulose than caustic, it is very probable that the yield obtained in this process exceeds that of the caustic process pure and simple. I am, however, unable to state definitely whether this is so or not, or what the yield actually is.

Passing now from the question of yield, I come to another of great importance, namely, the influence of the quantity of silica in straws on the loss of soda, and a consideration of the methods for mitigating this loss. It is almost unnecessary for me to remind you that the composition and quantity of the ash found in straws varies very greatly. The most complete analyses of the ashes of straws, which I have been able to find, are those published by Wolff (Ashen Analysen). The following table embraces his average results:—

COMPOSITION OF THE ASHES OF STRAWS. WOLFF'S "ASHEN ANALYSEN."

	Total Mineral Matter in Straw.	Percentage Composition of the Ash.								
		K <sub>2</sub> O	Na <sub>2</sub> O	CaO	MgO	Fe <sub>2</sub> O <sub>3</sub>	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	Cl	
Barley straw, average of 4 analyses.....	8.10	25.40	1.92	7.53	2.62	2.18	3.94	3.91	51.43	3.75
Oat " " 8 " .....	7.77	38.37	3.99	4.23	2.53	1.70	2.66	3.06	35.08	7.09
Rye " " 3 " .....	4.32	12.16	0.74	11.10	4.45	3.19	8.97	5.57	36.86	3.68
Wheat straw, one analysis .....	3.25		1.00	6.82	1.00	1.02	3.20	5.78	65.34	0.20

While the straw is being boiled substantially, the whole of the silica passes into solution as silicate of soda. The alkali thus combined is practically lost to the pulp maker, because it is rendered inactive for the process of digestion. The quantity of alkali thus rendered useless will vary, of course, with the amount of silica contained in that quantity of straw which will produce one ton of air-dry bleached cellulose on the quantity of potash rendered soluble, and also on the chemical constitution of the silicate. There being an excess of caustic soda always present, I have assumed the silicate to be a normal one, having the formula  $\text{Na}_2\text{SiO}_3$ . Assuming this to be correct, I have calculated, as shown in Table U., first, the quantity of straw required to produce one ton of air-dry cellulose; second, the amount of silica contained in this quantity of straw; and then, finally, the quantity of soda, reckoned as 60 per cent. alkali ( $\text{Na}_2\text{O}$ ), which would combine with this silica. Going a step further, by way of example, and assuming 21 lb. of 60 per cent. caustic to be the amount required to digest 1 cwt. of the Dutch wheat straw (Hypolden) above given, the total alkali (60 per cent.) used to produce one ton of cellulose is 10.29 cwt., and of this 4.39 cwt., or 42 per cent. would be neutralised by this silica.\*

It is this loss which increases the cost of soda so enormously in straw cellulose works. Unless the silicate of soda in the waste lyes or the recovered ash can be decomposed in some practical or economical way, or the recovered ash sold as such, the present highly efficient systems of soda recovery by multiple evaporation, by which 80 per cent. of the soda can be easily recovered, are of little value to the straw pulp maker. Indeed, in most cases where the silica approaches in quantity anything like that shown in the table, the recovered ash is of little value for further use in digesting. For reasons formerly stated, it is manifest that the most economical course to follow is to select those straws only for the manufacture, which by chemical test are shown to contain the least quantity of silica.

There are several methods, alike in principle, of reconverting this silicate of soda into available caustic alkali. The most simple way consists in adding bicarbonate of soda to the solution of the recovered ash, whereby the silicate is decomposed, normal carbonate of soda being formed with the separation of free silica. The same decomposition can be brought about by pumping gaseous carbonic acid through the liquor, but as this gas is not always available, and the cost of pumping is a serious item, and that the silica thus separated is in a very gelatinous state, this system is not practicable. Messrs. Sutherland and Kynaston, who took out a patent some years ago for the use of bicarbonate, claim to obtain the silica has a heavy or granular precipitate, in which state it can be separated from the carbonate of soda liquor with comparative ease. The separation of the silica is one of the great difficulties of this process. The use of bicarbonate serves the additional purpose of replacing the alkali which is invariably mechanically lost, but if the quantity of silica and the percentage amount of soda recovered are both great it can be shown by a simple calculation that the amount of bicarbonate required to effect the decomposition, abnormally increases the volume of the alkali liquors in circulation; that is to say, more bicarbonate is used than corresponds to the soda mechanically lost.

The next most important constituent of the ashes of straws that calls for particular attention is potash. While silica is considered as an impurity and of no value, the potash, on the other hand, might be utilised and turned to account. This problem is, however, as difficult to solve as that of getting rid of the silica in the waste soda lyes. If we refer to the table of composition of the ashes of straws, it will be found that the potash is very considerable in quantity, that, in fact, it ranks next to silica in this particular. It appears highest in the oat straw, rye coming next, then barley, and finally wheat. The oat straw contains more potash than silica, indeed, 2.98 per cent. of its weight. Assuming, as before, that 49 cwt.

of this straw will yield a ton of bleached cellulose, the amount of potash which passes into solution for this quantity is 1.46 cwt., which represents nearly 14 cwt. of potash alum. The above barley straw would yield a very similar amount, whilst the rye and the wheat would yield proportionally smaller quantities.

Some years ago I made several experiments with a view to recover the potash if possible in a useful state. The separation of it from the soda is very difficult, and so far as I am aware can only be accomplished in an imperfect way by crystallisation. The solubility of carbonate of potash being much greater than that of the corresponding soda salt. Of course it is practically impossible to get the potash by itself in a comparatively pure state in this way, and commercially it is too much contaminated with soda to be of much value, excepting probably in the artificial manure trade. I have, however, attempted to obtain it in the form of an alum by neutralising the alkalies in the mother liquors, after crystallisation, with sulphuric acid, and then adding sulphate of alumina and further concentrating and crystallising, obtaining a crop of crystals. These, however, consisted largely of sulphate of soda (Glauber's salts), but undoubtedly contained crystals of potash alum.

This method, the principle of which was applied by Newlands many years ago to the recovery of potash from sugar solutions, would be costly to carry out in practice, as it involves first the separation of the silica from the recovered ash; second, the separation of the bulk of the soda from the potash; and finally, the purification of the alum. On the other hand, the advantages of being able to purify the waste lyes, together with the value of the potash recovered would, I dare say, allow of considerable expenditure in dealing with the problem.

#### IV.—Fuel, &c.

It is always an important addition to manufacturing experience to be able to estimate from well-established data the amount of steam or coal consumed in carrying on any manufacture. The difficulties to be overcome in doing this naturally depend on the nature of the operations to be carried out, whether in fact heat is consumed in bringing about chemical reaction, or in the generation of mechanical force by any of the well-known types of steam engines; or in the process of artificial drying or evaporation. All these, namely, heat for—(1) accelerating chemical reaction; (2) for generating mechanical power; and (3) for drying, are used in the preparation of straw cellulose; and the conditions of the manufacture being well defined it is a simple matter to calculate the lowest amount of steam required to perform the various operations in the different departments of a straw pulp factory. It is not my intention to include in this paper the various formulae I employ to ascertain these items, as they more properly belong to another subject, but as this paper would be incomplete without reference to the fuel used to produce a ton of cellulose, I will simply state the results I have obtained by the application of these formulae. It will be convenient to calculate the quantity of steam required for each operation and then to total the amounts.

a. *Steam for Digesting the Straw.*—It is obvious enough that the quantity of steam required to boil straw depends upon the temperature at which the digesting process is conducted, the proportion of caustic lye to straw being in nearly all cases about the same. The higher the pressure or temperature (for the one corresponds to the other) the more heat is required to raise it to that temperature. If therefore, we know the respective weights and specific heats of the caustic lye, straw and boiler (wrought iron) it will be easy to calculate the total heat units required to raise them to the maximum temperature, and as the heating is done with high pressure steam it will not be difficult on the other hand to calculate the weight of steam that is needed to impart this necessary amount of heat. It is on this principle, one which indeed is applicable to a great number of parallel cases and is commonly known, that I ascertain the lowest amount of steam required to boil straw.

\* An allowance of course must be made for the amount of potash present in the straw as a silicate.

To take the individual case above quoted in which a charge of 40 cwt. of straw and 16,915 lbs. of caustic soda lye of 10<sup>15</sup> Tw. were boiled in a wrought-iron boiler weighing 7 tons at a maximum pressure of 60 lbs. above atmosphere (i.e., 307° F.) there were 4,199 lbs. of steam required to raise the boiler and contents to 307° F. This weight of steam represents the lowest amount that can possibly be consumed in performing the work. By a simple proportion sum, the steam needed to digest 49 cwt., or that quantity which produces a ton of cellulose can be ascertained. It is necessary to make an allowance for loss of heat by radiation which invariably takes place, but as this varies in different factories it can only be properly estimated by direct investigation. My own experience, which is somewhat limited in this particular manufacture, is, that an allowance of 20 per cent. is ample for well-arranged works in which the steam boilers are in close proximity to the digestors.

*b. Mechanical Power.*—This is usually generated by a steam engine of the simple condensing, compound, or triple expansion type, and as the steam consumed in the development of one indicated horse power per hour by each of these types of engines is generally known, and can, in any case, be estimated with accuracy, if the total amount of power expressed in horse power required to produce a ton of cellulose be known, the steam necessary for this department can be easily calculated. In well-arranged works, from 3 to 3½ horse power per hour are required per ton of pulp per week. That is to say, a factory producing 30 tons of air-dry pulp per week will require a mechanical power equal to from 90 to 105 indicated horse power generated continuously throughout the week (144 hours). This power includes the cutting and dusting, disintegration of the pulp, and in fact is sufficient to drive the whole machinery of the mill. Assuming a compound engine of modern construction to be used for generating this force, using say 18 lbs. of steam per indicated horse power per hour, then we have

$$\frac{90 \times 18 \times 144}{30} = 7,776 \text{ lbs. of steam per ton cellulose.}$$

*c. Drying.*—To ascertain by calculation, the amount of steam to dry the wet web of pulp is not so simple, although this case is well defined and can be expressed by a formula of very general application. The wet web of pulp which contains about 55 per cent. of water, 50 per cent. of which must be driven off, is passed over a series of drying cylinders heated by steam of from 8 to 10 lbs. pressure. Having regard to the special circumstances prevailing in such a system of drying it may be fairly assumed that the water in the wet web is evaporated as steam of 212° F., and that the water condensed within the cylinders is ejected from them at a temperature corresponding to the steam pressure within, viz.:—8 to 10 lbs. per square inch above atmosphere. The steam condensed within the cylinders is a measure of the water evaporated. If we know the amount of water to be evaporated from the wet web per ton of air-dry cellulose made, it is not difficult to estimate the necessary quantity of steam to perform this evaporation. The water to be driven off, as above indicated, may be fairly taken at one ton (2,240 lbs.) per ton of cellulose, and the quantity of steam condensed to water of temperature 240° F. (10 lbs. steam pressure) inside the cylinders to evaporate this water, i.e., convert it into steam at 212° F. and under atmospheric pressure, is 2,647 lbs. In this, as in the first case mentioned, an allowance must be made for loss of heat by radiation. After carefully considering similar cases and making tests, I have come to the conclusion that 15 per cent. of the calculated quantity is sufficiently large to cover this loss, in all cases where there are few stoppages in the drying, and where the ends of the drying cylinders are protected by non-conducting materials.

Collecting these results we then have per ton of cellulose.

Steam for digesting, including loss of heat by radiation	Lbs.
20 per cent.....	6,613
Steam for developing the mechanical power.....	7,776
Steam for drying, including allowance of 15 per cent....	3,944
Total steam required .....	17,433

To convert this into coal, it is necessary to know how much water is evaporated in the steam boilers per lb. of coal burnt. Assuming this to be 7 lbs. water, the above 17,433 lbs. of steam represents 2479 cwt. of coal. This, according to my observations, is rather lower than that usually used per ton of straw cellulose made, excluding the coal used for soda recovery, but I venture to think it fairly represents what can be done, and is a direct measure of the possibilities in the hands of pulp makers with regard to the economy of coal.

#### DISCUSSION.

Mr. A. SMETHAM said he had been interested personally in the distinctions which Mr. Beveridge drew in regard to the different straws and their value to the paper-maker, and that more particularly because in their use for feeding purposes various straws had distinct values. He noticed in the tables by Müller that the percentage of cellulose was found to vary to a very large extent, whereas with Mr. Beveridge's estimates the percentages for his practical purposes by the bisulphite tests for the cellulose were, if not identical, very nearly so, in the straws from the various sources. It had been puzzling him how to account for those differences. Doubtless in paper-making a certain quantity of the actual cellulose would be destroyed, partly in bleaching, and it might be for those reasons that the differences existed. He was not aware what process Müller used for the estimation of the cellulose, but taking his figures, and assuming for the moment that Müller had used a process which he (Mr. Smetham) thought, was common with nearly all agricultural chemists, viz., that of boiling with 2 per cent. solution of sulphuric acid, washing with water, then boiling with a 2 per cent. solution of caustic potash, washing again with water, then with a dilute cold solution of hydrochloric acid, and washing free from acid, then with alcohol and ether, and finally drying. It seemed difficult to believe that the quantity of cellulose, as stated by Mr. Beveridge, would really be above that in the table, the more especially as he noticed that the strength of the caustic soda used by the paper-maker was something under one-third per cent. It was true that the digestion went on very much longer in paper-making than in the laboratory (for half an hour) with the caustic; in practice, according to Mr. Beveridge's tables, it went on under pressure at a temperature of 307° for four hours, and possibly therefore there might be gradual loss. He imagined that the process in the laboratory would have been quite as drastic as the process of the paper-maker, and it was certain that Müller's results, so far as oat-straw was concerned, were in accordance with what was found when the straw was used for feeding purposes. The percentage of cellulose in straw depended very largely on the period of growth, and it was possible that the differences between Müller and Mr. Beveridge could be accounted for in that manner. Certain it was that the oat-straw was more valuable for feeding purposes than wheat-straw. He would be interested if Mr. Beveridge could throw some light upon that difference, and it would also be interesting to him if Mr. Beveridge would sketch in a rough way the process which he used on the laboratory scale for estimating the cellulose by the bisulphite test.

Mr. BATESON remarked that there was a process brought forward to add bicarbonate of soda and precipitate silica. Was that process likely to pay? The table exhibited showed that the percentage of cellulose varied a great deal, according to the quantity of caustic soda added. What was the reason of this variation? With regard to the recovery of potash in the residual liquors, would not the potash accumulate, and was there not a point where it would be taken away advantageously?

Mr. BEVERIDGE said the fact was the laboratory method which he had used for estimating the cellulose was that formulated by Müller, and did not involve the use of caustic potash at all, but was substantially that indicated in the body of his paper—namely, treating the sample first with water, then, after drying, with alcohol and benzol, and finally with successive baths of ammonia and bromine water,

an allowance being made for mineral matter in the cellulose residue. It was therefore distinct from the method mentioned by Mr. Smetham, which included digesting the sample in a 2 per cent. caustic potash solution, whose solvent action was much greater than that of ammonia. It was five years ago since he (Mr. Beveridge) tried Müller's method, and he confessed he was not so well acquainted now with the details as he ought to be. He threw over the process at that time because he found it did not give him a correct idea of the amount of cellulose available for paper-making, and, as a manufacturer, preferred the bisulphite method instead. This consisted in digesting the straw for six or seven hours in a bisulphite of soda solution containing about 2.75 per cent. total  $\text{SO}_2$ , about 55 per cent. of which existed in the free state, the other 45 per cent. being combined as monosulphite. The temperature employed corresponded to a steam pressure of 60 or 70 lb. above atmosphere. The apparatus he used consisted of a strong tube of antimoual lead inserted in a small wrought-iron boiler containing water. This boiler, which was heated by gas, acted as a steam jacket. The sample and bisulphite solution were placed within the antimoual lead tube. While managing a large sulphite wood-pulp plant on the Thames, where all the pulp boilers were stationary, he had used another and more convenient and reliable method. This consisted in simply placing the sample of straw and the bisulphite solution in a cylindrical leaden dish, sealing it up with the blow-pipe excepting a small pin-hole in the cover, and placing it inside one of the large wood-pulp boilers. The sample was "digested" under the same conditions of time and temperature as the wood within the boilers. In this way he got excellent results. He had varied the percentage of  $\text{SO}_2$  within certain limits, and had found that the yield of cellulose was not affected by a reasonable excess of  $\text{SO}_2$  as bisulphite. The percentage of  $\text{SO}_2$  must not, however, be too high, otherwise he thought hydro-cellulose was formed. These results were uniformly lower than the percentages given by Müller, and if the latter were correct he could not understand why paper-makers did not get a higher yield than 40 per cent. air-dry pulp by the most careful manipulation in the factory.

He believed that some substance was included in Müller's percentages of cellulose as given in the table which could not rank as cellulose. The fact that, in the instance quoted by Roth, in which a yield of 50 per cent. air-dry pulp was obtained, 7 ewt. of bleaching powder were required to bleach 1 ton of this pulp, plainly showed there was some substance present which required inordinate oxidation. He thought on the whole Müller's results were too high. The percentage of cellulose given by Müller for the oat-straw agreed more closely with his own, but that given for wheat and barley was too high according to his experience. Referring to the points raised by Mr. Bateson, the potash could not be allowed to accumulate in the liquors unless the silica was periodically removed. The amount of this silica was so great that where the percentage of soda recovered was anything like 70 per cent. of the amount put into the pulp boilers, after the recovered ash was used twice or thrice it became practically worthless for further use for digesting. It was obvious therefore that the silica must be removed before the potash could be allowed to accumulate to any great extent. The separation of this silica as a practical manufacturing operation was indeed a very difficult process, but if this could be adequately done, the only way, as far as he knew, in which the potash might be utilised was by the method mentioned in the paper, viz., by separating the carbonate of soda from the carbonate of potash by crystallisation, and, after neutralising the mother-liquors with sulphuric acid, adding sulphate of alumina to get a potash alum.

## THE MANUFACTURE OF CALOMEL IN JAPAN.

BY EDWARD DIVERS, M.D., F.R.S.

*Introductory.*—Calomel, in the form common in England and all countries under western civilisation, is now extensively used and is even manufactured in Japan, under the name of *kankō*. But mercurous chloride is also largely used there, under the name of "light powder," *keifun* (Chinese, *kingfun*), in another and very much older form, which is of signal purity, and made by a simple process as yet quite unknown in Europe. I witnessed this interesting process from beginning to end some years ago, and now make this publication of it to the Society, with full permission of the proprietor of the works I visited, Mr. H. Kokubu, who has aided me in every way he could, and notably with drawings, some of which illustrate this paper.

*Historical.*—According to Terajima Hokyō and Ono Ranzan, writers who lived in the last century, the first-named perhaps a little earlier, calomel was known in Japan as far back, at least, as the beginning of the eighth century, having then been presented to the Empress Gen-miyo; but their authority is the *Zoku Nihongi*, reference to which, Professor Haga, F.C.S., informs me, makes it clear that mercury itself, not its chloride, was the thing presented.

In the time of the writers above named, mercurous chloride was well known and was manufactured in Japan, not only at Isawa, a village in Isé, where it is still made, but also in the city of Osaka and in a town near it, called Sakai. Mr. H. Kokubu, manufacturer, tells me that records exist at Isawamura of his family having carried on the manufacture of *keifun* there for the last three hundred years.

Far earlier, namely, in the tenth century, Minamoto-no-Shitagō, in his work entitled *Wamyo-Ruijushō*, makes mention of a mercurial preparation named *kūfun* or "powder of mercury." It is, however, questionable whether this was mercurous chloride or mercuric oxide, and therefore whether calomel was known or not at this time. But since calomel, under the name of *keifun*, is mentioned by Chinese writers even earlier than this, it may be safely accepted that Japanese knowledge of this body is older than ours in Europe. The western knowledge of chloride of mercury dates from the first half of the sixteenth century, but the distinction between calomel and corrosive sublimate was not recognised till near the end of that century.

*Literary.*—The literature on Japanese calomel is meagre. Japanese writers of the old school have contented themselves for the most part with translating Chinese writings. Ono Ranzan mentions that the Japanese method differs from the Chinese in making use of water in place of alum and other chemicals, in which he came near the truth. The late Dr. Geerts, a Dutch pharmacist, who in the Government service did much in establishing Western pharmacy in Japan, treated of *keifun* in some metallurgical contributions he made to the *Transactions of the Asiatic Society of Japan*. What he wrote is contained in Vol. IV. (1875), and consists of information almost exclusively about Chinese calomel, and derived more from Chinese and Japanese writings than from any experience of his own. Concerning Chinese calomel English readers have the *Notes on Chinese Materia Medica*, among the "Science Papers" by the late Daniel Hanbury, F.R.S., edited by J. Ince. Hanbury mentions as the result of his own observation, the characters of *kingfun* and its great purity but for the presence of minute, transparent, acicular crystals of calcium sulphate. He refers to Porter Smith's *Contributions towards the Materia Medica and Natural History of China* for an account of the manufacture. Smith, however, takes his information solely from Pearson's account on p. 59, Vol. III. of Sir J. Davis's work *On the Chinese*. I have not seen this book, but it is clear from Porter Smith that Pearson, again, has only derived his information from the Chinese *Materia Medica*, *Pun-tsau-kang-muh*, and not from his own observation, and it amounts to this:—Common salt and mercury, of each 1 oz.; alum, 2 oz.; or salt, mercury, copperas, and saltpetre, in some

such proportions, are rubbed together and put into an iron bowl, which is then covered with a roomy earthen dish well luted down. This is exposed to the heat of a strong charcoal fire for four or five hours, when water is thrown on the cover and the cover taken off. On its inner surface the calomel is found adhering in the form of a beautiful, feathery, white sublimate. Ten parts of mercury are said to yield about eight parts of calomel. Dr. Geerts's paper, already referred to, contains essentially the same account, translated from the Japanese version of the Chinese work.

Lastly, there is a paper, in the Japanese language, on the manufacture of *keifun* at Isé, which is the forerunner of the present one. That paper appeared in 1887 in the *Journal of the Tôkyô Chemical Society*, written by Mr. T. Shimidzu, M.E., F.C.S., my former pupil and colleague, and it was his description to me of what he had seen that led to my own visit to Isé in company with Professor Haga in the following year. In one or two points I have availed myself of this paper to make my own account more complete.

**Of the Specific Properties of Keifun.**—*Keifun* is in very thin minute scales, lustrous, transparent, and white or faintly cream-coloured. It might be described as micaceous calomel. To the touch it is soft and smooth. Measured in bulk, dry, it is four times as voluminous, more or less, as the ground calomel prepared by the European process, and can be readily scattered by a puff of the breath. Rubbed hard in a porcelain mortar it gives the brown resinous streak characteristic of calomel, and the evidence therefore, according to pharmaceutical authorities, of its freedom from corrosive sublimate. Exposed to bright sunlight it gradually assumes a light brown colour, a colour, that is, having no affinity to grey or black. Moisture does not seem to favour this change, which is certainly not owing to any reduction to metal. European calomel suffers a similar change. *Keifun* is free from corrosive sublimate and from metallic mercury.

Hanbury found selenite in Chinese calomel, and Geerts found calomel of this form generally adulterated with selenite and mica, but whether what he examined was ever Japanese and not always imported Chinese calomel he does not show. I have found *keifun* as it came direct from Isé quite free from adulteration, and have not met with any adulterated.

#### *Of the Materials used in making Calomel in Isé, Japan.*

—The materials for making Japanese calomel are: mercury, an arenaceous red clayey earth, bay-salt, bittern or salt-mothers, and air. The mercury is imported from Europe, but in old times is said to have been found in the neighbourhood of Isé, as cinnabar.

The earth, called *mitsuchi* ("seed-earth"), is all taken from a neighbouring hill, Shunakayama, and, according to Mr. Kokubu, many other clays have been tried in place of it, always with bad results. It is of a rather light bright red colour, which changes to a duller and somewhat brown red on drying and gently heating the earth, and to a light ordinary brick red by a strong heat. As mined, the earth is seen to consist largely of colourless quartz grains. Besides the quartz a very little biotite is seen sparkling through it. The fresh damp earth does not form a compact mass, but a slightly cohering aggregate of damp crumbs. This texture appears to be due to the earth being a mass of quartz in small grains from the size of a hempseed down to that of impalpable particles, held together by plastic clay. For use, that which does not contain coarse quartz grains too abundantly is selected, and is made into briquettes and moderately baked on the hearth of the fire-place under the calomel pots. These briquettes are then as light and porous as the prepared porous clay used in Fletcher's gas-furnaces. The raw earth, air-dried, is readily rubbed into its constituents by the fingers; and the baked briquettes very easily and rapidly reduced to a soft powder, quartz grains and all, in the agate mortar. The larger grains of quartz in the raw earth are also very brittle. I have treated thus fully of the mechanical characters of the earth, because probably much of its efficiency is due to them. But its chemical character also calls for notice. As baked ready for use it contains in the thoroughly air-dry

condition still 5 per cent and more of water. Before ignition it is almost entirely decomposed by sulphuric acid, either in some days in the cold or quickly by heat. It is also largely acted upon by hot hydrochloric acid, and heated in sealed tubes with this acid to 120–150°, it is almost as fully decomposed as by sulphuric acid. It contains practically no silica soluble in hot sodium carbonate solution, but after acid treatment yields, of course, much silica to this reagent. The composition of the earth, as found in use at the works, but rendered anhydrous, is as follows:—

Quartz .....	58.1
Combined silica .....	24.2
Alumina .....	20.4
Ferric oxide .....	10.5
Magnesia .....	0.3
	107

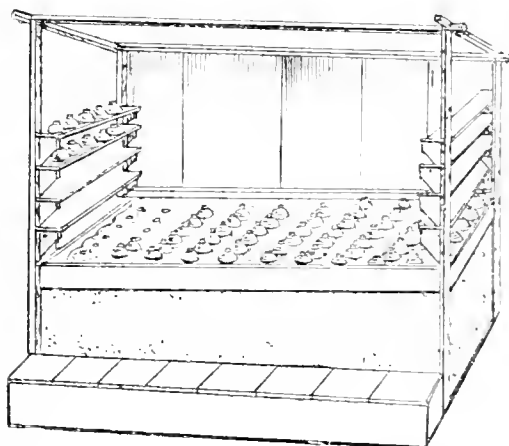
The magnesia is only got by fusion of the finely-ground earth with alkali carbonate, and belongs to the particles of mica scattered through it. Only doubtful traces of phosphoric acid could be found, and, what is specially to be noted, no lime whatever. The earth is thus nothing but quartz, kaolin, ferric hydroxide, and a very little biotite, and is probably valuable to the calomel maker not only for its highly porous texture, but also for its negative chemical qualities.

The bittern and even the rough bay-salt contain magnesium chloride, and this rather than sodium chloride must be the source of hydrochloric acid in the process. That air finds a graduated entrance to the other materials by diffusion during the process, will become evident from a consideration of the set-up of the apparatus.

**Of the Plant.**—The apparatus for making Japanese calomel consists of a table furnace supporting 60 cast-iron pots lined and surmounted with the Shunakayama earth or *mitsuchi*, on which rest, as covers and condensers and receivers of the calomel, unglazed clay cups bottom upwards.

In Fig. 1 the furnace as it is when in action is seen from the working side; 10 pots on the left side are shown still to be charged and covered. The wooden step in front is

Fig. 1.



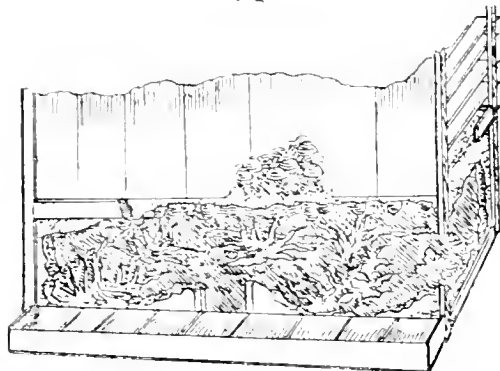
to enable the workman to reach over the table easily when charging the pots or emptying them. In Fig. 2 the furnace is seen from the back or firing side, and with the walls broken away to show its interior and the method of firing. In Fig. 3 the mounting of the pots is shown in three stages by sectional plans of the table.

On a smoothed clay hearth the walls of the furnace are raised in clay, building in the three stones which frame the stove-hole (Fig. 3). The walls are 2.6 ft. high and the enclosure is 7.6 ft. by 4.7 ft., measured outside. The



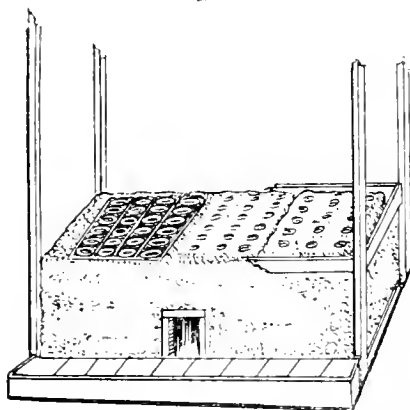
stoke-hole is 1 ft. by 1 ft., but a little wider than this at the base, and is without door. The table of pots and roof of the furnace is constructed (Figs. 1 and 3) by laying a square iron rod on each of the long walls, and on these 11 cross rods, also square, on which are to rest the flanges of the pots. The pots are then put in position as close

Fig. 2.



together as possible, hanging by their flanges, in 10 rows of six each, and plastic clay pressed into the openings left between the flanges and the roots, and the roots and flanges covered in so that only the mouths of the pots remain visible, as shown by the middle rows in Fig. 3. The furnace clay being thoroughly dry it is deeply laid over

Fig. 3.

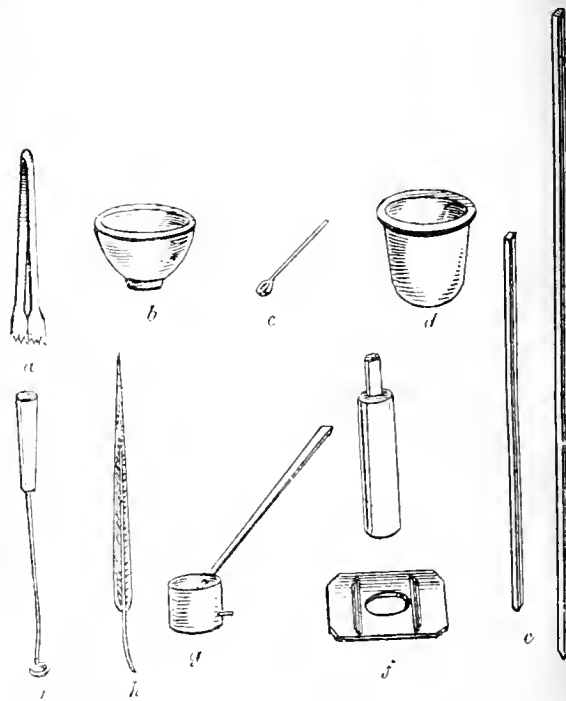


with the red earth mixed with a little bay-salt and moistened with bittorn in small quantity. The pots are also filled with the same moist red earth, except in a central cylindrical shaft (see the left side of the furnace-table in Fig. 1 or the right side in Fig. 3), reaching to the bottom of the pot which is left bare. The pot is 0.5 ft. deep inside, and across its mouth, inside, is 0.45 ft. It is shown in Fig. 4. The shaft or cavity left in the filling is 0.18 ft. in diameter, and is shaped by resting a wooden core on the bottom of the empty pot, and then pressing in the moist earth round it, smoothing off the top, and dropping a perforated board over the projecting core to hold down the earth while withdrawing the core, which is then removed by its handle. The furnace is now ready for work. It should have been mentioned that after the clay walls of the furnace are built they are framed in with wood to increase their stability and to give support to a wooden back and to shelving above the table, as seen in Fig. 1.

**Of the Firing.**—The firing the pots is kept far below what are usually regarded as furnace heats. The fuel employed is wood, and the Japanese are clever in the use of this economically and effectively in firing pots for boiling, evaporating, distilling, or subliming. The method of heating is seen in Fig. 2. Five lengths of firewood are ranged

along the back and front walls on the hearth, generally raised at one end by resting on a lump of clay. In the ordinary working of the furnace, as I saw it, the heat from previous work is sufficient to kindle the fresh wood. The flames rise up the sides and run over the bottoms of the pots, leaving the central space in the chamber free from flame. The air enters by the lower part of the stokehole and the products of combustion escape invisible by its upper part, so perfect is the combustion. At the time of

Fig. 4.



- a. Tongs for lifting and dropping the lumps of wet compost into the pots.
- b. Clay cup condenser.
- c. Brass mercury spoon.
- d. Iron heating pot.
- e. Rectangular iron bars for pots.
- f. Wooden core and board for lining pots.
- g. Bamboo water pot for wetting furnace top.
- h. Quill feather for emptying cups.
- i. Ped for lifting compost ball out of pot.

first lighting the fire, and of irregularities in stoking, some smoke is unavoidable, and to keep the top and table free from this smoke and from ashes, a wooden back is put in above the table, as shown in the figures. There is also a wooden hood and flue above the stokehole to carry off any smoke: this is not shown in the figures, but is similar to those put up in England and elsewhere over the working doors of furnaces to protect the workman from arsenic, sulphur, or other noxious fumes. The heating is so well effected that the pots two feet above the burning logs are made sufficiently hot, barely red-hot at the bottom, and yet the wooden frame on the outside of the furnace is not charred, and the workroom is not unpleasantly warmed.

About three bundles, or 40 lb., of wood serve for one firing, and it is remarkable to see so little fuel working so many pots.

**Of the Working.**—A compost of burnt *mitsuchi* with about a fourth of its weight of bay salt is made up with bittorn into lumps the size of large chestnuts.

The furnace being hot enough, the *mitsuchi* surface of the table is once for all freely wetted by a watering-pot, perhaps half a gallon of water being used, all of which is absorbed; a lump or two of compost is dropped, by tongs or by hand, into each pot in rapid succession, a very small



spoonful of mercury poured into each pot, the total charge for the 60 pots being somewhat less than one pound avoirdupois (more exactly,  $\frac{5}{8}$ th of a lb.), and a clay cup, bottom up, placed over each pot and adjusted by gently pressing and turning it round slightly. The cup is thus made to fit neatly on the earthen top without adhering to it in the least. The cups are thick and unglazed, but become very smooth inside by use. They have an inside diameter at the mouth of 6 ins. and a depth of 3 ins.

Thus arranged, the pots and cups are left for three hours, and during the latter part of this time the furnace is cooling. When cool enough, each cup is lifted in turn, and, with two turns of a feather, the *keifun* or calomel, which fills it in the form of a sparkling network of delicate crystalline scales, is transferred to a sheet of paper held under it, and the cup mouth downwards placed on a shelf of the furnace ready for use in the next operation.

The calomel, it will thus be seen, forms no adherent cake in the cup, but particles only loosely hanging together. So obtained, it is ready for the market, needing only to be packed in small wooden boxes for sale.

The spent lumps of earth and salt are lifted out of the pots by the tool shown in Fig. 4, and when the furnace has become still cooler, the fire is made up again, the furnace top freely wetted, and a new operation set going as before. The furnace is worked twice each day. Nothing could well be carried out with great simplicity and less expenditure of labour and time.

*Of the Yield.*—The loss, I am told, is about 16 per cent. of the theoretical amount of calomel, which is, I believe, about twice as much as is lost in the Western process.

*Experimental.*—If one of the cups is removed a few minutes after the operation has been started, much of the mercury is found in it as a sublimate of fine globules, mixed with only a little almost amorphous calomel, from which it would appear that the formation of the calomel results from reaction in the cup between the mercury in vapour and the active gases.

On dipping into the pot, uncovered during the process, a glass rod with a drop of water hanging to it and then withdrawing it, and testing the remainder of the water with potassium iodide and starch, no chlorine can thus be detected. The same is true when a drop of solution of potassium hydroxide is used. Nor can the slightest odour of chlorine be detected in the vapours issuing from the uncovered pot.

On passing air containing a little hydrochloric acid gas through a tube in which mercury is freely boiling, sparkling calomel is formed close to and mixed up with the mercury.

Red earth which has been used in the process turns moist red litmus paper blue, while fresh red earth is neutral.

*Theoretical.*—The nature of the materials used and the observations gained by the preceding experiments are sufficient to establish that the calomel is formed by a reaction between mercury vapour, oxygen, and hydrochloric acid gas, in which along with mercurous chloride, water is formed— $4\text{Hg} + 4\text{HCl} + \text{O}_2 = 4\text{HgCl} + 2\text{H}_2\text{O}$ , and that the formation takes place at a temperature near—above or below—the boiling-point of mercury, and much below that at which calomel freely volatilises. The source of the hydrochloric acid is certainly the magnesium chloride of the bittern and bay-salt, which heated in a moist atmosphere, even in the presence of sodium chloride, is as well-known partly converted into magnesia and hydrochloric acid. Hence the alkalinity of used *mitsuchi*.

The porosity of the walls of the apparatus, particularly of the layer of *mitsuchi* on which the clay cup rests, must be more than sufficient to allow enough air to enter during the working. I had supposed that the hot hydrochloric acid and air might, in contact with the earth, have yielded a little chlorine, but the temperature reached in the process normally worked seems never to reach that required for the liberation of chlorine.

The Chinese process, if correctly described, differs from that used in Japan in several material points, one of which is that the mercury is intimately ground up with the other materials, and one fails to see what reaction can take place between it and the two others, namely, salt and alum. Heated, the mixture must give off mercury and hydrochloric acid,

and then these, as in the Japanese process, will with air give the calomel, but this is independent of the previous intimate mixture of the mercury with the salt and the alum. Another point is that in place of the magnesium chloride of sea water as the source of hydrochloric acid, the Chinese are said to use alum or copperas, which, with the salt, will react to give hydrochloric acid. A third point is that the cover is said to be closely luted to the iron pot, which must nearly exclude the air, without which it is impossible to explain the formation of the calomel. Perhaps this is the reason why the Chinese process is said to take four or five hours firing, since this may give time enough for the needed oxygen to diffuse through the cup and lining, it will also account for the fact, if it is one, that the yield of calomel is markedly less in weight than the mercury used. Again, there are to be noticed the statements that the iron pot is exposed for hours to the strong heat of a charcoal fire, and that the hot clay cover is cooled by throwing cold water on it, statements which must be regarded as greatly exaggerated, if not erroneous. Lastly, the calomel-giving vapours are allowed to remain in contact with the hot iron of the pot, instead of being kept from it by the thick lining of earth provided in the Japanese process, a contact which such vapours could not stand without destruction. I think, therefore, that we may be fairly doubtful whether any reliable description has yet been given concerning the Chinese process, which we may expect to find to differ little, if at all, from the Japanese process, except in salt and alum being used in place of the mother-liquor of sea-salt or "water," as Ono Ranzan supposed it to be.

One thing to which attention may be called is that the Chinese are stated to add some nitre to a similar mixture when employed to give corrosive sublimate. For that being the case, it is seen that free chlorine, which would here be developed from the salt, nitre, and alum, is necessary for the production of the higher chloride, and that air and hydrochloric acid can only yield the lower chloride, a difference of much theoretical interest, and indeed of practical moment also. It is known that re-sublimation of calomel generates some corrosive sublimate, and, although authorities are not quite agreed as to whether reaction occurs between gold leaf and calomel vapour, it is hardly to be doubted that such reaction does occur. Now I have found that if in the Japanese apparatus the temperature of the cover be raised sufficiently to volatilise much of the calomel the remaining calomel is no longer free from corrosive sublimate. It must, therefore, be borne in mind that the calomel formed in the Japanese process is not, and cannot be, the result of true sublimation, but of precipitation as fast as formed from the three gaseous bodies which give rise to it. At the temperature at which mercury boils, calomel is either quite fixed or, at most, has a vapour of exceedingly small tension. In the two facts, that the three gases do not react to yield corrosive sublimate and that the calomel is not heated to its volatilising point, lie the explanation and, at the same time, the assurance that Japanese (and Chinese) calomel contains no corrosive sublimate.

*Summary.*—The Japanese prepare calomel pure, above all things free from corrosive sublimate. They heat balls of porous earth and salt soaked in bittern along with mercury, in iron pots lined with earth. The heat forms hydrochloric acid from the magnesium chloride in the bittern, and the mercury sublimes into the closely-fitting but unattached clay covers of the pots. Air enters by diffusion and oxygen and hydrochloric acid gas act together in the hollow cover on the vapour given off from the sublimate of mercury there formed. The cover thus becomes filled with a network of micaceous particles of calomel, precipitated at a temperature below its subliming point at the moment of its formation.

This investigation of an interesting product of Japanese industry has been carried out under the authority of the Imperial University of Japan. I cannot acknowledge fully enough the indispensable assistance I have received from my colleague, Assistant-Professor Haga, F.C.S.

# A REVIEW OF SOME OF THE METHODS IN GENERAL USE FOR THE ESTIMATION OF MANGANESE IN MINERALS AND METALS.

BY E. H. SANITER, F.C.S.

THE quantity of manganese ores, spiegeleisen and ferro-manganese sold in this and other countries is very considerable, the price being based on the actual content of manganese present.

As the results of analyses by different chemists vary somewhat considerably when operating on the same sample, I have considered the question to be of sufficient importance to devote a considerable amount of my time to ascertain the relative merits of the various known methods which are at the present time in general use.

I believe it is a fact that in England the process most generally used is that of Mr. Pattinson, or a modification of it used by Mr. Riley, in which carbonate of lime is replaced by zinc oxide.

On the Continent the volumetric process of Volhard is extensively used.

In America, according to Blair, the gravimetric process, in which the manganese is weighed as pyrophosphate, is considered to be the most accurate.

The gravimetric method, in which the manganese is weighed as  $Mn_3O_4$ , is, I believe, used more or less in all countries.

My first object was to obtain a substance containing a known quantity of manganese.

The manganic-manganic oxide, which, I believe, is generally used, is not to be relied upon unless very extraordinary precautions are taken in its preparation.

I have found it impossible to obtain it with the theoretical percentage of 72.05 per cent. of manganese, when all that metal exists as  $Mn_3O_4$ .

The following table gives the complete analyses of precipitates prepared by different processes.

These analyses have been conducted with the utmost care.

TABLE I.

Batch No .....	1.	2.	3.	4.
Mn <sub>3</sub> O <sub>4</sub> .....	Per Cent. 97.85	Per Cent. 99.51	Per Cent. 99.70	Per Cent. 99.788
Excess of O.....	0.03	0.13	0.05	0.020
MgO.....	0.16	0.04	0.03	0.016
CuO.....	0.75	0.05	0.14	0.125
SiO <sub>2</sub> .....	0.60	0.24	0.02	0.006
Fe <sub>2</sub> O <sub>3</sub> .....	0.25	Nil	0.04	0.045
NaO and Co.....	0.22	0.3	0.02	
CuO.....	0.14			
	100.00	100.00	100.00	100.00
Manganese.....	70.5	71.69	71.83	71.90
Bracketed impurities ...	1.21	0.27	0.08	0.051

The bracketed impurities are those which would be in part removed by Riley's after-treatment referred to later on.

The  $Mn_3O_4$  is taken by difference.

No. 1 batch consists of  $Mn_3O_4$  precipitates produced in the analysis of ferromanganese by the gravimetric method without after treatment of the precipitate.

No. 2 batch. A pure solution of manganese chloride was prepared from 84 per cent. ferromanganese by the careful separations of Si, Fe, Ni, Co, Cu. The manganese was then precipitated with bromine and ammonia in a glass flask, the resulting precipitate being converted into  $Mn_3O_4$  by calcination. The largest impurity is silica, dissolved from the flask by ammonia.

Nos. 3 and 4. These batches were prepared from a pure solution of manganese by precipitating in the cold with carbonate of ammonia in a porcelain dish, the precipitate

TABLE II.

Batch.	Actual Mn. present.	Manganese found by			
		Weighing as		Volhard's	Pattinson's
		Manganic- Manganic Oxide.	Pyrophosphate.	Volumetric Method.	Volumetric Method.
No. 1—		Per Cent.	Per Cent.	Per Cent.	Per Cent.
A. ....	70.5	..	..	70.19	69.79
B. ....		..	..	70.05	69.96
Average .....		..	..	70.12	69.87
No. 2—		..	71.74	71.86	70.93
A. ....	71.69	..	71.77	71.41	70.80
B. ....		..	71.75	71.38	70.86
Average ...		..	71.75	71.38	70.86
No. 3—		..	71.88	71.50	71.09
A. ....	71.83	..	71.80	71.50	71.17
B. ....		..	71.84	71.50	71.13
Average .....		..	71.84	71.50	71.13
No. 4—		72.05	71.95	71.39	71.22
A. ....	71.90	71.90	71.94	71.45	71.26
B. ....		71.97	71.94	71.42	71.24
Average .....		71.97	71.94	71.42	71.24
Average of above results, No. 1 batch excepted .....	71.81	..	71.84	71.43	71.07

being subsequently calcined. It would be an improvement to precipitate the manganese as sulphide with sulphide of ammonium instead of carbonate, by this means separating the lime and magnesia. The sulphide of manganese is then converted into  $Mn_3O_4$  by calcination.

Having now obtained a quantity of manganomanganic oxide containing known percentages of manganese, the next step was to carefully estimate the manganese in each of the samples of  $Mn_3O_4$  given in Table I. by the four methods, the accuracy of which it was proposed to investigate.

The results of these analyses are given in the table at the bottom of previous page.

Results marked A are by my assistant and B by myself. Ashless filter-papers have been used and every possible precaution taken to ensure accuracy, the burettes being calibrated, and dichromate solution standardised on two different specimens of "pure iron," both of which had been completely analysed for impurities, the one being that used by Mr. Stead, of Middlesbrough, for this purpose; the other, that which I have always used myself, and which corresponds exactly with that of Mr. Stead.

In reviewing the results given in this table, I propose to consider each method separately as to its general usefulness and as to its accuracy, by comparing the results obtained by it with the actual percentage of manganese given in the first column.

**Estimation as  $Mn_3O_4$ .**—The ordinary method of conducting this operation, is, after separating the silica and iron &c. with ammonia and ammonium acetate, redissolving, and reprecipitating, to precipitate the manganese in the filtrate with bromine and ammonia, small quantities of Ni, Co, and Cu, not being previously separated, boil, filter, wash, dry, and calcine the precipitate at an almost white heat to  $Mn_3O_4$ , weigh.

From the weight thus obtained, reckoned as pure  $Mn_3O_4$ , the percentage of manganese is calculated.

In order to ascertain what amount of error was introduced by this procedure, an average lot of about 20 precipitates, obtained in estimating ferromanganese, similar to that shown in Table IV., were ground up together, and analysed for impurities as shown in Table III.

TABLE III.

ANALYSIS OF  $Mn_3O_4$  PRECIPITATES.

$Mn_3O_4$ .....	99.09 per cent. = 71.4 per cent. manganese.
Excess of oxygen .....	0.02 "
Silica .....	0.21 "
Peroxide of iron .....	0.05 "
NiO and CoO.....	0.22 "
CuO.....	0.14 "
Ag.....	0.16 "
FeO.....	0.07 "
	100.00

After making the necessary allowance for filter-ash, the precipitates contained only 71.4 per cent. Mn. against a theoretical 72.05 per cent., thus showing an excess of 0.91 part on the 100 parts of manganese present, when assuming these precipitates to be pure  $Mn_3O_4$ .

It should, however, be stated that many chemists, after weighing the  $Mn_3O_4$  precipitate, dissolve it in hydrochloric acid, and separate the  $SiO_2$ ,  $Fe_2O_3$ ,  $NiO$ ,  $CoO$ , and  $CuO$ , the combined weight of which is deducted from the original weight of the  $Mn_3O_4$  ppt. before calculating. This procedure considerably reduces the error, but still leaves that due to lime and magnesia. When the method is applied to estimation of manganese in manganese ores where much lime, magnesia, and baryta are present, this source of error becomes very serious, and may be as much as 1 to 2 per cent.

The bracketed impurities in Tables I. and III. are those which are removed by this after-treatment of the precipitate.

As a check on Table III., showing impurities in  $Mn_3O_4$  precipitates, a sample of ferromanganese was subjected to complete analysis, the manganese being estimated as already described and calculated, taking the precipitate as pure  $Mn_3O_4$ ; also with the correction shown to be necessary in Table III.

The result is given in Table IV.; the totals obtained confirm the analysis in Table III.

TABLE IV.

## COMPLETE ANALYSIS OF FERROMANGANESE.

	Per Cent. Stead's Tables as usual.	Per Cent. 81.27% Corrected by Table III.	Per Cent. 81.19% By Pottal son's method, corrected by Table II.
Mn	100.00	100.00	
Fe	0.20	0.20	
Cu	0.20	0.20	
Ni, Co	0.20	0.20	
C	0.79	0.79	
Si	0.65	0.65	
P	0.23	0.23	
	100.65	100.94	

Even when this process is worked with every possible care, all metals, except manganese, being separated, the use of a glass vessel for precipitation contaminates the precipitate with a considerable amount of impurities, as shown by Batch 2, Table I., which only contains 71.69 per cent. of manganese and is prepared in this way, a quarter per cent. of silica being taken up from the glass.

**Low results** are also obtained from several causes, the chief of which is the presence of ammonia salts in the  $MnO_2$  precipitate when being calcined, which gives rise to a deficiency of oxygen and in some cases a volatilisation of manganese. In order to demonstrate this, two precipitates of equal weight of hydrated  $MnO_2$  were calcined, the one being washed, the other unwashed, and containing a large quantity of acetate, chloride, and bromide of ammonia, with the following result:—

	Washed.	Unwashed.
	Grm.	Grm.
Weight after calcination .....	0.568	0.482

A deficiency of 0.086 gm. on the unwashed.

In order to determine the extent to which the O in  $Mn_3O_4$  varies, a considerable number of precipitates were tested for O in excess of  $MnO$ , and the O was found to vary between 4.0 and 7.2 per cent., thus confirming the results obtained by previous investigators. *This source of error, alone, condemns the method;* the low oxygen results being attributable in part to the too rapid heating of the oxide in the presence of ammoniacal organic salts.

**A Modification of the  $Mn_3O_4$  Method.**—If after the separation of all other metals from the solution of manganese, the manganese is precipitated with ammonium sulphide, filtered and washed, as directed by Fresenius (Quant. Anal. Vol. 1. p. 204), and the precipitate calcined, very strongly nearly pure  $Mn_3O_4$  is obtained.

The  $Mn_3O_4$  results in Table II., Batch 4, were obtained in this manner, and are fairly accurate.

The precipitates obtained by this method are less likely to vary in their percentage of oxygen, as their physical condition is fine and porous. Both precipitates were tested and showed 6.97 per cent. of oxygen in excess of  $MnO$  and no sulphur.

The solubility of the sulphide precipitate seems to be very slight, the filtrate being examined and containing respectively 0.3 and 0.4 mgrm. of manganese, which was added to the result.

**The Pyro-Phosphate Method.**—This method when properly conducted is generally conceded to be the most accurate process for the estimation of manganese, and this is borne out by the results shown in Table II.

The directions given by Blair (*Anal. of Iron and Steel*), if strictly adhered to, give good results. There is, however, one point in which I think a slight improvement might be made.

After the separation of all other metals, there appear to be small quantities of lime and magnesia along with the manganese, even when working on spiegel or ferromanganese, as shown by analyses in Table I., Batches 3 and 4, which were precipitated as carbonate from this solution.

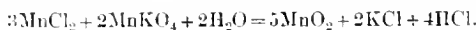
The lime and magnesia probably accumulate from vessels and reagents used during the numerous previous operations.

In order to separate this lime and magnesia, the manganese should be precipitated as sulphide, as previously described, and, after washing, be dissolved through the filter with hot hydrochloric acid and washed with hot water, the filtrate being collected in a platinum or porcelain basin, the former preferably, for precipitation as phosphate. The most important points in Blair's description, and points which cannot be too strongly insisted upon, are:—

(i.) The crystalline state of the precipitate. (I have obtained results 5 per cent. too low through inattention to this point.)

(ii.) The washing with cold ammoniacal solution of nitrate by ammonia. Hot water should not be used as it dissolves the precipitate and causes low results.

*Volhard's Volumetric Method.*—This method depends on the precipitation of manganese from a slightly acid solution by permanganate of potash, as shown by the following equation:—



I have used it as follows:—A quantity of the substance equivalent to 0.4 gram. of manganese is dissolved in hydrochloric acid.

Separate iron and chromium, if present, with ammonia and ammonium acetate and precipitate manganese with bromine and ammonia.

After ignition this precipitate is dissolved in hydrochloric acid and neutralised with zinc oxide suspended in water, any excess being dissolved by adding nitric acid drop by drop.

The solution is then diluted to about one litre with boiling water and titrated with a solution of permanganate of potash until a permanent pink colour is obtained.

The solution of permanganate is carefully standardised against pure iron or a solution of ferrous sulphate of known strength. From the figures thus obtained the percentage of manganese is calculated. On referring to Table II. it will be seen that the results obtained only indicate 99.5 per cent. of the manganese.

This points very forcibly to the necessity of standardising the permanganate solution with oxide of manganese of known composition, instead of, as generally recommended, against iron, as there is always a doubt as to the precipitate being absolutely all  $\text{MnO}_2$ .

The results obtained on Batch 1, Table II., show that small quantities of nickel and copper do not affect its accuracy.

*Pattinson's volumetric method*, which depends on the precipitation of the manganese as  $\text{MnO}_2$  and its titration with ferrous sulphate, is fully described in Mr. Pattinson's paper on "The Determination of Manganese" (this Journal, Vol. XI., p. 336), and in using it the instructions there given have been exactly followed out.

On referring to the results obtained by it in Table II. it will be seen that they only show about 99 per cent. of the manganese present.

Looking at the fact that this method is very largely used, and that Mr. Pattinson says in his recent paper that he always obtains results showing 100 per cent. of the manganese present, I thought it desirable to obtain further evidence as to the accuracy attainable by it. I therefore sent out portions of No. 4 batch of oxide, sufficient for duplicate estimations, to five experienced chemists all accustomed to use this process for the estimation of manganese.

The following are the results, including those of my assistant and self:—

#### NO. 4 BATCH OF OXIDE, CONTAINING 71.9 PER CENT. OF MANGANESE.

No. 1....72.08 per cent. = 100.25 per cent. of manganese present.

" 2....71.93	" = 100.04	"	"	"
" 3....71.26	" = 99.11	"	"	"
" 4....71.22	" = 99.06	"	"	"
" 5....71.20	" = 99.03	"	"	"
" 6....71.15	" = 98.96	"	"	"
" 7....71.03	" = 98.79	"	"	"

The first two results confirm Mr. Pattinson's experience; the other five agree in indicating only about 99 per cent. of the manganese present.

These results show that on the same sample different chemists find considerable variations, due to this method. I find, however, that the results obtained are constant, even if low, when the method is worked by one person under the same conditions.

The results obtained on impure oxide, Batch 1, Table II., agree with Mr. Pattinson's conclusion that small quantities of copper and nickel do not materially affect its accuracy.

I have tried the modification of this method, in which carbonate of lime is replaced by zinc oxide, and find that it is very uncertain in its results, but when the precipitate is submitted to a very much more thorough washing than is necessary with  $\text{CaCO}_3$  the results are the same. Much excess of  $\text{ZnO}$  should be avoided.

*The above facts show the absolute necessity for every chemist, using this method, to carefully standardise his working on manganese oxide of known composition, and to use the factor thus obtained in calculating his results.*

*General Conclusions.*—The weighing of manganese as  $\text{Mn}_2\text{O}_3$  is utterly unreliable if precipitated as  $\text{MnO}_2$ , but is somewhat better when precipitated as  $\text{MnS}$ .

The weighing as pyrophosphate is accurate if the solution is absolutely free from other metals; the numerous operations necessary render this very tedious.

The volumetric methods of Pattinson and Volhard have a tendency to give low results in the hands of many chemists, and if used should be carefully standardised on manganese oxide of known composition at frequent intervals. Thus standardised they are accurate and more rapid than the gravimetric methods.

I cannot close this paper without acknowledging the hints as to procedure derived from Mr. Pattinson's recent paper on the "Estimation of Manganese," and also recording my thanks to my assistant, Mr. Byrom, for the careful manner in which he has checked my results.

#### DISCUSSION.

Messrs. J. and H. PATTINSON wrote as follows:—

"Through the kindness of Mr. Saniter, some ten days ago we saw a rough draft of his paper. We have not a copy by us, and we do not know how much he may have modified it, but speaking generally we conclude that the gist of his paper, so far as regards Pattinson's process for testing manganese, may be summed up in two statements—1st, that in Mr. Saniter's and some other chemists' hands, this process does not indicate the total amount of manganese present; and 2nd that, when using Pattinson's process, every chemist ought to check it against a compound of manganese of known composition, so as to determine in this way a coefficient by which to correct the result obtained by Pattinson's method."

"We regret that Mr. Saniter did not point out to us sooner the differences between his working of the process and ours, for we believe that these differences will be found to be caused by some easily adjusted differences in the reagents or instruments used, or in the manner of carrying out the process, and not by any fundamental error inherent in the process. We feel convinced that this method, in the hands of any moderately skilled chemist, should indicate 100 per cent. of the manganese present. We are told by Mr. Saniter that in testing manganese ores he invariably obtains

a lower result than we do ourselves, and he says that several other chemists' results confirm those obtained by him. On the other hand, during the last 15 years that we have used this process, we have had a large number of instances to show that other chemists of undoubted standing can obtain the same results by it as we do. This method was also examined by Dr. C. R. A. Wright with Mr. Menke (see *Jour. of the Chem. Soc.*, Vol. XXXVII., pp. 22 and 19), and by Professor Ledebur (*Chemiker Zeitung*, Vol. VIII., pp. 910, 927, and 963; *Jour. of the Iron and Steel Institute*, 1884, No. 1, p. 269) and found by them to give correct results. We may add that we have recently had an opportunity of checking the method in the case of two samples of manganese ore analysed by Professor Fresenius, who determined the manganese by the gravimetric method described in the second volume of the sixth German edition of his "Quantitative Analysis," p. 390, and in one case the percentage of manganese found by us was 0.01 per cent. higher and in the other case 0.02 per cent. higher than that found by Fresenius; that is, in both cases the results are practically identical."

"With regard to Mr. Saniter's recommendation that each chemist should check the method for himself with some pure compound of manganese or some compound of manganese in which all the impurities have been estimated with a view to determining the correction that requires to be made upon his particular method of working our process, we think that this would lead to still greater differences amongst chemists than now exist owing to the very great difficulty there is in making pure compounds of manganese, or in estimating small quantities of impurities in them."

"We are sorry that we have not had an opportunity of testing any of the preparations of manganomanganic oxide ( $Mn_2O_4$ ) made by Mr. Saniter, but Mr. Stead, of Middlesbrough has tested both some of Mr. Saniter's preparation of this oxide and some made by us, and in both cases he found the theoretical yield of manganese to be given by Pattinson's process."

"We trust that we may have an opportunity of comparing our methods of working with Mr. Saniter's methods more thoroughly than we have yet done, when we have no doubt but that some simple explanation will be found to show why he gets lower results by this method than we do."

Mr. G. WATSON GRAY thanked Mr. Saniter for having brought his experience before them. Although Liverpool was not a centre where much steel was dealt with, yet manganese was used in the district, and therefore had a certain amount of claim on their attention. The estimation of manganese by precipitating with ammonia and bromine was of course very long and tedious an estimation generally occupying a couple of days. In addition to the many impurities that required removing from the precipitate in order to arrive at a correct result, Messrs. Pattinson have pointed out in the *Journal* that when the method is used it ought to be supplemented by estimating the amount of peroxide of manganese present, and thereby arrive at the correct amount of oxygen contained in the ignited precipitate, for it is not always  $Mn_2O_4$ . They have shown in their paper what a good many chemists knew—that a great deal depended upon the thickness of the platinum crucible, along with the time and other conditions of heating, whether it be a bright or dull red or a white heat, and many more apparently trifling things. The thickness of the platinum crucible might seem strange to some, but Messrs. Pattinson's experience only confirmed what other chemists have observed. The pyrophosphate or the second method dealt with by Mr. Saniter had always given high results in the speaker's hands. It certainly seemed to be the method used in the States, where they always managed to find more manganese in a sample than is done in England. Volhard's permanganate method had the disadvantage of requiring the permanganate to be standardised daily, in addition to many other minor points of an objectionable character, and he did not wonder that Mr. Saniter found a higher percentage of manganese than when using the Pattinson method. Messrs. Pattinson's method had always given within 0.1 of 100; it may have exceeded 100, as already shown by Mr. Saniter, and in other cases it might be 0.1 below 100. He thought that a good many of the other faults that had

been found with the use of the method were due to chemists not taking due note of the details. If Mr. Saniter worked out Pattinson's modified method, published in the *Journal*, using a small amount of chloride of zinc along with the ferric chloride, he would doubtless find 100, if he has been getting a low result by neglecting the addition of zinc chloride, because the latter seemed to have the advantage of preventing the formation of permanganates. Using oxide of zinc as the precipitant in place of carbonate of lime was objectionable, as the chlorine was thereby very difficult to wash out. Mr. Pattinson originally tried it years ago when he first brought the method out. Some one on the Continent suggested the use of zinc, and probably that accounted for Mr. Riley adopting it. Probably in some cases the burettes had not been correct, as a good many were slightly out in the graduations, and when they came to 100 per cent. a little went a long way. In the preparation of pure oxide he could quite endorse what Mr. Saniter had said. It was very difficult to prepare a pure compound, and for that reason he would agree with Messrs. Pattinson in recommending that chemists do not start to make their own corrections for bad workmanship, because those who had prepared the pure oxide knew how very hard it was to obtain anything like purity.

Dr. KOHN asked whether Mr. Saniter had any direct evidence of the volatilisation of manganese in the presence of ammonium salts when it was ignited as manganomanganic oxide, as mentioned in the paper? Also, had Mr. Saniter tried the electrolytic method for the separation of the manganese, since it occurred to him (Dr. Kohn) that that might be an advantageous method for the separation of the manganese previous to its final determination on account of the freedom of the separated manganese peroxide from impurities, such as silica?

Dr. HURTER asked what was the value of one unit of manganese in ferromanganese, and was it possible to draw two samples of the same delivery of ferromanganese which did not differ more in composition than the various analyses enumerated differed among themselves?

Mr. SANITER, in reply, said that as far as he was aware there were no differences between his working and Mr. Pattinson's working of his process. The only difference is in the results obtained, the instructions given by Mr. Pattinson for the working of the process having been strictly followed out. He did not doubt Mr. Pattinson's results, which generally agreed with his own corrected result. He wished distinctly to state that, as shown in the table, some chemists had obtained 100 per cent. Mr. Pattinson had referred to a manganese ore which had been tested by himself and by Professor Fresenius. He had also an opportunity of testing that manganese ore, and he also agreed with Messrs. Pattinson and Fresenius within a few hundredths per cent., using the co-efficient obtained by testing some  $Mn_2O_4$  supplied by Mr. Pattinson, accepting his statement as to the amount of manganese which the  $Mn_2O_4$  contained, which he believed to be correct, simply because it confirmed his previous experience as given in Table II. He obtained only 99.35 per cent. of the manganese present in that  $Mn_2O_4$  prepared by Mr. Pattinson and used that co-efficient to calculate the ore just mentioned and then the result obtained read within a few hundredths per cent. of the results obtained by Fresenius and Mr. Pattinson. He quite recognised that Mr. Pattinson's results were 100 per cent.; at the same time he was not prepared to accept the opinion that the difference would be sure to be cleared up. The differences were of very long standing and had not yet been cleared up. His paper did not pretend to explain why those differences occurred, but to suggest the means of avoiding discrepancies in the ultimate analyses reported by chemists. Mr. Pattinson had also remarked that his method had been investigated by several distinguished chemists some considerable time since, amongst whom Professor Ledebur is mentioned. Now it was a fact, he believed, that in Germany Volhard's method was said to give 100 per cent. of the manganese present. Now if this were so his (Mr. Saniter's) results, being constantly different, should yet agree on the two methods. They did not. Volhard's

method gave higher results as compared with Mr. Pattinson's method. Mr. Pattinson also mentioned the difficulty in obtaining pure manganese compounds, but although it was difficult, he (Mr. Saniter) thought that any chemist, after he had obtained a fairly pure compound, should be able to analyse it for impurities, and thus arrive at the manganese it actually contained. Mr. Pattinson rather narrowed the point down to one between himself and the speaker, which was very far from his wish. His Table V. they would see showed that this was not the case. He sent his preparation to Mr. Stead, who obtained the theoretical result. Five other chemists obtained results considerably under 100 per cent. of the manganese present; it was those results they could not explain. The results were there, and all he wished was that chemists should check the method to be sure of the results they were obtaining. Mr. Gray had said that he obtained high results by the pyrophosphate method. That was quite possible, as lime or magnesia or baryta in solution must inevitably come down unless the manganese is first separated as sulphide. Personally he had tested against American chemists, and their results were not high, but low. Mr. Gray also pointed out that permanganate of potash was very undesirable as a reagent. There was great prejudice against permanganate of potash. He (Mr. Saniter) had used Volhard's method in testing manganese ores, and they had tested their permanganate solution months apart, and found it practically permanent when kept in the dark; he did not know whether it was so in the light. He was bound to say that for testing iron it was almost more delicate than bichromate, the pink colour being more definite than the green spot. He had stated in his paper that every precaution had been taken; he had used zinc chloride for the last three years. The burette was also carefully calibrated. It was not very difficult to arrive at the impurities. Dr. Kohn had asked if he had any direct evidence as to the volatilisation of manganese. In the experiment given he had estimated the amount of oxygen in excess of  $MnO$ , and found the whole deficiency was not due to the reduced quantity of oxygen in the precipitate. He had not tried the electrolytic method; it might be good, but he was afraid time was against them in commercial laboratories, whereas by Mr. Pattinson's method thoroughly accurate results could be turned out in a couple of hours. With regard to Dr. Hunter's question as to the value of the unit of manganese, he thought it was a good deal higher in ferromanganese than in manganese ores, but he had no figures at the moment. He did not think it was possible to draw two samples from a large cargo which would agree so closely as analyses on the same sample.

*Meeting held in the Victoria Hotel, on Friday,  
February 2nd, 1894.*

MR. IVAN LEVINSTEIN IN THE CHAIR.

The CHAIRMAN said that he desired to again call attention to a paper read by Mr. Carter Bell on the desirability of raising the flash point of mineral oils used for lighting purposes, for the reason of setting right some misapprehension that had taken hold of the minds of the Directors of the Ship Canal. The same subject had been considered at a subsequent meeting of the Chemical Section of the Manchester Chamber of Commerce, and reference was made to the probable danger that might arise to shipping and property belonging to the Ship Canal if large quantities of mineral oils of a low flash point were stored near the docks, and a resolution of the Section that it was desirable in the interests of public safety to raise the flash point to  $100^{\circ}$  was adopted by the Board of Directors of the Chamber. He was, however, very sorry to hear that the reference to the Ship Canal should have been misunderstood by the Directors of the canal, who were under the impression that the Section of the Chamber intended to agitate for only raising the flash point for oils arriving at the Manchester Docks. If that impression got into the heads of some people it would of course do considerable injury to the canal. With regard to the tolls and charges of the Ship Canal Company on chemicals, it had been suggested that a special sub-committee of our Section should be appointed to consider this question, but in his (the Chairman's) opinion, this was unnecessary, because this was already in the hands of the Chemical Section of the Chamber of Commerce, which was practically elected by them and represented the commercial interests of the chemical industries of the district. A full and exhaustive report on the subject had already been presented to the Chamber of Commerce and published in its monthly record, and they would find on reference to that report that the charges and tolls were in some instances heavier than the charges hitherto made, and this was principally due to the expense in carting to and from the docks. They had had an interview with Mr. Marshall Stevens, the manager of the canal, who went fully into their complaints, and if he did not succeed in overthrowing the report, he certainly enlightened them upon several points. There were reasons which he (the Chairman) was not prepared to state, that the freight from Liverpool to Manchester should not be reduced. In regard to the proposed meeting of merchants as to rates, sentiment would not bring business to the canal, but with equal rates he thought every loyal importer and exporter would support it.

## Manchester Section.

CHEMICAL THEATRE, OWENS COLLEGE.

*Chairman:* Ivan Levinstein.

*Vice-Chairman:* Edw. Schunck.

*Committee:*

G. H. Bailey.  
F. H. Bowman.  
R. F. Carpenter.  
G. E. Davis.  
C. Dreyfus.  
H. Grimshaw.

P. Hart.  
J. M. Irving.  
E. Knecht.  
W. H. Perkin, jun.  
Sir H. E. Roscoe, M.P.  
C. Truby.

*Hon. Local Secretary:*

J. Carter Bell,

Bank House, The Cliff, Higher Broughton, Manchester.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

SESSION 1893-94.

Friday, March 2nd:—

Mr. T. Clarkson, A.R.S.M. "A New Sampling Machine."  
Dr. F. H. Bowman, F.R.S.E. "Molecular Chemistry."

## NOTES ON THE "FERTILISERS AND FEEDING STUFFS ACT, 1893" (56 & 57 Vict. c. 56.), AND ON THE "FERTILISERS AND FEEDING STUFFS REGULATIONS, 1893."

BY J. CARTER BELL, A.R.S.M., ETC.

UNDER this Act, which came into force on the 1st January of this year, the farmer can bring a sample of manure or feeding stuff, together with the invoice of the goods, to a district analyst, and if the sample does not accord with the conditions of purchase as shown by the invoice, which has to state the percentages of soluble and insoluble phosphoric acid, nitrogen, and potash, if any present, then the seller is liable, under this new Act, to a penalty. All who have given any attention to the analyses of such substances as are covered by the Act, will be aware that various results may be obtained by different analysts, depending upon the methods of estimation. To illustrate this, I produce a circular issued by the Manchester Corporation some years since, in which it is stated "In consequence of damaging

statements having been circulated with reference to this manure ('made from human excrement, blood, bones, fish, &c.') the Health Committee have had samples drawn from stock by one of the professors of the Victoria University and by Mr. Carter Bell, and analysed by the under-mentioned chemists, with the following results<sup>1</sup>; then follows a table giving results obtained by seven analysts of repute—I abstract the salient points, analysts being represented by numbers:—

	Analysts.						
	1.	2.	3.	4.	5.	6.	7.
Organic matter*	29.68	36.14	33.03	29.24	31.53	38.52	35.54
Moisture.....	12.36	10.77	15.93	15.08	13.49	15.11	15.07
*N = ammonia.	2.81	2.99	2.82	2.61	2.50	2.84	2.73
Tribasic phosphate.....	3.66	6.39	4.32	4.72	4.80	4.60	5.16

No one can deny that the variations here are very great, and this time the usual excuse of bad sampling cannot be put forward, for the manure in the state of powder was well mixed in the laboratory and quickly put into bottles, corked and sealed in the presence of witnesses; in the estimation of the nitrogen we may say that the seven fairly agree, but in the other constituents the agreement is sadly wanting.

This Act is calculated to be of great benefit to the farmer and to the honest dealer. It is highly desirable that the effect should not be vitiated by conflicting analytical reports, hence I suggest that a committee, composed of manufacturers and analysts, be appointed at an early date, and the question of the analyses of manures and feeding stuffs be investigated by them. The question of sampling is an important one, but this is well covered by the "Regulations" issued by the Board of Agriculture. This society will be doing good work in suggesting that certain methods be employed for the estimation of the various constituents of the manures or feeding stuffs in question.

#### DISCUSSION.

Mr. T. TYRER said he was under the impression that in the matter referred to, sampling was regarded as of equal importance with analysis. He did not wish Mr. Bell to understand that he under-estimated the value of analysis. As a manufacturer he would prefer to have good sampling with an agreed method of analysis. There had never been any difficulty in determining what method should be adopted. The subject of manure analysis was brought before the London Chamber of Commerce and was thoroughly discussed by a committee and referred to the Manure Makers' Association, which had full powers. He believed he was correct in saying that the present Board of Agriculture had been in intimate connection with the Royal Agricultural Society of England, and they had assigned to Dr. Voelcker the duty of determining this question; therefore he could not see that any good result would be achieved by this Society taking the matter up again. The question of manure had become a matter for legislation, but he agreed from a manufacturer's point of view with having perfect methods of sampling. This difficulty of sampling had been pointed out by the Chairman, but dealers on one hand and sellers on the other often objected, inasmuch as the obtaining a fair sample was a matter of considerable difficulty. Wood pulp in hard masses certainly presented some difficulties. The decision almost unanimously arrived at by the Sections of the Society as to sampling had, in his opinion, equal force to-day.

It was proposed by Mr. Carter Bell that the Society form an analytical committee to investigate the various methods of analysing manures; this was seconded by Dr. Gerland, was put to the meeting, and carried *nem. con.*

#### NOTE ON THE ESTIMATION OF MOISTURE IN WOOD PULPS.

BY J. CARTER BELL, A.R.S.M., &c.

THE subject of Messrs. Readman's and Gemmell's paper before the Glasgow Section in December last, viz., The Estimation of Moisture in Wood Pulps, is of considerable interest and importance alike to the manufacturer and user as well as to the analyst to whom the estimation may be referred. With a view to a proper appreciation of the subject, and a satisfactory solution of the difficulties attending the analysis, I will first mention the method I adopt, and conclude with certain suggestions calculated to secure uniformity in the statement of results of analysis. I expose the sample to a temperature of 212° F., and so ascertain the absolute moisture present in the sample by difference. In stating my results, I allow a definite percentage of moisture. This I consider to be a much more scientific and satisfactory method than that of air-drying in a room at from 59° to 60° F., inasmuch as by the one process we arrive at a constant result, and by the second the result must be variable for many reasons; thus, the actual temperature of room, the varying amount of moisture in the atmosphere, the form and manner of exposure, i.e. in sheets or the layers separated, &c., &c. As an indication of this variation in results when air-dried, I give the following estimations taken from my note-book of wood pulp air-dried and also dried at 212° F.

October 1893. WOOD PULP exposed for 100 Hours to Temperature from 59° to 60° F.:—

Air-dried.		Dried at 212° F.	
	Per Cent.		Per Cent.
Moisture.....	59.3		69.3
	59.2		67.3
	59.4		67.0
	51.5		67.0
	55.8		66.5
	55.8		67.8
	61.8		65.5
Mean of 70 samples.)	58.4	Mean of 70 samples.)	66.77 moisture.
Air-dried pulp....)	41.6		
	100.0		

wood pulp, calculated to contain 10 per cent. of moisture would be 36.9 per cent. of air-dried pulp.

WOOD PULP, November 23rd, 1893, Air-dried for 60 Hours:—

	Per Cent.
100 grms. torn in pieces.....	Moisture 64
100 " " torn.....	63
50 " torn.....	65
100 " not torn.....	59
50 " ".....	61
50 " ".....	63

Mean of six samples "air-dried" for 60 hours... 62  
Percentage of moisture when dried at 212° F.... 66.6

In this case the manufacturer would be called upon to pay either for 37 tons or 38, whichever method had been used for estimating the moisture.

Three lots of 500 grms. each were weighed from the same sample. Nos. 1 and 2 were dried at 212° F., and the moisture was 56.1 and 56.4; these two dried lots were exposed in different rooms for 30 hours to temperatures



varying from 50° F. to 56° F.; the percentage of moisture was 52 in both cases, calculated on the original 500 grms.

The No. 3 500 grms. was exposed in a room for 24 hours to the same temperature as the above; the percentage of moisture was 48·2, or 51·8 of air-dried pulp.

According to experiments Nos. 1 and 2, the paper manufacturer would be called upon to pay for 48·6 tons of air-dried pulp containing 10 per cent. of water, but according to No. 3 it would be 51·8 tons.

One authority suggests that in estimating the moisture we first ascertain the absolute amount by drying at 212° F. and then allow 8 per cent.; others suggest 10 per cent., and a third 12 per cent. I repeat with emphasis that in my opinion we must adopt the method of estimation at 212° F., with subsequent allowance of a definite and agreed percentage of moisture. My suggestion is that this subject is of sufficient importance to justify the appointment of a "joint committee" composed of equal numbers of wood-pulp manufacturers or dealers, of paper manufacturers, and of analysts, all of recognised status, so as to ensure that the recommendations of such joint committee shall be regarded henceforth as the basis of contract. Let a committee so composed, through the agency of this Society, thoroughly investigate the whole subject and so secure for buyer and seller alike equal justice, and remove the onus from the analyst of stating results obtained by a method of estimation varying from that adopted by the opposing analyst.

## NOTES ON THE OXIDATION AND CORROSION OF IRON AND STEEL.

BY WILLIAM THOMSON, F.R.S.E.

I HAVE lately been experimenting to find the best protective coating for iron and steel work with special reference to structures which are situated in the neighbourhood of the sea, and exposed to the spray of the salt water. It required a number of experiments before a satisfactory method was obtained for making the tests. In comparing a number of different paints the thickness or weight of the coating must be considered as these vary to a large extent.

I obtained pieces of steel  $4 \times 1\frac{3}{4} \times \frac{1}{8}$  ins. drilled each with a hole in the top, these were weighed and painted with one coating of each of the paints and weighed again, then left for about a week exposed to the atmosphere, and again weighed, the difference between the two first weights gave the weight of the wet paint employed which was calculated for the square yard of surface whilst the first deducted from the third weight gave the weight of dry paint. The coatings varied between three quarters of an ounce for boiled linseed oil alone, to seven ounces for oxide of iron paint, tar 1·56, solution of pitch 1·24, red lead 6·24, &c.

These pieces of painted steel were then suspended and sprayed from time to time with a saline solution composed of a mixture of the chlorides of sodium and magnesium in about the proportions in which they would be found in sea-water, but this showed no satisfactory result, as there was no appearances shown on any of the samples to indicate rusting underneath the covering. I then immersed all the plates in a glass vessel containing sufficient saline solution to half immerse each and observed that the clear solution

### EFFECTS OF CAUSTIC SODA ON THE STRENGTH OF IRON AND STEEL WIRE $\frac{1}{8}$ TH INCH THICK.

	Solutions.						
	Before Treatment.	Cold.			Hot.		
		Water.	5 Per Cent. Caustic Soda.	12 Per Cent. Caustic Soda.	Water.	5 Per Cent. Caustic Soda.	12 Per Cent. Caustic Soda.
Steel wire:—							
Breaking strain in lbs.—							
1st piece.....	2,085	2,050	2,132	2,121	1,965	2,095	1,980
2nd „.....	..	..	2,010	1,960	..	2,030	2,020
Averages.....	2,085	2,050	2,071	2,040	1,965	2,062	2,000
*No. of twists in 6 inches—							
1st piece.....	13	10	14	12	2	12	12
2nd „.....	..	..	13	8	..	12	13
Averages.....	13	10	13·5	10	2	12	12½
Puddled iron wire:—							
Breaking strain in lbs.—							
1st piece.....	1,074	1,135	1,115	1,110	1,062	1,098	1,085
2nd piece.....	..	..	1,116	1,110	..	1,105	1,062
Averages.....	1,074	1,135	1,115½	1,110	1,062	1,101	1,073
*No. of twists in 6 inches—							
1st piece.....	12	12	11	8	10	7	9
2nd „.....	..	..	9	4	..	7	8
Averages.....	12	12	10	6	10	7	8½
Flat hoop steel, $\frac{1}{16}$ th inch wide by from $\frac{3}{16}$ ths to $\frac{1}{4}$ th of an inch thick:—							
Breaking strain in lbs.—							
1st piece.....	2,800	..	3,000	2,800	..	2,975	2,725
2nd „.....	3,200	..	3,350	3,500	..	3,200	2,825
3rd „.....	3,050	..	..	..	..	..	..
Averages.....	3,027	..	3,175	3,150	..	3,087	2,775

\* In this test, pieces of the wire 6 inches in length were twisted till they broke, and the number of twists required are here recorded.

after two or three days gradually began to become turbid, and in a few days more threw down a precipitate of the peroxide of iron. Some time later it could be observed that the iron underneath some of the coatings of paint was undergoing oxidation to a much greater extent than under others. It suggested itself to me that if each plate of iron were placed in a separate glass beaker with the saline solution the turbidity of the clear liquid would be some criterion as to the extent of the rusting underneath.

On making a second series of experiments in this way I observed that the production of a turbidity formed a very good criterion as to the protective power of certain paints, and it has enabled me to make a large number of experiments with different coatings which showed that oxide of iron paint, white lead, and the ordinary paints of commerce had comparatively little protective influence on the iron as compared with red lead, the latter showed no signs of turbidity in the saline solution whilst all the others had become turbid and deposited a considerable precipitate of ferric oxide.

The study of this subject was subsequently carried on in conjunction with Mr. Harry Smith, F.I.C., which has resulted in the discovery of some valuable applications for the prevention of corrosion, but as he has applied for a patent to cover his special claims I will leave him to describe these further experiments himself.

I took two small pieces of iron, attached them by copper wires, the one to a plate of copper of small size, and the other to a plate of zinc, these being allowed to stand in the saline solution. It was remarkable to observe how rapidly the iron in contact with the copper became corroded and how completely the iron in contact with the zinc was preserved from corrosion or oxidation, not only on the part in the solution itself but also on that which was not immersed. After a few weeks an electro deposit of zinc was formed on the immersed portion of the iron plate in contact with the zinc.

It suggested itself to me as probable that for the protection of bridges and towers it might be advisable to place a large ball of zinc in wet ground in metallic contact with the iron of the structure by means of wires which I believe would tend materially to prevent corrosion at comparatively small cost.

I have lately been considering the effects of riveting iron and steel on the one hand, and different kinds of steel on the other, in the manufacture of iron and steel boilers with a view to find whether the contact of these different materials would tend to cause corrosion in the one or in the other, by reason of the one acting as electro negative to the other and so inducing electric action resulting in oxidation and corrosion of one of the plates.

I lately came across a peculiar effect produced on the bowling hoop of a steel boiler which had been attached by iron rivets, the bowling hoop being deeply corroded at certain points, especially in the line along the outside ridge. I had cut from the steel bowling hoop a piece of steel  $\frac{1}{2}$  in. square by  $\frac{1}{8}$  in. long, and from one of the iron rivets I had a piece of similar size also cut; a hole was drilled through each near the top into which a copper wire was fixed; for comparison with these pieces I also obtained similar pieces in size and shape which I marked "C," this was a new steel rivet; "D" was a steel rivet closed by machine and held in the dies till nearly cold; "E" was a piece of new mild steel (unworked).

The piece of iron taken from the rivet in the boiler above mentioned was Low Moor iron which I marked "A," the piece cut from the steel bowling hoop I marked "B." The whole of these samples were pushed through a piece of cardboard, so that all could be immersed in any liquid at the same time. By merely touching the copper wires attached to each with the wires leading from a Thomson's tangent galvanometer, the direction, and to some extent the intensity, of the current of electricity produced (if any) could be ascertained by noticing to which side the spot of light was deflected and the rapidity with which the deflection took place. On looking at the annexed table it will be seen that "A," the piece from the iron rivet, and "B," the piece from the steel hoop when placed in sulphuric acid, and connected to the galvanometer the rivet was electro positive to the

steel at first and thus acted the part of the zinc or more easily corroded material, but within a few seconds afterwards the spot of light swung round in the opposite direction, showing that the iron rivet had become electro negative to the steel. The piece of iron rivet was then placed in contact with the piece from the new steel rivet "C," and the same effect took place, the iron rivet being electro-positive at first and electro-negative afterwards. Exactly the same thing took place when the iron rivet was placed in contact with the second steel rivet, closed by machine "D," but when placed in contact with a piece of "mild steel new unworked" "E," the iron remained from the first electro negative, the mild steel being electro positive.

The general results, as shown by the table given below, are that the three samples of steel "C," "D," and "E," all react strongly amongst themselves, and therefore it seems to be quite as likely that one steel plate may act as electro negative to another steel plate, as it is for a piece of malleable iron to act as electro-negative to a steel plate, although it may be taken as a rule that steel corrodes more readily than iron when placed under favourable conditions.

Combinations,	Sulphuric Acid (And 1 part to Water 9 parts).	Caustic Potash Solution. Sp. gr. 1.311.	Chloride of Ammonium Solution. Sp. gr. 1.033.
A & B	+ A at first, + B afterwards.	+ B at first, the current rapidly going down to zero.	+ A very strongly on continued action, + B very weak, almost nil.
A & C	+ A at first, + C afterwards.	+ C very strongly	+ A very strongly, afterwards the current decreased to zero, but no reversal.
A & D	+ A at first, + D afterwards.	+ D " "	+ A at first weakly, afterwards + D weakly.
A & E	+ E	+ E " "	+ A strongly at first, gradually becoming very weak, but showed no reversal.
B & C	+ C	+ C at first fairly strongly, then + B fairly strongly.	+ C strong at first, afterwards very weak.
B & D	+ B slightly, + D afterwards.	+ B at first fairly strong, then reversing to + D, reversing again to + B rather strongly.	+ B strong at first, afterwards + D weakly.
B & E	+ B momentarily, then + E afterwards.	+ B momentarily, then + E very weakly.	+ B strong at first, afterwards weak, but no reversal.
C & D	+ C momentarily, then + D afterwards.	+ C very strongly	+ C " "
C & E	+ C strongly at first, afterwards becoming extremely weak. Sometimes on one side and sometimes on the other.	+ E fairly strongly.	+ E " "
D & E		+ E very strongly	+ D " "

Copper with all the samples was "+" (electro-positive) in caustic potash solution, and "-" (electro-negative) in sulphuric acid and chloride of ammonium solutions.

The + sign corresponds to the electro-positive, i.e., the zinc element which becomes corroded, whilst the sign - corresponds to the copper element which does not corrode.

Much interesting work in this direction has been made by Mr. Thomas Andrews, Asso. M.I.C.E., F.C.S., &c.\* He shows that in some combinations of steel of different

\* On "the Relative Electro-Chemical Positions of Wrought Iron, Steels, Cast Metal, &c. in Sea-Water and other Solutions." Read before the Royal Society of Edinburgh, and also one by the same author on "Galvanic Action between Wrought Iron, Cast Metals, and various Steels during Long Exposure in Sea-Water." Read before the Institution of Civil Engineers, Vol. LXXXVII, Session 1883-84, Part III., and a third by the same author, on "Corrosion of Metals during Long Exposure in Sea-Water." Proceedings of the Institution of Civil Engineers, Vol. LXXXII, Session 1884-85, Part IV.

kinds with each other and with iron that the one element after being electro-positive gradually becomes electro-negative, and this change will take place many times during long contact with each other. In other cases the relative galvanic positions of the two pieces of metal remaining constantly the same. Under such conditions it seems an advisable thing that in making boilers some use should be made of this simple means of determining whether the one metal intended for use in the boiler would be likely to act strongly upon another when both are placed in galvanic contact with each other in a saline solution.

I have lately been investigating a curious case, in which a number of steel boilers were completely destroyed by using them for producing steam, and at the same time for evaporating solutions of caustic soda from 5 to 12 per cent. The result of the use of the boilers in this manner was, in about two years, to crack many of the plates along the line of the rivets, but not between the rivet holes, and in many cases the rivet heads were broken off. I was informed that this was one of the peculiar properties of caustic soda upon iron or steel, that "it took the nature out of the steel, and caused them to become brittle."

I made a number of experiments to find whether caustic soda had any effect whatever upon iron and steel, by taking—

A. Four pieces of puddled iron and four pieces of steel wire, and placing them in a five per cent. solution of caustic soda (made from ammonia soda ash) contained in iron tubes closed by a screw cap.

B. Four pieces of each as above, placed in a 12 per cent. solution of caustic soda as above described.

C. Two pieces of each wire placed in distilled water in an iron tube similarly arranged.

D. Four pieces of hoop steel placed in 5 and 12 per cent. solutions respectively of caustic soda as above mentioned.

In all these experiments one set of each was kept at the boiling point of water constantly for 84 days, and the other set kept in the cold, the whole being immersed for 195 days (about 6½ months).

The breaking strains of the pieces of wire in samples untreated in comparison with those which were thus treated were kindly determined for me by Mr. John Thewlis Johnson of Manchester, and the breaking strains of the hoop-steel by Mr. Alderman W. H. Bailey of Salford.

The results as a whole show that caustic soda appears either hot or cold, to have little or no chemical or damaging action on the iron, which is what one would have expected. I had in my possession till lately some pieces of bright iron which had been put by my late partner, Dr. F. Crace-Calvert, in a solution of carbonate of soda and left for upwards of 30 years, and they were as bright at the end of that period as they were when first immersed. I am of opinion that the damage to the steel boilers is a purely mechanical one, caused possibly by the caustic getting under the rivet heads and between the plates, crystallising and thus breaking them by expansion underneath.

#### DISCUSSION.

The CHAIRMAN wished to know what in the opinion of the author of the paper was the best paint to be used for the protection of iron?

Mr. THOMSON replied that red lead was usually employed by the Admiralty for painting iron ships, and might be considered the best, notwithstanding that it very often caused poisoning to the men engaged in cleaning the interior of ships.

Mr. PETER HART.—Some boilers used for boiling caustic soda solutions only gave way near the furnaces, and the bolt heads broke off. This showed that it was purely a mechanical action, and not the result of corrosion from the action of caustic soda.

## Dorsetshire Section.

Chairman: A. Smithells.

Vice-Chairman: T. Fairley.

#### Committee:

J. E. Bedford.	C. Rawson.
F. Branson.	F. W. Richardson.
J. B. Cohen.	Jas. Sharp.
J. J. Hummel.	G. W. Slatter.
J. Lewkowitsch.	G. Ward.
W. McD. Mackey.	T. B. Wilkinson.

Hon. Local Secretary:

H. R. Procter, Yorkshire College, Leeds.

Notices of Papers and Communications should be addressed to the Hon. Local Secretary.

#### SESSION 1893-94.

March 5th:—

Messrs. Liechti and Hummel. "The Mordanting of Wool with Chromium." Part III.  
Nomination of Officers for next Session.

April 2nd:—

Messrs. Hummel and Perkin. "The Tinctorial Properties of some Indian Dyestuffs." Part I.  
Annual Meeting for Election of Officers.

May 7th.—Mr. H. R. Procter. "The Qualitative Recognition of Tanning Materials."

Meeting held at the Queen's Hotel, Leeds, on Monday, February 5th, 1894.

MR. THOS. FAIRLEY IN THE CHAIR.

## SUBSTANTIVE DYES AND SUBSTANTIVE DYEING. A CONTRIBUTION TO THE THEORY OF DYEING.

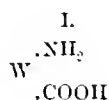
BY CARL OTTO WEBER, PH.D., F.C.S.

THE theory of dyeing has so far scarcely exercised any direct influence upon the art of dyeing, none of the exceedingly numerous methods for dyeing and printing having been evolved from a theory of dyeing in a similar manner, as, for instance, the manufacture of triphenylmethane colouring matters from the well-known series of theoretical researches on their constitution. At present two essentially different theories of dyeing are opposing each other, one of which considers dyeing as a chemical process, while the other regards it as a merely physical phenomenon. Witt's "solution theory" is no doubt the most notable representative of the last named class. It appears that the adherents of these theories never seriously attempted a rational compromise between their divergent opinions, one always striving to prove the other unsatisfactory or untenable. This of course might be justified if proof had ever been furnished that the whole of the dyeing phenomena, as observed in the dyeing of the various fibres with dyes of different classes, belong to one order of chemical or physical phenomena only. But indeed we know that the behaviour of each of the different fibres towards the same dye shows great differences, and so does the behaviour of one and the same fibre towards dyes of the various classes indicate very considerable variations. That there exists an undeniable connection between these differences and the chemical nature of these textile materials and dyes is an old observation. Especially well established is the fact that a coloured substance, in order to be applicable as a textile dye, must of necessity possess the properties and constitution of either an acid or a base.\* Thus, indigo

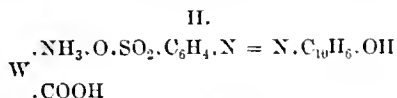
\* This definition of course does not and is not meant to cover also the pigment dyes, amongst which not only the well-known dye pigments, but also aniline black and indigo must be classed.

cannot be called a dye, but should be described as a pigment colour, while on the other hand indigo-caruine is a true dye. There exists no exception to the above rule, which certainly seems to confirm the chemical theory of dyeing. But we are forced to the conclusion that the dyeing processes must be divided into at least two different classes, according to whether the dyes are directly assimilated by the fibre (substantive dyeing), or whether they require the intervention of a mordant, or are formed immediately in and upon the fibre (adjective dyeing). The first case forms the subject of this investigation, together with the relations between the chemical nature of fibre and dye, upon which substantive dyeing depends. The second case (adjective dyeing) will not occupy our time here. Dyeing in this case is a process practically independent of the fibre,\* in so far as the latter assumes no chemical rôle in the fixing or formation of the dye or pigment, but merely acts as a carrier for the latter. These dyes therefore either mechanically adhere to the fibre, or they occur in the cellular cavities of the latter in the form of insoluble precipitates. For this reason, all dyes of this class are very liable to show the defect known as "rubbing off," and indeed they are never without it. Indigo, aniline black, Turkey red, and all mordant dyes on cotton, also the azo dyes produced upon the fibre are pigment dyes of this class, just like chrome yellow, Prussian blue, or iron buff. There are scarcely two opinions possible with regard to this kind of dyeing and dyes.

Of the various textile fibres, wool has no doubt the greatest affinity to colouring matters of all kinds, and is capable of decomposing the basic dyes into free acid and colour-base which combines with the wool, also of combining directly with the colour acids of sulphonated dyes. In all these cases the wool is dyed in shades identical with those of the salts, or rather the lakes of these dyes. This property of wool to combine with colour-bases as well as with colour acids, has received its full explanation by the valuable investigations of Knecht,† who most conclusively demonstrated this function to be due to the amido-acid nature of the wool-fibre, particularly to lanugine acid. It is a matter of little importance for the discussion of this subject, whether the wool fibre is a homogeneous substance or not, or whether lanugine acid pre-exists on the fibre or is a decomposition product of the latter, which forms in the course of the dyeing process. The fact that wool is capable of forming lakes with colour bases and colour acids in the manner of an amido acid is well established and we can therefore express the chemical nature of the wool fibre by the formula—



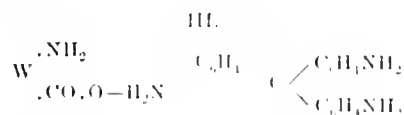
in which W stands for an unknown radicle, which may or may not be homogeneous in itself and which to us is of no concern beyond being the carrier of an amido group and a carboxyl group, i.e., of basic and acid properties respectively. Therefore while this formula only in the widest sense of the term could be called an empirical formula, it contains on the other hand no hypothetical assumption, the presence of amido and carboxyl groups in the wool fibre being a matter beyond dispute. This elementary formula being granted, wool dyed with naphthol orange would then be—



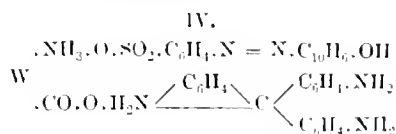
and likewise wool dyed with magenta.

\* Except in the case of the sulphonated or carboxylated, and mordant-dyeing wool dyes, which occupy a very interesting intermediate position between substantive and adjective dyes.

† These investigations have evidently been suggested by certain remarks of Prof. Hummel (Journ. Soc. Dyers and Col., Vol. 1, pp. 208–210), who appears to have clearly recognised the nature of the process of dyeing wool long before Knecht undertook his researches.



The process of the assimilation of these and all analogous colouring matters by the wool fibre is a purely chemical reaction, which is in itself sufficiently intelligible without Witt's theory of rigid solutions. What takes place in these dyeing processes is simply the formation of lakes between the amido or the sulphonic group of the wool and the respectively antagonistic sulphone or amido group of the acid or basic colouring matters. The second of our above formulae expresses the fact, that in dyeing wool with a sulphonated colouring matter, the amido group of the wool combines with the sulphonic group of the dye, while the carboxyl group of the wool remains unaffected. The third of our formulae represents the process of dyeing wool with magenta as lake formation between the carboxyl group of the wool, and one of the amido groups of magenta. Consequently we should expect that the free carboxyl group of wool dyed with some acid colouring matter, would still be capable of combining with basic dyes in the same manner and degree as undyed wool. Such indeed is the case. A skein of wool dyed in the presence of a large excess of scarlet R was carefully washed after dyeing. On immersion of this skein in a bath of magenta, side by side with a white skein of the same weight, which was treated in a magenta bath of the same strength, it was found that both skeins assimilated exactly the same quantity of magenta. Considering, however, that the solutions of all acid colouring matters precipitate the solutions of all basic colouring matters, lakes being formed, which must be described as salts of colour bases with colour acids, it might be suggested that the magenta in the above dyeing test did not combine with the carboxyl group of the wool, but formed a lake with the acid dye upon the fibre, in which case the wool would simply be dyed with a kind of pigment, a magenta-scarlet lake. But lakes of this description are, without exception, easily soluble in alcohol. However, in treating with alcohol the hank dyed with scarlet and magenta, only a very small quantity of magenta, but not the faintest trace of scarlet is dissolved. This is an undisputable proof that upon such skein scarlet and magenta occur quite independently of each other; in other words, wool dyed in this manner is a compound lake of the formula—



The disciples of the "solution hypothesis" might observe that the negative result of the alcohol test was not the necessary result of a chemical combination of the above description, but simply the consequence of the greater solubility of those dyes in the substance of the wool fibre than in alcohol, for this hypothesis certainly has the advantage of offering a cut and dried answer for every emergency of this kind. Now, assuming for the nonce this hypothesis to be right, then we should have dissolved in the wool fibre magenta side by side with scarlet, so that just in this case under all circumstances the above-mentioned magenta-scarlet would of necessity be formed, but this is exactly what is disproved by the negative alcohol test. Witt's hypothesis is very difficult to refute, because it is almost uncontrollable, and if we attempt to disprove it, we must bear in mind that it assumes the colouring matters to be in a state of solution in the fibre, i.e., in the free state. As soon as any chemical action between fibre and dye is allowed, the hypothesis becomes altogether superfluous. I shall show later on that we possess the means for the experimental determination of this matter.

The second objection raised against the chemical theory of dyeing contends that no chemical combination between the wool fibre and the dyes did take place in dyeing, as the



of very much the same order as the formation of a salt from acid and base. Witt's solution theory applied to substantive wool dyeing becomes involved at every step in insuperable difficulties.

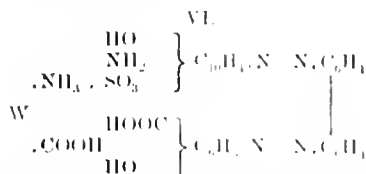
Especially interesting are the phenomena of substantive dyeing on cotton. This fibre, or rather cellulose, is a very indifferent substance as compared with wool or silk. This indifference is even more pronounced in its behaviour to dyestuffs, as compared with substances producing deeper chemical changes. The aldehydic nature of cellulose, and the absence in it of lake-forming acid or basic groups, goes a long way towards the explanation of this indifference. As soon as we fix upon the cotton a carboxyl group (tannic acid) or an amido group (by Vignon's process), the fibre behaves to basic and acid dyes respectively, like wool. On the other hand, it is well known that without such a preparation, cotton is capable of assimilating direct many basic dyes and also the numerous so-called benzidine dyes. This is particularly surprising with regard to the last named class of dyestuffs, which, from a chemical point of view, are of the same type as the familiar wool scarlets and croceines, which altogether lack the capability of dyeing cotton direct, while most of the benzidine dyes are also wool dyes of high excellence.

But substantive dyeing on cotton, even with the basic dyes, appears very puzzling, there being no lake-forming group whatever in cotton. It is true that our knowledge of the chemical constitution of cotton is very deficient as yet; but still we know for certain that any salt-forming energy the hydroxyl groups of cellulose might possess, is very small, as is readily inferred from the fact that no free acid is ever observed in dye baths of basic dyes even when they are completely exhausted by the cotton.\* This observation therefore goes to prove that cotton, unlike wool, which only takes up the free colour-bases, assimilates the complete colour salts. But, nevertheless, it will have to be taken into consideration that oxycellulose shows a very considerably increased affinity for basic dyes, so that strongly chemicked cotton dyes much darker shades with these dyes than merely soured cotton, and this gains additional significance by the fact that oxycellulose is of a much more pronounced acid nature than ordinary cellulose. But in dyeing oxycellulose with basic colours we again fail to discover free acid in the dye baths, and it appears therefore as if the phenomenon of substantive dyeing on cotton with basic colours could not be understood as a chemical process, and would therefore justify the introduction into the theory of dyeing of a new principle, such as Witt's solution hypothesis.

Adjective dyeing (mordant dyeing) is admitted by every one to be a purely chemical process, that substantive wool dyeing is accomplished by virtue of chemical processes of an identical order (lake formation) has been proved above. On the other hand we shall see presently that from cogent chemical reasons we must in other cases admit the existence of the dye in the fibre in a state of solution. It is only to be expected that occasionally two of these processes concur in producing the dyeing result. In this case the dyeing operation is really a very complex process, an example of which may prove of interest.

The dyeing of alizarin upon chromed wool is a typical case of adjective dyeing. The dyeing of diamine fast red upon wool in an acid bath is an equally typical case of substantive wool dyeing, consisting, according to what I have shown above, in the formation of a lake between the amido group of the wool and the sulphonic group of the dye.

\* In relation to this a recent observation by Knecht (Journ. Soc. Dyers and Col. 1893, 114) is of interest. He noticed that in placing a drop of a solution of magenta upon bibulous paper, it expands rapidly into a circular stain, and subsequently a halo of apparently pure water forms round this stain, and this water is of a distinctly acid reaction. Evidently a dissociation of the magenta into hydrochloric acid, and a basic salt of magenta has taken place. Whether this dissociation is caused by the difference in the ratio of diffusion of the components of magenta, or has to be considered as the action of the weak acid cellulose of the paper cannot be decided yet; but it is worth pointing out that this observation stands on a par with the phenomena accompanying the formation of dissociation le's (this Journ. 1891, 398) from basic dyes and china clay.



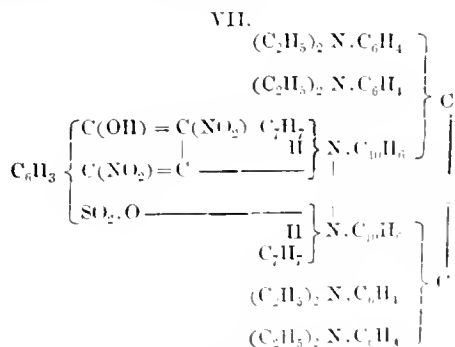
Kertesz showed that by treating wool thus dyed with chromium fluoride, the fastness of the dye is increased in a most remarkable degree. This action of chromium fluoride is due to the formation of a chromium lake in the oxy-carboxyl group—



of the dye, which, in this respect, behaves exactly like the ortho-dihydroxyl group of the alizarin dyes. Dyes not containing one of these two lake-forming groups are quite indifferent to chromium fluoride, and this is the reason why the practical results obtained with this chromium salt led to such a difference of opinion as to its value. When indiscriminately applied to a number of wool dyes selected at random, the results obtained with this salt are sure to prove confusing, if not discouraging. But whenever a wool-dye contains besides the sulpho group, either of the two above-mentioned lake-forming groups, subsequent treatment with chromium fluoride is certain to act beneficially. Our above example therefore indicates a dual dyeing process which is at the same time both a substantive and an adjective dyeing process. Similar combinations can occur between adjective dyeing and substantive dyeing respectively, and solution. It is scarcely necessary to point out that combinations between substantive dyeing and dyeing by solution, are only possible in such cases where the chemical energy of the fibre, which is the primary condition of substantive dyeing, is extremely weak. This, however, is exactly what we observe in the case of cotton being the material to be dyed. This fibre is necessarily devoid of basic properties, and its acid properties are of the weakest description. The temptation under these conditions is, no doubt, very great to look upon this indifferent behaviour of the cotton fibre as *prima facie* evidence for Witt's solution theory. Nevertheless, I am of opinion that substantive dyeing upon cotton with basic dyes can be satisfactorily explained as a chemical process of a similar kind as the process of substantive wool dyeing.

The fact that, as above stated, cotton does not fix the base only of the basic dyes, but takes up the whole of the colour salt, appears strongly in favour of Witt's hypothesis, and this important fact that cotton, substantively dyed with basic dyes, contains the latter still in combination with their respective acids can easily be demonstrated.

Rawson\* showed some years ago that the strength of naphthol yellow can be most accurately determined by titration with Night blue. This means, in other words, that between definite proportions of these two dyes a chemical reaction takes place, and this reaction is simply lake-formation between the Naphthol-yellow acid and the Night blue base, the lake formed being—



\* Journ. Soc. Dyers and Col. 1888, 82.



On the other hand, naphthol yellow does not decompose an already formed night blue lake, just as barium chloride is incapable of forming a lake with naphthol-scarlet dyed on wool. This loss of reactive energy of both the basic and acid constituents of lakes is a very remarkable fact. In some cases it goes so far that the lake-forming metallic oxide can no longer be detected in these lakes by means of the ordinary reagents. The behaviour of the alumina in alumina alizarate and of the iron in naphthol green are typical instances of this kind.

Now, if we dye a skein of cotton with night blue, wash it, and pass it then through a solution of naphthol yellow S, the skein at once assumes a dark green colour and the dye is remarkably fast. If we repeat this experiment with a skein of cotton mordanted with tannic acid, we find that in this case the treatment with naphthol yellow produces no alteration. It is evident therefore that in the first case the dye, without any chemical change, migrate from the dye-bath into the cotton fibre, and all the reactions the aqueous solution of this dye is capable of, are consequently also obtainable with the dye on the cotton fibre. The tannin-mordanted skein, however, contains the night blue in the form of its tannin lake, and this accounts sufficiently for its indifference towards naphthol yellow. This leaves no doubt that whatever is the cause of night blue, like many other basic dyestuffs, dyeing cotton substantively, no lake formation can have occurred in the salt-forming amido group of the dye.

These facts with regard to substantive dyeing with basic dyes present all the requisite appearances for the consideration of this dyeing process as a solution phenomenon in the sense of Witt's hypothesis. But however weighty in this respect the above stated facts might appear, they are after all only a one-sided view of the subject, in so far as we have only taken notice of the behaviour of the dyes in this substantive dyeing process, without considering the behavior of the cotton fibre. The latter, although being a very indifferent chemical substance, is proved, through the recent observations of Cross, Bevan, and Beadle\* to be a rather stronger acid than it was generally supposed to be. But apart from this observation the capability of cotton to form saltlike compounds with the alkalis and several metallic oxides, especially plumbic oxide, is a well established fact. In dyeing scoured cops with basic dyes and cutting them in sections at various stages of the dyeing operation, we find that the strength of the dye is not decreasing from the outside of the cops towards the centre, as should be the case if the dyeing was due to a solution of the dye in the fibre. On the contrary we find the half-dyed cops to consist of a perfectly white centre surrounded by a sharply defined annular zone, which is quite evenly dyed. Exactly the same is observed in the dyeing of tannin mordanted cops. We know that this must be due to the definite capacity of combination tannic acid has for every basic dye, but at the same time we must necessarily assume the existence of similar relations between the cotton fibre or substance and the basic dyes. In this manner only can we explain the progressing by leaps of the dyed zone in the cops, instead of the gradual decrease of the depth of shade from the outside to the inside of the cops. We shall now have to enquire into the cause of this phenomenon.

Some time ago I showed that all normal tannin lakes are di-acid salts of the colour bases, and this fact seems to me to contain the solution of the above problem. The acid nature of the cotton substance is indisputable, but it is too feeble to abstract, like wool, from the basic dyes the colourbase with liberation of the acid. It is, however, quite strong enough to fix the whole of the basic colour salt, forming with it a di-acid lake by combining with one of the free amido-groups of the dye. It can be readily inferred that such a lake must be very unstable, especially against alkalis, and indeed we find generally that substantive basic dyes on cotton are very sensitive to soap.\* This view of the nature of the substantive dyeing process of cotton is thoroughly confirmed by the much increased affinity to

basic dyes shown by oxycellulose, an increase which, while being a necessary consequence if lake formation is the fundamental principle of this case of substantive dyeing, is utterly unaccountable from the point of view of Witt's solution theory.

Entirely different are the circumstances in regard to direct dyeing with the so-called benzidine dyes. According to their constitution these dyes must be counted amongst the acid dyestuffs. Indeed, most of the benzidine dyes are much better wool dyes than cotton dyes. The quite unexpected property of these colouring matters to dye cotton direct in either a neutral or alkaline bath, naturally called forth various attempts towards the explanation of the surprising fact. Mohlau\* thought to explain it from his observation that cotton could be "mordanted" with benzidine-chloride, and by subsequently diazotising this mordant and developing it in naphthionate, congo red was produced upon the fibre. From this he thinks himself justified to conclude that it is the paradiamido-diphenyl group of the benzidine dyes which is the cause of their seeming affinity to cotton. If such an affinity really exists, which by the way I absolutely deny, it can only be intelligible if it consists in the affinity of the amido groups of diamido-diphenyl to the weakly acid cellulose molecule. But in converting diamidodiphenyl into benzidine dyes, these amido groups are transformed into diazo groups, and their basic properties become absolutely extinct the same moment the actual dyestuff is formed. Consequently the cause of the alleged affinity between diamidodiphenyl and cotton, cannot by any means be the cause of the affinity existing between benzidine dyes and cotton. Even if we are prepared to concede the existence of some occult attraction intervening between the diphenyl residue and cotton, we only gain a very questionable advantage, explaining the mysterious affinity of the diphenyl dyes to the cotton fibre by the equally mysterious affinity of the diphenyl group. The discovery of numerous direct dyes containing no diphenyl group, deprived this so-called explanation of its last support.

Schultz thought the desired explanation might be found in the fact that the solutions of the benzidine dyes really are exceedingly fine suspensions only, which on prolonged standing deposit practically the whole of the apparently dissolved colouring matters. Schultz therefore evidently considers the dyeing of cotton with the benzidine dyes a mechanical process of the same order as the bleaching of textile fabrics with ultramarine blue suspended in water. It certainly cannot be denied that amongst the benzidine dyes a remarkably great number of difficultly soluble products occurs, but on the other hand there is at present no scarcity of very readily soluble dyes of this class. Moreover, the fastness of the benzidine dyes against rubbing is infinitely greater, as a dyeing process of the kind proposed by Schultz would yield. This author's explanation is further scarcely compatible with the fact that all these dyes are applied in boiling dye-baths, while for a dyeing process of that kind cold baths should answer quite as well. It is therefore evident that Schultz's explanation is contrary to the most essential facts concerning the application of these dyes.

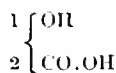
That, however, cotton dyed with benzidine dyes contains the dye in its original form, i.e., as a colour salt, is evident from the fact that the sensitiveness of these dyes against acids is exactly the same, whether we have the dye fixed upon or in the cotton fibre, or whether we have it in aqueous solution. Remembering further that cotton is always dyed in alkaline solutions of these dyes, it is also clear that no decomposition of the colour salt into free dye-acid and alkali can take place, for the simple reason that cotton does not possess, like wool, amido groups capable of combining with free dye-acids with formation of a lake. But indeed it can be proved beyond doubt that benzidine dyes dyed on cotton exist in the latter in an absolutely unaltered condition. If we treat a skein of cotton dyed with one of these dyes in the solution of a barium salt, the colouring matter contained in the fibre is at once converted

\* Certain basic dyes, however, as, for instance, indazine (Casella), dyed on cotton without a mordant, seem, contrary to the above statement, to stand soaping perfectly well. But this is in every case due to the more or less complete insolubility of the free bases and oleates of these dyes in aqueous soap solutions.

\* Ber. 1888, 2014.



into a barium lake. Numerous inorganic salts can be used with the same result in place of barium chloride. Thus we see that these colouring matters in becoming fixed upon the fibre do not lose their characteristic lake-forming properties, and this fact implies that the assimilation of these dyes by the cotton fibre is certainly not due to some process of lake-formation, as in this case subsequent treatment with barium-chloride could have no effect. Still more striking is the behaviour of cotton dyed with these direct colours against solutions of the basic dyes, the latter being fixed by the former exactly as if the cotton had been mordanted with tannic acid. This fact, which was first observed by Messrs. Bayer and Co., of Elberfeld, who considered it a specific property of the benzidine dyes, has never been properly understood, and was the cause of a great confusion of ideas, which finds characteristic expression in the following passage from Schultz's *Chemistry of the coal-tar colours* (II. German edition, Vol. II., 341). This author says that, "the affinity of the benzidine dyes to the cotton fibre is so great that dyes of this group behave upon the cotton like mordants so that basic dyes can be dyed upon such cotton without previously mordanting it." It is difficult to see what the capability of the benzidine dyes to form lakes with the basic dyes has to do with the supposed affinity of the former to the cotton fibre. We might as well say that the affinity of tannic acid to the cotton fibre goes so far that cotton mordanted with tannic acid can be dyed with the basic dyes. As a matter of fact the capability of the benzidine dyes to form lakes with metallic salts or basic dyes is in no way connected with their property to dye cotton direct, but is merely a function of the dye-acid contained in these dyes, which by virtue of its acid nature acts exactly like tannin, the specific function of which is also purely that of an acid forming insoluble salts with the basic dyes. Moreover, that behaviour of the benzidine dyes is by no means characteristic of them. Any acid dye, no matter whether it be applicable to cotton or not, will show the same capability to form lakes with the basic dyes, as long as there are any free sulpho groups, or sulpho groups in combination with alkali only. A case of this kind we have already met with in the night blue-naphthol yellow lake. The behaviour of cotton dyed with benzidine dyes therefore only proves that these dyes exist in the fibre in the free state; in other words, as in a solution. Lake-formation between cotton and benzidine dye is out of the question, and this also explains the rather low average of fastness to light which these dyes show on cotton, while many of them on wool produce shades of an extraordinary degree of fastness, not only to light, but also to milling. It is an unquestionable fact that the fastness of a dye shows considerable variations according to the method of its application, and we find invariably that the fastest shades are obtained by that method which best provides for the specific lake-forming tendencies of every dye. Diamine fast red on cotton is very unsatisfactory, the dye having no chance whatever to form a lake. Dyed on wool in the manner of an acid dye, lake-formation in the sulpho-group takes place, and we obtain a shade of very considerable fastness. By subsequently treating the dye with chromium fluoride, a chromium-lake is formed in the residual group



of the dye, and the result is a shade of almost unexampled fastness.

In the above we have, by means of chemical reasons, established the fact that the benzidine dyes exist in the cotton fibre in the form of their original colour salts, and not in the form of lakes. For this reason these dyes react upon the fibre with all those substances, and in exactly the same manner as their solutions in water. The next question is therefore as to the manner in which these dyes are fixed by the cotton fibre. It might appear now as if an answer to this question might readily be found in Witt's solution hypothesis, this hypothesis being in perfect accord with the above discussed facts. On the other hand, however, we

must not forget that Witt's hypothesis goes beyond the mere adoption of the physical principle of solution for the explanation of the dyeing process in question, but makes the further assumption that the solvent in this dyeing process by solution is the substance of the cotton fibre, cellulose, itself. And this is the point where I hold that Witt's solution hypothesis is vulnerable. This hypothesis is an ingenious analogy, but, like all analogies, it also is an incentive to superficiality, and its uncontrollable elasticity renders it almost unfit for scientific purposes.

All benzidine dyes are applied in dye baths containing besides the colouring matter always certain indifferent salts, generally also alkalis and soap. The presence of these salts reduces the solubility of the dye, or, as we may put it, imparts to the dye bath the properties of a more concentrated solution of the dye, as is expressed by the ratio of dye and water present. A higher concentration of the dye bath, however, is tantamount to a higher osmotic pressure of the dissolved dye and the well known practical advantages of the addition of these salts, only confirm therefore the above conclusion, deduced from chemical facts that the dyeing of cotton with these dyes undoubtedly is a solution phenomenon. This leaves only the question after the solvent, into which the dye diffuses, to be decided. Witt of course declares that osmotic migration of the dye into the cellulose itself takes place, with formation of what Knecht first termed a "rigid solution." This I most decidedly dispute, and maintain that the dye simply diffuses in the water contained in the cotton, and that the dyeing process comes to a standstill as soon as the osmotic pressures in and outside the fibre have attained to a state of equilibrium.

At the outset, Witt's hypothesis is beset with numerous improbabilities, as will be readily seen, if we apply it to the dyeing of a developed diamine black upon cotton. In the first instance, according to Witt, diamine black would form a "rigid solution" with the cellulose. This being so, the subsequent diazotizing and developing baths can only be of any use, if cellulose is also capable to dissolve nitrous acid, and all the innumerable phenols, naphthols, aromatic bases, and their derivatives. Moreover, if we take into account that by treatment with the salts of barium, zinc, nickel, cobalt, iron and copper, benzidine dyes dyed on cotton can be quantitatively converted into their respective lakes, we must needs assume that all these salts are likewise soluble in cellulose. As a matter of fact, we would have to conclude that whatever substance is soluble in water, appears to be soluble also in cellulose. Witt himself will probably admit that this necessary consequence of his theory is nothing short of a monstrosity. It can, however, be shown experimentally that it is the water contained in the cellular cavities of the cotton and not the cellulose itself which acts as the solvent for the benzidine dyes. It is obvious that if Witt's explanation is correct, cotton or cellulose which is free from water and incapable to absorb any, should take up the benzidine dyes even easier than ordinary cotton.

Dinitrocellulose is manufactured at present in such perfection that it cannot be distinguished from loose cotton, and even the structural differences which are observed under the microscope are very slight indeed. This dinitrocellulose can be dyed with benzidine dyes exactly like ordinary cotton. By adding dinitrocellulose to acetone, solution ensues immediately and by pouring the solution on glass plates and allowing the solvent to evaporate, thin films of dinitrocellulose are obtained. Of course every trace of the cellular build of the cotton has disappeared and with it the water inclosed in these cells. In trying to dye these structureless films of dinitrocellulose with these benzidine dyes, we find that even after hours' boiling these films are as white as ever and indeed never take up the faintest trace of these dyes. This shows that in one and the same dyebath dinitrocotton is dyed to a bright shade, while dinitrocellulose remains colourless. The former possesses cellular cavities, either containing water or at any rate capable of being filled with water, dinitrocellulose of course does not contain any water, nor is it capable to

\* Except for the most delicate shades.

absorb water or to retain it. This leaves no doubt that the dye is located in the cellular cavities of the fibre and not in the substance of the latter. Exactly the same of course holds good for cotton itself.

From this I conclude that in this dyeing process the cellular cavities of the cotton are filled with the hot, more or less concentrated solution of the dye, and on cooling the skein, the dissolved colouring matter separates out more or less completely as a fine coating of colouring matter upon the walls of those cellular cavities. After drying the skein so dyed, its residual moisture depends entirely upon atmospheric conditions, but represents always a fraction only of the water required to keep in solution the colour deposited within the fibre. Even then, it appears, is the presence of water essential for the brilliancy of the dye. This is clearly seen if we deprive such a dyed skein completely of its moisture, either by subjecting it to heat, or by immersing it for a few minutes in absolute alcohol. The dye assumes in every case a dull or even dirty, and always surprisingly weak shade. Not infrequently can it be observed that the dye so treated altogether changes its shade, which is due to the fact that several benzidine dyes possess a different shade in the hydrated, than in the anhydrous state. It is a well-known fact, which has frightened many a dyer, that cotton dyed with diamine black R O assumes a dull maroon shade on the hot dyeing cylinders, but acquires its blue-black shade again as soon as the fibre has recovered its natural amount of moisture, on exposure to the air.

A further confirmation of the conclusions above arrived at is to be found in the microscopical aspect of cotton dyed with benzidine dyes. We observe numerous fibres the cellular walls of which are perfectly colourless, while the central canal appears more or less filled with colouring matter. This is particularly clearly visible in cotton dyed with benzopurpurin and immersed in absolute alcohol. The position of the dye is still better defined after treatment with acids, and observation between crossed Nichols. That this particular location of the dye cannot be distinctly observed on each particular fibre fragment is not to be wondered at. The cellular walls of the cotton are laterally perforated by an enormous number of very fine canals, and any colouring matter accumulated in these perforations, naturally will cause the appearance as if in certain parts the substance of the fibre itself was dyed through.

The view of the nature of cotton, substantively dyed, with benzidine dyes, which I have put forward above, makes the surprising readiness with which these dyes, upon the cotton fibre, can be subjected to numerous reactions a matter of course, while in the light of Witt's hypothesis it must appear quite puzzling, and requires the assumption of quite a monstrous dissolving power of cotton for compounds of every description.

In the above, I believe to have furnished conclusive proof, first that the benzidine dyes exist in the cotton in the free state, and not in the form of lakes, and further that the dyes or their solutions are occluded in the cellular cavities of the cotton fibre. This, however, does not explain, why only a limited number of acid dyestuffs are capable of dyeing cotton direct. That the diphenyl residue has nothing to do with this property, we have already seen, and indeed numerous dyes are known, which, although containing the diphenyl residue, do not possess the properties of a direct dye.

Now, it is evident that if the dyeing process in question is not a selective solution process, as Witt contends, but a phenomenon of diffusion as I suggested above, it must be possible to discover material differences between the constants of diffusion of acid dyes dyeing cotton direct, and of acid dyes not possessing that property. Up to now I have been able to examine, in this respect, a comparatively small number only of acid dyes, including benzidine dyes, but without exception it was found that the direct-dyeing dyes possess exceedingly low diffusion coefficients, while those of the ordinary acid dyes are invariably very high. It was further observed that of two direct dyes the one possessing the smaller diffusion coefficient, has the greatest avidity for the cotton fibre. Likewise did it become unmistakably evident that in the ordinary acid dyes a decrease of the coefficient of diffusion is invariably marked by an increase

of the capability to dye cotton. The complete series of these most interesting, and, I believe, also technically very important experiments, will be brought before this society at some future date.

At first sight the above observation appears strange, if not even improbable, it being by no means obvious why the dyes with the smaller diffusion coefficient should be the better cotton dye, considering that the ease with which a dye penetrates into the cotton increases with its diffusive capability. Indeed it can readily be shown that benzidine dyes penetrate very much slower into the cotton fibre than any of the other acid dyes. But the diffusion of the latter out of the cotton takes place quite as readily, while the slow diffusion of the benzidine dyes into the fibre, corresponds an equally slow diffusion of the dye out of the fibre. This is at the same time the explanation of the disagreeable "bleeding" of these dyes in washing, and which never ceases as long as there remains any dye in the fibre.

We are now in a position to give a complete definition of the process of direct dyeing with benzidine dyes, by describing it as *an aqueous solution of a dye of very small coefficient of diffusion, formed inside the cellular cavities of the cotton fibre by means of a dye-bath possessing a high osmotic pressure. The fastness of these dyes, as regards their "bleeding" off the fibre, is proportionate to the inertia of diffusion of these dyes and inversely proportionate to the magnitude of their diffusion coefficient.*

From the above it naturally follows that if by some means we could lower the diffusion coefficient of any of the ordinary acid dyes, this dye should now behave against cotton like one of the benzidine dyes. Without anticipating the results of my investigations on the comparative rate of diffusion of the coal-tar colours and the relation of this function to their constitution, which I intend to treat in a future paper, I may say that I found a very simple and ready means to considerably retard the rate of diffusion of a great number of dyes. This means consists in their conversion into barium salts. By converting croceine 3 B into its barium salt, we obtain a product which is fairly soluble in water. The diffusion coefficient of this salt is very much smaller, as the one of the sodium salt, which is the commercial form of the croceines. By making now a comparative dye test with the quickly diffusing sodium salt and the slowly diffusing barium salt, employing neutral bathis as in dyeing with the benzidine dyes, we observe very soon a marked difference between the two skeins under operation, a difference which is increased to a surprising extent, on the skeins being subsequently washed. The skein dyed in the solution of the sodium salt assumed a pale pink shade only, the skein dyed in the solution of the barium salt shows a deep, bright scarlet. This is a direct proof for my contention that *the capability of an acid dye to dye cotton like a benzidine dye is only a function of the coefficient of diffusion of the acid dye.*

The above-described method to reduce the rate of diffusion of an acid dye may, in some instances, be capable of advantageous practical application. In the present paper it was not my endeavour to arrive at any results of immediate practical bearing, but rather to contribute to the consistent and rational development of a theory of dyeing, which not only satisfactorily explains all the phenomena observed in dyeing, but will also prove itself a useful tool for further research, whether of scientific or practical purpose. Of course it might be objected that in the above arguments the behaviour of the silk fibre has not been taken into consideration. This omission is, however, intentional, and appeared advisable to me on account of the very vague nature of our present knowledge of the constitution of silk, under which circumstances it seemed to me rather hazardous to build conjectures upon such a doubtful basis. Witt's hypothesis rests largely upon the properties of dyed silk, and the behaviour of silk against dyes, but most of his arguments are merely analogies, the conclusive power of which is somewhat doubtful. This applies particularly to the inordinate importance this author attributes to the fluorescence certain dyes show upon the fibre. The state of solution of a dye, and any fluorescence it may exhibit, do not by any means bear such a simple relation to each other, that the appearance

of the latter could be said to imply the coexistence of the former. A boiling solution of rhodamine shows no fluorescence, but the latter appears on the cooling of the solution. Applying Witt's argument to this observation, we would have to assume that the hot solution is not a solution of rhodamine, on account of the absence of fluorescence. The phenomenon of fluorescence is obviously of a very complex nature, as it depends upon the molecular condition of the fluorescing substance, the temperature, nature of the solvent, colour of the light, and the presence or absence of other compounds. We further know substances which show fluorescence both in the solid state and in solution, whilst other compounds show most exquisite fluorescence in the solid state, but not a trace of it in solution. Such a complicated phenomenon like fluorescence, which itself requires explanation, is scarcely a suitable means for the explanation of other matters of doubt. For the rest I believe that it will be possible to explain even the dyeing of silk from the same elementary principles which I employed above in regard to wool and cotton.

#### DISCUSSION.

The CHAIRMAN remarked that the section was much indebted to Dr. Weber for coming from Manchester to give his paper on the chemical philosophy of dyeing, and invited comments upon his papers from the members present, especially those interested in the theory or practice of dyeing.

Mr. RAWSON said that Dr. Weber's theory of substantive dyeing was both novel and plausible. He asked if any steps had been taken to ascertain whether, in the case of benzidine dyes, an acid was liberated by the decomposition of the dye. In the case of wool dyed successively scarlet and magenta, Dr. Weber had quoted, as evidence that a compound had been formed with the wool, the fact that the dye was not removable by alcohol; but was it not possible that the magenta was fixed by the scarlet, notwithstanding the fact that the compound away from the fibre is soluble in alcohol.

Mr. RICHARDSON said that the paper was an important and valuable one, and the author was obviously awake to other theories than his own. He thought that silk might very well be used for experiments such as those described in the paper, for he had himself shown that it contained  $\text{NH}_2$  and  $\text{COOH}$  groups in about equal proportions, but no aldehyde group. The absence of this group would lead him to expect that the silk fibre would not stain blue with ferric ferriyanide; was he right in supposing this? (Dr. WEBER: Yes.) Also had Dr. Weber experimented on artificial silks, the nitro-celluloses and their recent non-combustible products?

Dr. LEWKOWITSCH thought that Witt's theory of solution had been propounded largely because Van't Hoff's ideas happened to be in the air at the time. Dr. Weber had suggested that, in benzidine dyeing, the migration of the dye into the water of the fibre was due to the addition of salt to the solution in the vat; why should not the salt equally diffuse in, and hinder the entrance of the dye?

Mr. PROCTER said that Dr. Lewkowitsch had to some extent anticipated him. He was not clear that osmotic pressure could take the colour into the water in the cavities of the fibres, since the salt would probably diffuse in at least as rapidly as the colour. Probably in dyeing as in tanning, the colloid character of the fibres themselves had much to do with the observed phenomena.

Mr. ALFRED WHITAKER said that he had noticed that a small piece of cloth dyed with a benzidine dye did not bleed when held under a tap, whereas a larger piece on a roll did. He wished to know why was it that crocein-scarlet would not stand washing whereas beta naphthol if diazotised and developed with para-nitraniline would? Would it be possible to pad benzidine-chloride mother liquor on cloth, diazotise, and develop it in the same manner and so get a fast colour?

Mr. ALFRED WHITAKER, called the theory put forward by Dr. Weber would seem to account for the fact of very strong baths being necessary in the dyeing of benzidine colours.

Dr. WEBER: Yes, and also of the bath being left unexhausted.

Mr. ALFRED WHITAKER, proceeding, wished to know if any method of fixing such colours in the fibre had been found by Dr. Weber. It was easy to get goods to a certain depth of shade, but after this point no greater depth of colour could be obtained. This also bore out Dr. Weber's theory; but by means of alternate baths of colour and fixing agent (if such had been found), it might be possible to get such deep shades on the goods as was now impossible.

Dr. WEBER, in reply, said that he had never, in the case of cotton, looked to see if any decomposition, with liberation of free acid, had taken place. It would, however, have been indicated by a colour change, the acid corresponding with Congo red being, for example, Navy blue in colour. The criterion as to the existence of an acid in the free state or combined lay in its ability to form a lake, or not. As to the case of successive dyeing alluded to, it was true that Chrysamine was insoluble in alcohol, but he had been dealing with wool-scarlets, the lakes of which with basic dyes are soluble. He quite agreed with what had been said about the silk fibre; at the same time the wool fibre had one advantage that silk did not possess,—it yielded decomposition products, e.g., laugenic acid, which had the same capacity for dyeing or forming lakes as the wool itself. Artificial silks he had not experimented with, having found it impossible to reduce their  $\text{NO}_2$  groups to  $\text{NH}_2$  groups. As to the process by which the dye was taken up, the fibre when placed in the dye-bath became filled with water and then acted as a dialysator, the (crystallised) dye diffusing until its osmotic pressure inside the fibre was equal to that in the solution outside, the maximum had then been reached, and no more dye could be taken up. It was for this reason that a benzidine bath could never be exhausted. Crocein washed poorly because it had a high coefficient of diffusion; para-nitraniline washed well because it contained no lake-forming groups, and so could form no sodium salt that might diffuse; it was a pigment, not a substantive dye. The difference in the fastness for washing observed in dyeing with one and the same direct dye was due to the relation between the shade produced and the concentration of the dye-bath. You might dye to a certain shade by boiling for 40–60 min. in a bath till the maximum of coloration was obtained, and you might also get this shade by boiling for a shorter time in a stronger bath, but the colour was not so fast in this second case as in the first. There was a great future for direct dyeing by means of the "developed" dyes, but it was a delicate process, and necessitated the use of ice, which was expensive in this country. There would be a much greater use for benzidine dyes if they could be made fast; on cotton they were not fast, not being there as lakes. It was no use to look for metallic mordants; copper had been tried, but the colour was spoiled by soap. The only way was to find an organic compound of an albuminoid nature that would form a lake with the dye. Laugenic acid would do this, but was too expensive. The author had himself obtained such a lake, of benzo-purpurin with an albuminoid substance derived from cotton; it was deposited as a slimy film in a bath that had been used for some weeks, but its true nature was as yet undetermined for it had not been found possible to decompose it either with acids or with alkalis.

## Glasgow and Scottish Section.

Chairman: R. Irvine.

Vice-Chairman: E. J. Mills.

Committee:

G. Beilby.	W. I. Macadam.
W. J. Chrystal.	J. S. Macarthur.
J. Clark.	T. L. Patterson.
C. J. Ellis.	J. Pattison.
G. G. Henderson.	J. B. Readman.
R. A. Inglis.	E. C. C. Stanford.
R. Irvine.	D. R. Stewart.
J. Falconer King.	R. R. Tatlock.

Hon. Secretary and Treasurer:

J. Stanley Muir, Chemical Laboratory, University of Glasgow.

Members who desire to read Papers, exhibit Apparatus, &c., or introduce Discussions during Session 1893-94 are requested to give early notice to the Hon. Local Secretary.

### SESSION 1893-94.

Glasgow:—

March 6th:—

Mr. James F. Techer. "Oil Gas."

Prof. E. J. Mills and Mr. W. Macfarlane. "On the Oxidation of Aniline."

May 1st.—Prof. E. J. Mills and Mr. Saweis. "On the Action of Alums on Gelatin."

Edinburgh:—

April 10th.—Dr. J. B. Readman. "The Effect of the Saline Constituents of Water on the Character of Beer."

Meeting held Tuesday, February 6th, in the Philosophical Institution, 4, Queen Street, Edinburgh.

### MR. R. IRVINE IN THE CHAIR.

THE CHAIRMAN, before beginning the business of the meeting, called on the Secretary to read a resolution expressive of the thanks of the Section to Mr. Fawsitt, the lately retired Chairman.

THE SECRETARY read as follows:—

"The committee and members of the Scottish Section of the Society of Chemical Industry record with sincere regret their acceptance of the resignation of Mr. Chas. A. Fawsitt, who has for the past 18 months so ably and indefatigably acted as their Chairman of Section. They cannot allow the occasion to pass without expressing their warmest thanks to him for the success which has attended the meetings of the Section during his tenure of office."

THE PRESIDENT, in moving the adoption of this resolution, said that Mr. Fawsitt had tendered his resignation of the Chairmanship of Section, because he considered that a chairman resident in Edinburgh would be better able to further the arrangements for the Annual Meeting of the Society in Edinburgh in 1894. In accordance with Mr. Fawsitt's desire the committee had accepted his resignation and had unanimously elected Mr. Robert Irvine to the vacant post.

The resolution was unanimously adopted and the Secretary was instructed to forward a copy of the minute to Mr. Fawsitt.

THE PRESIDENT then took the chair.

### THE PRESIDENT IN THE CHAIR.

## ON THE PREPARATION OF CARBON BLACKS FROM NATURAL GAS IN AMERICA.

BY GODFREY L. CABOT, BOSTON, U.S.A.

THERE is a certain small industry peculiar to the United States, of which, I think, no accurate account has yet appeared in any scientific journal or standard technical work, and which I am therefore encouraged to bring to your notice in the hope that it may at least have the charm of novelty to most of you. I refer to the manufacture of lamp black from natural gas. The substance thus obtained, known to the trade as carbon black, and also less frequently as hydrocarbon black, gas black, satin gloss black, jet black, silicate of carbon, and various other names, is collected on the under side of cast-iron plates or rollers, from which it is automatically removed by suitable scrapers of iron or steel. This gas is burned from gas jets such as are used for illuminating gas.

As is the case with so many more important inventions, the merit of discovery has been claimed by different people and belongs to no one alone.

The first person who collected soot on a surface thrust into a flame made carbon black.

For the purpose of this sketch, however, the industry may be traced to certain printing ink makers in Philadelphia and New York, who found that soot thus made by artificial gas gave a beautiful gloss and intense colour to printers' ink, differing totally in both of these respects from the lamp black obtained in the ordinary way by burning oily or resinous matters in an insufficient supply of air, allowing the smoke to settle in deposition chambers. A small amount was thus made in a private way at an expense of 2.25 dols. to 3.1 dols. per lb., till in 1872, or thereabout, Peter Neff, of Ohio, and Messrs. Haworth and Lamb, of Massachusetts, made experiments with natural gas for the same purpose, and the two latter erected in New Cumberland, Pennsylvania, the first factory in which carbon black was successfully made on a commercial scale. Others soon followed, too numerous to name, seeking thus to utilise the natural gas which was at that time going to waste in enormous quantities. This gas is always found with petroleum, and for many years it was chiefly regarded by the unscientific men who controlled the oil industry as a dangerous nuisance to be blown off and got rid of in every possible way.

The amount of this valuable and irreplaceable substance which has been thus utterly wasted, it is impossible to estimate with any approach to accuracy, but I think I am perfectly safe in saying that it may be measured in hundred of trillions of cubic feet. I myself have a well from which probably three billions of cubic feet of gas was wasted before any was utilised. Here, then, was an industry of which the raw material was natural gas, and the finished product easily paid for transportation over the worst roads.

The amount of capital required to start the business was comparatively small, and the price of the black high. The result was a crop of small concerns, most of which perished after a short existence.

Experience soon showed that the quality of the gas and its adaptability for making black, varied greatly in different localities; that the duration of the supply was very uncertain; and that the average uneducated man, even though backed by Yankee push and ingenuity, did not possess the requisite engineering or business ability to invent, erect, and maintain in successful operation the requisite machinery, nor to obtain and keep the requisite amount of trade.

The first factory erected contained a series of flat-bottomed cast iron pans arranged in rows and filled with water, which was supposed to improve both the quantity and quality of the black. Under each row of pans was a line of burners, ordinary gas tips, the flame of which impinged upon the iron and thus deposited the black. At intervals of 20 minutes a car travelled from one end of the row of plates to the other, supported on rails and drawn by a wire

rope, and a scraper fixed in the open top of this ear or travelling black box removed the black, which fell into the bottom of this ear, and was thence removed by hand, bolted, and packed in barrels for market.

Of all the factories that have since been erected perhaps no single one has used gas capable of producing so much black per thousand feet or of so soft a quality, and if there were any of this black still for sale, small quantities of it could be sold for four or five times the present market price.

The first lot of 500 lb. put on the market was sold for 2.50 dols. per lb.; the next 1,000 lb. brought 1.50 dols., and the factory paid for itself in three months. A year's output was soon contracted for at 1.25 dols. per lb., but as competition increased the price rapidly fell. By 1881 black was selling at 35 c. per lb.; by 1882 at 31 c.; in 1883 an offer of 24 c. per lb. for a 2,000-lb. lot was refused. Meanwhile various other methods of manufacture came into vogue. Many manufacturers tried cooling the depositing surface with water in various ways and all abandoned it, including the original factory, it having been proved that no material advantage was thus gained, either in quantity or quality, and an additional complication and expense, involving considerable, mechanical difficulties were thereby introduced. At first the great object of the manufacturers was to obtain the most intense colour, and the greatest gloss, and in this respect the original brand was soon surpassed, but at the expense of softness and opacity.

All carbon blacks bear a general family resemblance; are very intense in colour; glossy whether rubbed in the dry or in varnish; have an extraordinary mixing strength, which, however, varies greatly and compares differently with lamp black according to the medium in which they are ground with the diluting pigment, i.e., whether water, oil, varnish, or what not. Generally speaking, carbon black ground in oil with 100 times its weight of white lead gives a very dark grey, showing about three times the strength of a good lamp black and from five to ten or more times the strength of the poorer blacks.

They take twice as much oil to make a varnish of a given consistency as do ordinary lamp blacks, but are much harder to mix, harder to dry, and more apt to curdle or form clots.

All carbon blacks will mix with water by simply shaking them with it; lamp blacks, generally speaking, will not, and this is a convenient way of determining, in cases otherwise doubtful, whether a black is lamp black or carbon black.

The process originally used by Messrs. Haworth and Lamb is still in use at Sayenburgh, Butler County, Pa., by their successors, Messrs. Nolen and Boardman. Peter Neff, of Ohio, was the next to enter the field, using a process somewhat similar to the first. His gas came from the geological horizon known as the Berea grit, and was poor in quality for the purpose of black making. He used no water, and shaped his benches slightly concave below, a doubtful advantage. His was the only factory for carbon black ever operated in Ohio, and never exceeded an output of 125 lb. a day. It has not been in operation for two or three years or more.

The next process in point of time was the roller process, in which the collecting surface is a cylinder revolving on its axis. Various patents were taken out, and two or three factories were put in operation on this principle. It is very expensive both in cost of plant, repair, and consumption of gas, but is still used in one factory, and the black can be sold in small quantities at a high price.

When carbon black sold at 60 cents a lb., it was thought by many that if it ever became equally cheap it would replace the higher grades of lamp black made from oil, and, to a certain extent it has; but there are some uses for which lamp blacks are preferred, and there are brands which have remained practically unchanged in price for twenty years, and have increased the sale, at a price much higher than that of carbon black now.

Meanwhile the output of carbon black has been increasing at a very rapid rate with a downward tendency in the price. In 1881 there was probably made from 400,000 to 500,000 lb., all of it on various modifications of the bench principle and the roller principle. In 1883, a new mechanism

attained commercial success—that, namely, of a large plate, 24 feet in diameter, pierced with holes for ventilation, and revolving over stationary burners, and a stationary scraper, and a black box beneath it, from which last the black was removed by screw conveyers, and thus carried to the bolt, a cylindrical revolving sieve, through which the black fell to the bin beneath, from which it was lifted and packed by hand.

This machinery took the place of labour in sifting the black, and the loss in handling, due to the extreme lightness and fluffiness of the black, was diminished. As it comes from the sieve, the black is an extremely light, fine powder, weighing less than 5 lb. to the cubic foot. A gentleman, in conversation with a friend of mine, dipped his finger into a barrel of this unpressed black without noticing he had done so till his attention was called to it.

It will sometimes oscillate in waves at most like water, floating, as it were, on the air, which it mechanically imprisons. This may partly be due to its electrical condition from the friction of the scrapers working at a high temperature of 300° C. on a perfectly dry surface. The black is easily affected by electricity. If you rub sealing-wax smartly on the sleeve and pass it over the black, it will fly up as it caught by a baby eclone.

It is so dry and fine that I have seen it fly through a hole no bigger than a steel knitting needle, just as sand falls in an hour glass.

To transport it to Europe, or even shorter distances, it is necessary to compress it, which is a tedious and difficult job, far more so than with ordinary lamp black. The first successful packer was a screw press, worked by hand. To compress the black it was necessary to expel the air, and the difficulty lies in preventing the black spitting up with the air, which may be accomplished by covering the plunger of the press with a sheepskin, woolly side out. The wool retains the black, but permits the air to escape. A very important point is to shield the black from all air currents, from the time it is removed from the plates till it has been packed.

A slight air current will float away a considerable amount of black, causing loss and a nuisance.

In the years 1884 and 1885, small cast-iron rings, rotated spasmodically by a ratchet mechanism, came into use as depositing surfaces. These are 3 ft. outside diameter, 2 ft. inside diameter, and have a ring of gas jets underneath them. This process was the invention of Mr. A. N. Blood, since dead. By it, one half of the total output of carbon black is at present made. It is customary to put these in rectangular buildings, dimly constructed of sheet iron and steel, each containing 84 such rings in six rows, all actuated from a shaft outside the building.

The best output yet obtained is about 1 lb. per 1,000 ft. But I think there is at present no factory that reaches this average, and some not one-fifth of this.

Another process that has established itself, after five or six years of costly failure, is upon plates 24 ft. or so in diameter, with gas flames and black box rotating beneath it. Another process, introduced in the year 1891, and in use at one of the two largest factories, consists of a series of small, independent, rectangular plates, with a scraper moving back and forward under each by a reciprocating motion. It is a noticeable fact that mechanical success or failure has depended more upon the skill with which the different methods have been applied than upon their inherent excellence.

Since 1885 most of the factories have been so arranged that the black is handled wholly by machinery. From below the bolt it is raised by an elevator to a large bin, out of which it is packed by machinery. An automatic modified flour packer, working on the principle of a screw propeller, has now come into general use.

In 1886 the production reached 900,000 to 1,000,000 lb., and the price had sunk to 8 cents, although much was still sold at higher figures.

In 1889 some lots were sold that only netted the producer 3 cents. Since then the price has been better, netting the producers perhaps 7 cents a pound at their factories, on an average, for the last three years.

The output has meanwhile increased to about 10,000 lb. per day, and will amount this year to fully 3,000,000 of pounds.

The uses of the black are for printers' ink, paints, mineral black stove polish, shoe leather, rubber goods, fertiliser, colouring cement, mortar pulp and artificial stone, harness oil, stenciling, mixing with other blacks, such as lamp black, ivory black, &c., to improve their colour.

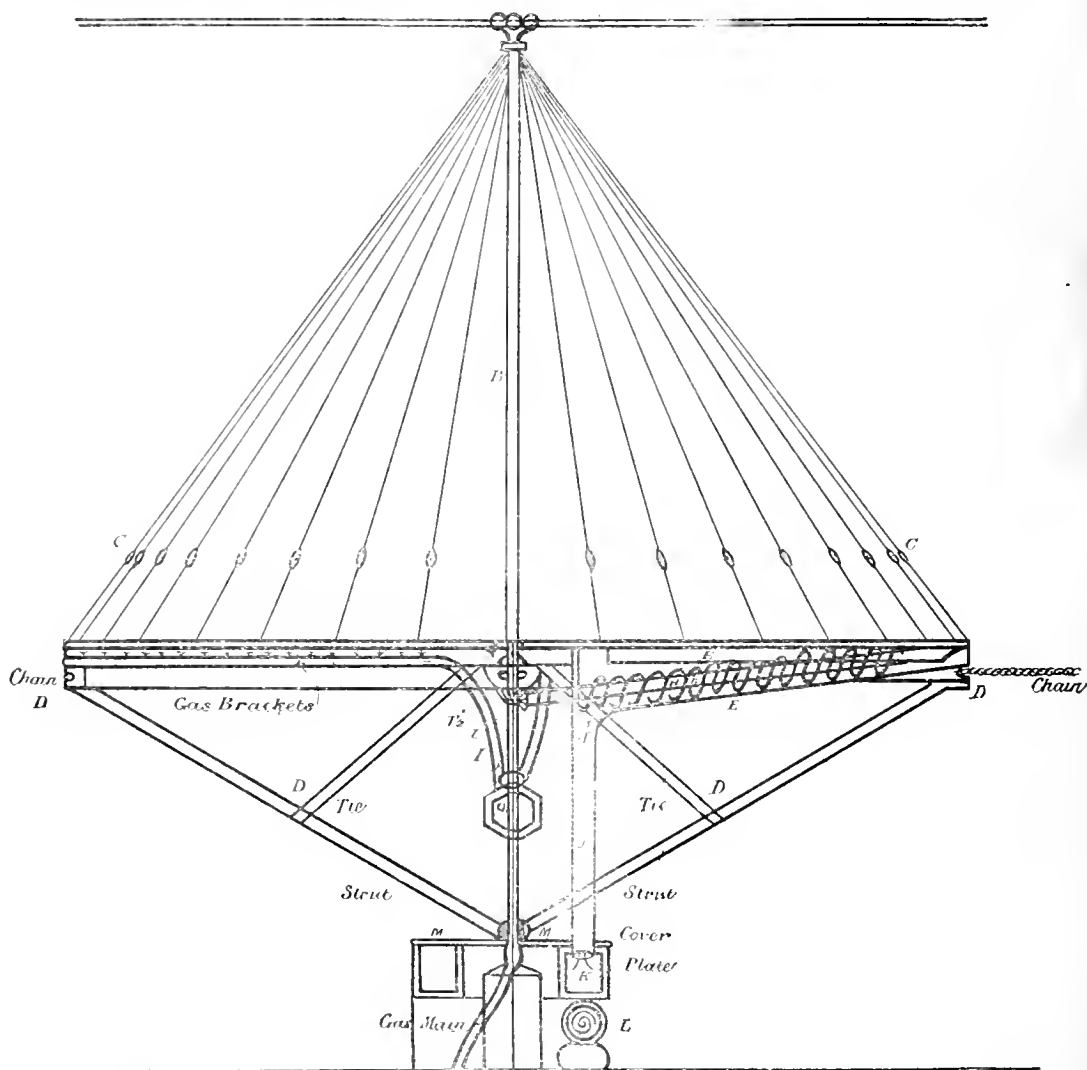
Chemically speaking, they contain 92 to 93 per cent. of carbon, 5 to 6 per cent. oxygen, and 1 to 2 per cent. hydrogen, and no trace of any mineral matter, nor have I ever known of gas black being adulterated, which I think is much to the credit of the trade.

*Description of Apparatus for the Production and Collection of Carbon Black from Gas.*

A circular plate of cast iron 24 ft. in diameter A, cast in 18 pieces and carefully levelled, is hung to a 16 in. mast B, a 3½-in. double extra strong tubing by guy wires C, tapped into a cap at the top of the mast and adjusted as to length

by twin buckles C. Beneath these, supported on pulleys at the foot of the mast and balanced by ties running horizontally to a collar on the mast, revolves once in the 15 minutes or so a framework driven by a chain D at its circumference, and carrying a black box and scraper E, a track of angle-iron for the chain to lie upon, and a gridiron of 1½-in. pipe with from 700 to 1,400 gas burners such as are used for illuminating purposes F, is supplied from a 4-in. O D feed line placed as near the middle of this gridiron as the mast, &c. will allow, and looking like the backbone of an unsymmetrical skeleton G.

This feed line takes its gas in turn from a 1½-in. pipe I, from a gas box rotating on a central mast, which is fed with gas from below. In the rotating black box is a screw conveyor H, actuated by a pinion at the inner end J, that engages in a small bevel box fixed on the mast. The black discharges downward from a spout into a circular trough-like box K, shown in section in accompanying sketch, and from here it falls through a hole in the bottom into a long conveyor that carries it to the elevator L, when it passes through a bolting machine made of very fine wire cloth, and



is then packed by machinery. On the circular box above reverted to, rotates a cover M, and two cardinal points are to be noted:—

1st. No draughts of air must touch the black at any stage of the manufacture.

2nd. It is made and handled wholly by machinery up to the time that it has been packed; thus a uniform machine-made black of excellent quality is obtained.

## NOTE ON THE ABOVE PAPER.

BY ROBT. IRVINE.

This manufacture is an adaptation of a process well known in England long before the discovery of natural gas in connection with the American oil wells. The same quality of black was made with similar apparatus, although on a much smaller scale, by printing-ink manufacturers here 30 years ago, who employed ordinary town gas for the purpose. My own experience confirms Mr. Cabot's as to the quantity of gas required to produce 1 lb. of black. Burning 1,000 ft. of gas per hour the produce never exceeded 1 lb.; and, of course, considering the price of town-made gas, this meant a first cost of at least 5s. per lb. on the black produced.

This beautiful substance, with its velvet-like gloss, is quite different in appearance from lamp black made by burning oils. But as regards *colour value* or covering power, there is between the two not the difference Mr. Cabot asserts. When the two varieties are reduced with, say, 100 per cent. of zinc oxide or chalk, the shade given shows very little difference between the two in colour value.

Of course any idea of foreign competition with this American manufacture is out of the question, and America will hold the field until her resources of natural gas are exhausted.

With regard to the matter of price. Taking 7 cents a lb. as the lowest price at which gas black can be delivered with profit in this country, as compared with the cost of black prepared from heavy dead oils (which yields 15 per cent.), we have the oil black at a cost considerably less than the gas black as at present imported.

Both of these blacks are contaminated by such bodies as chrysen and pyrene, which are of dirty brown colour; these dissolve in the varnish used for the manufacture of printing ink, and are apt to communicate this to the printed matter, and in process of time shows a kind of halo surrounding each letter. This is especially noticeable in old books or filed newspapers. These impurities can only be eliminated by igniting or calcining the black at a red heat.

It has often been suggested, and I believe has been tried experimentally, that the gas from the shale retorts in the oil manufactories in Scotland could be profitably employed for this manufacture; but considering the enormous quantity of gas required to produce this black it is most probable that the shale gas can be more profitably employed as fuel.

The PRESIDENT said that this process was obviously only applicable where gas could be obtained for nothing. Even Glasgow gas at 2s. 6d. per 1,000, and containing more carbon than Edinburgh gas, would not pay to burn for the manufacture of carbon black.

The form of press employed in packing this black was of interest, and he asked Mr. Irvine to further explain its method of use.

Mr. IRVINE said that the light substance was first put into a barrel capable of sustaining a level pressure, and in place of using an ordinary piston, such as would be employed with a coherent substance, the front of the piston was covered with a sheepskin the wool side outward. The wool allowed the air to escape but retained the solid black. The press worked admirably. Impressed black was so light that a large feather-bed tick filled with it could easily be lifted with one hand.

Dr. MARSHALL asked if blacks were made from poor coal gas enriched with the vapour of light oils.

Mr. IRVINE said that a process on that principle was being worked out at present and had been covered by patent.

Dr. READMAN asked whether it was possible to extract the oily impurities by means of solvents.

Mr. IRVINE said that although possible such a process would be too expensive, owing to the difficulty of handling so extremely light a material. By calcining this impure black the objectionable impurities were completely eliminated with a loss of about 15 per cent. of black.

## ON THE PRESENCE IN PAPER OF RESIDUAL CHEMICALS USED IN ITS PREPARATION.

BY ROBERT IRVINE, F.R.S., AND G. SIMS WOODHEAD, M.D.; F.R.S.E.

The simultaneous removal of excise restriction from the manufacture of paper, and the publication of newspapers form an era in the Victorian age, and the effect which followed was marked by the great increase of newspapers and their circulation, causing an extraordinary demand for increased supplies of paper.

Thirty years ago, most (if not all) of the paper produced in Great Britain was manufactured from rags; the process then obtaining being the simple and homely one of boiling with weak leys of soda, followed by the use of bleaching materials in very limited amount. Paper was then practically made up of pure fibre interwoven or felted together. Now cheap papers are a peculiar compound of China clay, stucco, and resin, held together by a skeleton of fibrous material obtained from wood, esparto grass, or straw, from which the fibre is eliminated only by drastic and costly chemical treatment. Had these new raw materials not been at hand, the rag supply of the world would have been insufficient to meet the present wants of Great Britain alone.

As a natural consequence paper fell in price, and expensive chemical operations necessitated the use of cheap loading materials as compensation—whilst the careful but tedious washing of the fibre or half stuff had either to be partially or wholly done away with.

It is unnecessary for our purpose to enter into the details of this great industry; but a very brief sketch of the modern processes in use will enable us to point out some of the dangers to the stability of paper thus introduced.

*Raw Materials.*

Esparto grass, wood pulp, and straw, are now practically the materials used in the construction of paper.

At the present day large quantities of the two former are imported into this country. Esparto, a grass from Spain and the north of Africa; wood in the form of dried pulp, prepared principally in Norway, Sweden, and Germany, by acting on it with sulphurous acid and sulphites (and the so-called sulphate of soda), also in some cases by the use of caustic soda. This is supplied to papermakers in a dry condition in the form of coarse thick sheets of a light brown colour.

The esparto or grass consists of a fibrous core surrounded by a sheath of silicious and gummy matter, which is firmly attached to the central portion, and to remove which chemicals (usually caustic soda) are plentifully used, the amount required being generally 10 per cent. of the weight of grass employed.

## ANALYSIS OF ESPARTO GRASS BEFORE TREATMENT WITH SODA.

	Spanish.	African.
Ash.....	3.72	3.34
Water.....	9.75	8.45
Water extract.....	10.98	10.05
Fat, &c.....	2.15	2.51
Cellulose.....	50.19	50.16
Incrusting bodies.....	27.23	28.83
	103.72	103.34
Less ash.....	3.72	3.34
	100.00	100.00



### Boiling.

During the boiling process much of the gummy and silicious matter is removed by the alkaline lye, to which it gives a deep brown colour, while at the same time a portion of the more delicate fibrous matter of the grass is destroyed. (In the case of wood fibre prepared by the sulphite process this treatment is not required.) The boiled material or stuff is reduced to a fibrous magma by suitable machinery; at the same time the yellow colouring matter and the soda are removed as far as possible by washing, but this is often so incomplete that from 1 to 2 per cent. of soda may be found in the washed stuff previous to the

### Bleaching Process.

For this purpose chlorine, in the shape of a solution of chloride of lime, is employed, in quantity varying according to the quality of the material, but the amount is never less than from 3 to 4 per cent. (equal to from 8 to 10 per cent. of dry bleaching powder) of the grass used, and sometimes even more. The action of this bleaching agent is sometimes accelerated and increased by heat or by the addition of sulphuric acid to its solution which sets free chlorine and its oxidation acids, the bleaching operation thus being performed very quickly (here also there is a definite loss of fibre). The entire removal of such an amount of chlorine from the bleached pulp by washing is a most tedious and difficult operation and requires an extravagant amount of water such as few ordinary mill supplies could afford. On the other hand, if the chlorine compounds are not entirely removed from the pulp the destruction of the paper within a brief space of its manufacture is certain, hydrochloric acid being formed by a partial breaking down of the cellulose which destroys its texture. This was a common enough occurrence when the increased demand sprang up consequent upon the remission of the duty, and when thus carelessly prepared the finished paper after being stored for some time became brittle and so lost its textile qualities, falling to pieces on being manipulated, a serious enough matter in a pecuniary sense for the paper maker, but unfortunately much more so in effect when such paper was used for printing valuable books.

To remedy this evil, advantage was taken of the property hyposulphites possess of decomposing hypochlorites. This cheap material could be more profitably used by the paper-maker than the enormous waste of water required to remove the last traces of chlorine from the pulp. Indeed the general practice which obtains in the present day often merely provides for getting rid of the chlorinated water from the bleached half stuff by pressing out that which can be removed in this manner and then adding antichlor; subsequent washing with water being either very partial or in some cases almost wholly dispensed with.

We have therefore in many bleached paper pulps the products derived from the action of hyposulphites upon the chlorine compounds, these consist principally of chlorides and sulphates which are in a general sense innocuous, but there is also a notable proportion of finely divided sulphur separated in the process which is precipitated and becomes entangled in the fibres and is thus transferred to the finished paper. The pulp at this stage is generally alkaline owing to the presence of unsaturated lime in the bleaching solution.

### Sizing.

For this operation alum, or acid aluminous compounds in conjunction with resin soap is freely used. Printing papers may in consequence give a distinctly acid reaction. In some cases starch is added to the pulp, which during the drying of the paper gives it a fine glossy surface. In such papers it is not impossible that there may be organic acids formed as products of the decomposition of the starch in presence of the water which paper attracts to itself when exposed to the damp atmosphere of our climate, this water generally amounts to one-tenth of its weight.

To counteract the brown tinge which even well bleached grass or wood fibre always retains, ultramarine blue is added to the finished pulp, which (as already stated) is sometimes of an acid character. When this is so, it gives

rise to another source of impurity, the ultramarine being decomposed by the acid; sulphur compounds are set free, which are peculiarly disagreeable, owing to the formation of hydrosulphuric acid. In this case also sulphur is separated and is transferred to the finished paper.\*

The presence of sulphur and its volatile compounds in ordinary paper may be detected very simply.

A small quantity of the paper under examination (about 1 oz.) is placed in shreds in a glass flask and covered with distilled or rain water. The neck of the flask is loosely plugged with a coil of white blotting paper which has been previously soaked in a solution of (basic) acetate of lead and dried. Gentle heat is applied but the contents of the flask are not allowed to boil—a watch-glass is placed on the top of the plug—in a short time the blackening of the lead compound, owing to the formation of sulphide of lead, will show if sulphur, sulphides, or loosely combined sulphur compounds are present.

It is true the amount of sulphur is generally very small, varying from mere traces to 0.5 per cent. in good papers, although it may be present in greater amount in cheap flimsy newspapers where obviously it does no particular harm; but where such papers are used for printing books or pamphlets, it may give rise to most objectionable results.

The sulphur compounds derived from the use of hyposulphites when that substance is used as an antichlor and those from ultramarine are distinguished in the following manner.

By treating the paper with water alone as described, if sulphur or sulphides derived from hyposulphites are present the plug of acetate of lead soaked paper will become coloured by the sulphide of lead formed, rendering it faintly brown or black in colour according to the amount present. If no such coloration occurs sulphur or sulphides are absent as derived from hyposulphites.

Of course this test is only of value when papers are neutral or slightly alkaline. If the paper gives an acid reaction the hydrosulphuric acid evolved may be partly or wholly derived from the ultramarine used, but if the paper is neutral and has given no colour reaction to the lead paper when heated with water alone, if a few drops of acetic or hydrochloric acid be added to the contents of the flask colouration at once shows.

Some ultramarines are so unstable that even the carbonic acid present in a loosely combined or free condition in hard waters is sufficient to cause decomposition and evolution of hydrosulphuric acid.

When sulphur and linseed oil are brought into contact there ensues a distinct and well-known chemical reaction, the sulphur breaks up the oil and combines with its hydrogen, hydrosulphuric acid being set free.†

As one of the principal uses of paper is that of a printing medium, and as in all the higher classes of printing ink, linseed oil is used as a vehicle or medium for the black or other colouring materials used in its manufacture, if free sulphur or sulphides or volatile sulphur compounds are present, the infallible consequence of using such papers for printing purposes is the production of sulphuretted hydrogen and other like products which impart their characteristic and disagreeable odour to the resulting book or periodical.

We admit that even the most carefully prepared printing inks retain the odour of the materials used in their manufacture, but this is very trifling, and we have become accustomed to associate all printed matter with the not unpleasant balsamic odour of the press-room, which odour, however, has no relation to the fœtid and disgusting smell which is engendered when sulphur is present in the paper used.

These results seem forcibly to show:—

1. That the use of hyposulphites as antichlor, even when the antichlored pulp is thereafter thoroughly washed, is unsafe and a mistake.

\* Practical papermakers have realised, to their cost, the trouble free sulphur gives in corroding the wire cloth and the dandy rolls which form such an important and costly portion of the paper-machine. The sulphur combines with the copper of the brass wire tissue, forming sulphide of copper, and the wire gauze becomes rotten long before it otherwise should be.

† The so-called vulcanising of linseed oil by this means is a well established process in the arts.

11. That the introduction of ultramarine to "clear" the brown colour of the fibre is also unsafe when acid aluminous compounds are used in excess in the sizing operation.

Both substances under certain conditions obtaining in paper manufacture may introduce, proportionately as they are used, sulphur or sulphides into the paper, with the consequences we have mentioned.

The remedy seems simple, and we advise the substitution of sulphites or bisulphites or even sulphurous acid for hyposulphites, the same end being gained by their use, these reducing agents destroying the active chlorine compounds without sulphur being precipitated. We understand many paper-makers now use sulphites in place of hyposulphites.

The most perfect antichlor for chlorine bleached pulp or cotton cloth would of course be peroxide of hydrogen, could a cheap method of its preparation be found (there are indications that the present costly process may soon be simplified and cheapened), peroxide of hydrogen would then supersede chlorine, presenting as it does the most perfect bleaching agent known.

As regards the use of ultramarine, we would advise the substitution of Prussian or aniline blues (the latter of which, however, is a very unstable colour), which cannot under any circumstances give rise to the unpleasant odour of sulphuretted hydrogen emitted by ultramarine when acted upon by even weak acids. The increased cost of these colouring matters will be more than recompensed by their higher tinctorial value. It is true that the ultramarine supplied to paper-makers is of the most stable character possible to a pigment into the composition of which alkaline or earthy sulphides enter as an essential part, but it must in any condition be a source of danger alluded to.

More complex changes than these we have indicated may occur when paper containing starch and sulphur or its volatile compounds is used for printing purposes, and it is not impossible to suppose that evil-smelling bodies allied to the mercaptans may be formed, especially if the paper is in mass as in bound books and exposed to heat and moisture.

The whole question we have raised is one well worthy of most careful study. The evil we refer to has caused much trouble and inconvenience lately, more especially in connection with one of the most widely circulated periodicals of the day, the pages of which, sometime since, smelt very vilely. We investigated the matter, and the smell was undoubtedly traced to the presence of sulphur compounds in the paper used; not that this paper was specially bad in this respect, containing only 0.20 per cent. of free sulphur, yet in this case the general conditions favoured the evil. In the case we refer to hyposulphite had undoubtedly been used by the paper-maker (who simply followed the course of his forefathers) and where such anti-chlor is used sulphur must be one of the products.

Of the many samples of paper we have examined we have found "Scotch printings" very free from "antichlor sulphur," but almost invariably we have found all papers to contain a notable quantity of sulphides derived from ultramarine.

We have not referred to the existence of these evils in regard to the high-class writing papers, but we have no doubt they will be found to exist there also.

The smell of paper and printing ink has in the past been "put up with," but the taste of the reading public has now become fastidious in respect to the physical as well as the moral purity of their mental pabulum.

## Obituary.

### M. ALFRED SOLVAY

FOUNDER AND DIRECTOR OF THE SOCIÉTÉ SOLVAY ET C<sup>IE</sup>, MEMBER OF THE SOCIETY OF CHEMICAL INDUSTRY, MEMBRE HONORAIRE DE L'ASSOCIATION DES INGÉNIEURS SORTIS DE L'ÉCOLE DE LIÈGE, &c.

ALFRED SOLVAY was born in the year 1840 at Rebecq, in Brabant, Belgium. His inclination and tastes were towards agricultural pursuits, but his father, M. Alexandre Solvay, who was a merchant, an owner of quarries, and a salt manufacturer, decided that he should pursue a commercial career. Accordingly at the age of 20 he was placed first with a shipowner at Antwerp and subsequently at Hull. In 1862, his brother, M. Ernest Solvay, who, at Brussels, had commenced the industrial application of his now well-known ammonia process for the manufacture of soda, invited Alfred to join him. This invitation the latter at once accepted, and about the end of the year 1863 the Solvay Company was founded, with M. Alfred Solvay as its director. Thus post he retained up to the time of his death.

The first manufactory was erected at Couillet, and operations were commenced in 1864. In 1867 the firm was awarded a bronze medal at the Paris Exhibition.

The first-fruits of success as regards public recognition of the merits of the ammonia-soda process, were reaped by its originators at the Universal Exhibition of Vienna in 1873, when they were awarded a diploma of honour, and the attention of the manufacturing world may be said henceforth to have been definitely directed to this new departure in alkali-making.

After this the two brothers were engaged in starting large works in England and France in 1874 and 1876; in Germany in 1880, 1883, and 1885; in Russia in 1883 and 1892; in America in 1884; and in Austria in 1885. The organising of so vast an association, meant for Alfred Solvay, who was chiefly concerned therein, a problem involving in the highest degree the qualities of diplomatic judgment and tact.

In 1879, he resigned with great regret, the directorship of the works at Couillet, and went to live in Brussels, in order to devote himself, with his brother, to the general administration of the numerous works, which he had contributed to found, and set in operation. Their development he followed step by step, and visited them regularly in turn each year. In spite of all this labour, Alfred Solvay nevertheless found time to devote to public affairs, and, spite of his extreme repugnance to municipal official positions, he allowed himself to be chosen as "conseiller provincial" of the canton of Châtelet in 1878, and in the same year as "conseiller communal" of Couillet. In 1893 he appeared in the Senate as representative for the arrondissement de Nivelles.

In the year 1877, the distinction of "Chevalier de l'ordre de Leopold" was conferred upon Alfred Solvay, and in 1890 he was exalted to the rank of an officer of that order.

His decease took place on the 23rd of January last at Nice, whither he had gone for a few days' rest.

In considering the character and personal qualities of Alfred Solvay, it is necessary to turn to certain incidents in the development of the work of his life, during which they were called forth.

The advent of the new industry was encompassed with difficulties. Its development demanded all the energy, courage, and patience the two brothers could put forth, to attack and to solve the many technical and commercial problems which then arose.

To this end, however, one circumstance was particularly favourable. This was the extraordinary confidence established between the brothers, so differently gifted that the one might be truly said to be complementary to the other.

We have thus a picture of the one brother (M. Ernest Solvay) continually engaged in the conception of ideas, and planning of designs and schemes, whilst the other was continually striving to carry them into practical execution, thus leading to inevitable improvements, revision, and consolidation. The community of confidence and the brotherly forbearance necessary for development on such lines, could not but prove a source of great strength to the entire concern.

In the year 1892, Alfred Solvay founded the Bacteriological Institute of the University, in Léopold Park, and to this end he presented the sum of 100,000 francs to the city of Brussels.

This memoir should not be closed without some reference to the tribute paid to the deceased by his workpeople at Couillet, and embodied in a funeral discourse delivered in their name by M. Bruyère, one of their number. The following sentences are extracted from it:—

"M. Alfred Solvay was no ordinary master or patron; he loved his workpeople, and lost no opportunity of coming to their aid on any occasion requiring it. We likewise were happy at any time to testify our affection for him, in return for the love we know he bore to us."

"Our venerated patron and master was of a compassionate and sympathetic nature." "The suffering of others was a sorrow to him; his greatest happiness he found in doing good; his benevolence was inexhaustible, and numerous indeed are the examples amongst ourselves of that fact."

The farewell, forming the close of this discourse, bears strong testimony in itself, as well as from the point of view of the workpeople, to the inestimable qualities of one, who had not himself suffered impoverishment in the acquirement of riches, but who seems to have chosen to treat the latter merely as contributory to the acquirement of the inner wealth of a benevolent heart.

It is as follows:—

"Adieu, cher et regretté patron, votre vie n'a été qu'une longue suite de bienfaits. Nous conserverons éternellement le souvenir de tout ce que vous avez fait pour nous."—W. S.

## Journal and Patent\* Literature.

Class.	Page
I.—General Plant, Apparatus, and Machinery .....	134
II.—Fuel, Gas, and Light .....	138
III.—Destructive Distillation, Tar Products, &c. ....	142
IV.—Colouring Matters and Dyes .....	142
V.—Textiles: Cotton, Wool, Silk, &c. ....	146
VI.—Dyeing, Calico Printing, Paper Staining, and Bleaching .....	148
VII.—Acids, Alkalis, and Salts. ....	151
VIII.—Glass, Pottery, and Enamels. ....	153
IX.—Building Materials, Clays, Mortars and Cements..	154
X.—Metallurgy .....	154
XI.—Electro-Chemistry and Electro-Metallurgy .....	158
XII.—Fats, Oils, and Soap Manufacture. ....	162
XIII.—Pigments and Paints; Resins, Varnishes, &c.; India-Rubber, &c. ....	162
XIV.—Tanning, Leather, Glue, and Size .....	164
XV.—Manures, &c. ....	165
XVI.—Sugar, Starch, Gum, &c. ....	165
XVII.—Brewing, Wines, Spirits, &c. ....	167
XVIII.—Chemistry of Foods: Sanitary Chemistry and Water Purification; Disinfectants .....	168
XIX.—Paper, Pasteboard, &c. ....	170
XX.—Fine Chemicals, Alkaloids, Essences, and Extracts	170
XXI.—Photographic Materials and Processes .....	173
XXII.—Explosives, Matches, &c. ....	173
XXIII.—Analytical Chemistry .....	174

\* Any of these specifications may be obtained by post by remitting *s.d.*—the price now fixed for all specifications, postage included—to Sir Henry Reader Lack, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

## I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

*The Wearing Away of Metal Plates by the Action of Steam Jets.* J. Walter. Chem. Ind. 16, 170—171.

THE author was lead to the consideration of this subject by the behaviour of a steam-jacketed copper boiling pan. The copper pan was originally 8 mm. thick, and the steam entered the jacket at its lowest point. Being rather too small, the copper pan, after it had been at work for some years, was replaced by a rather deeper one, but one which in all other respects was exactly similar to the original pan. After the second pan had been in use for about a year, a hole was blown through the bottom, opposite the steam inlet to the jacket.

Believing that the mishap was due to the metal having been worked down too thin at this point, a third pan was made, slightly thicker—12 mm.—which was carefully measured all over, before putting into use.

To avoid a second accident the pan was tested from time to time, and after about 14 months' work it was found that the bottom had been reduced to the thickness of paper.

This time the author attributed the wear to the hydrochloric acid used for cleaning out the pan. He had previously noticed the "pitting" produced on iron plates by the action of steam jets, and, although he could not in this case trace the wear to the steam jet, he placed the steam inlet at the side, as a precaution. Some 11 months later he was convinced that the wear was due to the steam jet, as he found the pan again worn away; not at the bottom, but opposite the jet. The reason the original pan lasted so much longer, is due, the author thinks, to it being shallower than the others, and therefore not so near to the steam inlet and not subjected to such intense action. The author mentions a similar case that occurred with a lead-lined vessel which was heated by blowing in steam from pipes, perforated with rows of holes on each side, fastened to the bottom of the vessel by lead straps. From the stirring of the contents of the vessel one pipe got twisted, whereby the row of holes on one side pointed downwards. Very shortly the steam jets issuing from them pierced a row of holes through the lead bottom.

It is pointed out that the action of a steam jet is similar to that of a sand blast. In the former the abrading particles, instead of being sharp grains, are minute particles of water and rust, &c. from the pipes. Besides the purely mechanical abrasion there is also chemical action. The steam carries with it air, which oxidises the freshly-exposed surface, and the oxide film thus formed is more readily removed than the metal itself. The appearance of holes produced by the impinging of steam jets on to copper and cast-iron plates is described, and the author concludes by pointing out that once the cause of such wearing away is recognised, its prevention becomes easy.

This prevention can be effected by placing over the point of impact thick metal discs about 200 mm. diameter; by making the direction in which the steam enters parallel to, instead of across, the jacket space; or by placing a thimble, closed at the top and perforated at the sides, over the steam inlet.—R. B. P.

*The Internal Rusting of Boiler Shells.* Pfeifer. Papier Zeit. 18, 2617—2618 and 2654—2655. From Mitt. aus der Praxis des Dampf-kessel und Dampf-maschinen-Betriebes.

THIS report commences with an account of the physical and chemical phenomena which promote and cause the formation of rust upon iron. The internal rusting of boilers is dealt with under two headings, namely, when the boiler is in use, and when it is standing idle. The causes of rusting are first considered, the most important being the introduction of air with the feed water. By properly placing the feed pipe, namely, so that the feed water enters the boiler near the low-water level, and thus meets the hottest layers of water, the air is quickly expelled and passes out of the boiler with the steam, unless pockets exist

in which it can accumulate. Such pockets are sure to rust rapidly, and it is recommended that they should be covered internally with a protecting paint or filled up with cement, provided that they are not subjected to external heating. It is also recommended that the feeding should be completed before the withdrawal of steam ceases for the day, in order that the water left standing in the boiler over night may be as free from air as possible. An efficient circulation is also a means of preventing rusting, as it hinders the formation of air bubbles on the shell, which, if they remain clinging to it, cause rusting. The author is of the opinion that faulty construction is more often the cause of internal rusting than unfavourable conditions of working, and summarises the means of preventing rusting as follows:—

*First, whilst the boiler is working—*

1. Removing air from the feed water before it enters the boiler.
2. Removing air from the water whilst in the boiler, and preventing its accumulation in pockets, &c.
3. Addition of chemicals to the feed water.
4. Protective coatings applied to the inside of the shell.

*Second, whilst the boiler is standing idle—*

1. Removing all moisture from the boiler, (a) by blowing it off whilst hot, (b) by producing an air current through it, (c) by placing hygroscopic bodies inside.
2. Direct protection of the shells, (a) by painting with tar, varnish, &c., (b) by covering with protective paints, and such an alkaline coating as milk of lime.
3. Protecting the shells from varying temperatures by keeping the draught in the flues constant, and so as to prevent moisture alternately depositing and evaporating on the shell.
4. Protecting the shell by completely filling the boiler with water from which all air has been expelled.—R. B. P.

#### *The Removal of Boiler Scale by Means of Petroleum.* Papier Zeit. 18, 2916—2984.

AFTER some opinions stated *pro* and others *contra* with regard to the use of petroleum for removing boiler scale, the following is quoted as the opinion of Mr. Haage, the chief engineer of the Saxon Boiler Inspection Company:—that experiments made in 1873 showed that on account of the varying composition of boiler scale, the method was not generally applicable; in some cases it caused the scale to fall off, in others it merely loosened it, whilst in many cases it had no effect. It is also pointed out, in some correspondence given, that as the petroleum mixes with the steam the latter cannot be used for many industrial purposes, such as paper-making, when this method of removing boiler scale is adopted.—R. B. P.

#### *The Ether Motor of P. de Susini.* Freytag. Dingl. Polyt. J. 289, 104—107.

In this motor steam is replaced by sulphuric ether vapour, which is produced at relatively much lower temperatures, viz., 35° C. In the latest form of this motor the sulphuric ether vapour is generated by the direct action of steam, and is then used in the cylinders of a compound cylinder motor differing but little from an ordinary steam engine. The inventor claims a saving of 65 per cent. as compared with results with an ordinary condensing steam engine. Figs. 1 and 2 show the general arrangement of a plant on this system. A is an ordinary boiler, the steam from which passes along *a* to the cylinder jackets of the motor B, which are thereby raised to a rather higher temperature than that of the ether vapour entering the cylinders; from here it passes along the jacket *a'*, surrounding the motor inlet pipe *b'*, to the ether vapour generator C, any water condensed either in the pipes, cylinder jackets, or in E flows back to A, to be re-evaporated. The ether vapour is produced by the heat from the steam in E, and its pressure is regulated by the reducing valve R, which controls the pressure, and therefore the temperature, of the steam entering C; the ether vapour passes along the steam-jacketed pipe *b'* to the motor B, in which it performs work

successively in the two cylinders; it then passes into the condenser D. The liquid sulphuric ether formed here by condensation is forced by the pump E back into the generator C, where it is re-evaporated.

Fig. 1.

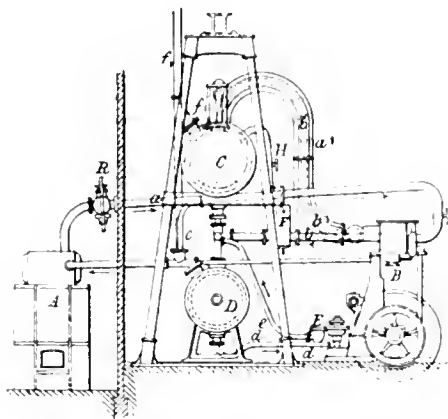
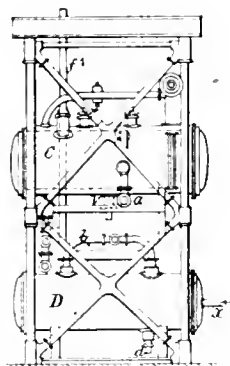


Fig. 2.

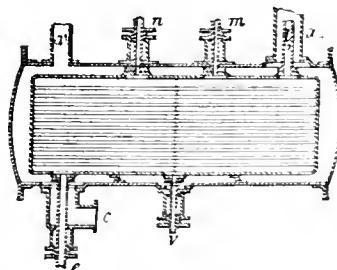


SUSINI'S ETHER MOTOR.

The difficulties formerly encountered in sulphuric ether motors consisted principally in leakage taking place at pipe and cylinder-cover joints, and at piston and valve rod stuffing boxes. These have been overcome by the design adopted, and by the exact work obtainable with modern machine tools.

As before stated, A is an ordinary steam boiler and B an engine differing but little from an ordinary inverted cylinder compound steam engine. Its cylinders are 229 mm. and 452 mm. diam. respectively and the stroke 300 mm. The construction of the ether vapour generator is shown in Fig. 3. It consists of two concentric cylinders. The inner

Fig. 3.

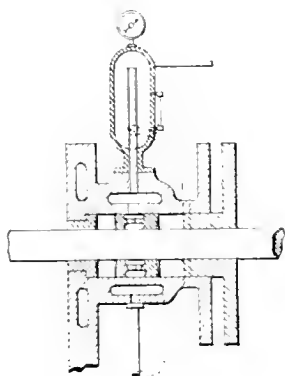


BOILER FOR SUSINI'S ETHER MOTOR.

one contains the sulphuric ether to be evaporated, and is traversed by a number of tubes passing through tube plates at each end. At the centre is a baffle plate through which the tubes pass, and which is perforated with holes between the tubes to allow the liquid in the cylinder to circulate. The tubes, of which there are 860, are 16 mm. diam. and 1 mm. thick. The steam, after it has passed round the cylinder jackets of the motor, enters at  $a^1 a^1$ , or steam direct from the boiler can be admitted, if desired, through the branch II, Fig. 1. Any water condensed, passes out through  $c$  back to the boiler;  $v$  serves for filling in sulphuric ether and  $m$  and  $n$  are connected respectively to pressure and vacuum gauges. Two safety-valves, one for the outer and the other for the inner cylinder, are fitted, and blow off into the pipe  $f^1$  (Fig. 1) leading to the outer air. The ether vapour produced passes to the motor through  $b^1$ . The condenser D is very similar in construction to C. The vapour passes into the inner cylinder, and cold water entering at  $x$ , Fig. 2, passes through the tubes, of which there are 1,264 of 11 mm. diam. and 0.5 mm. thick, and flows back through the space between the inner and outer cylinders to the exit pipe  $y$ , Fig. 2. D has also two safety-valves discharging into the pipe  $f^1$ .

Fig. 4 shows the method of packing the piston and valve rods. Each stuffing box is surrounded by a casing

Fig. 4.



STUFFING BOX FOR SUSINI'S ETHER MOTOR.

filled with glycerin, which passes through suitable apertures into a small piston in the stuffing box. The glycerin prevents the escape of vapour and at the same time lubricates the rod. Any vapour that may find its way into the stuffing box passes up into a small reservoir situated on top, and passes out through the upper pipe to the condenser D, Fig. 1. This reservoir is partially filled with glycerin, which passes into the casing round the stuffing box through the dotted pipe. The ether vapour on its way to D passes through F, where any glycerin it may carry over is arrested. F consists of a cylinder containing a series of concentric baffles. The plant is designed to work with live steam, but will work equally well with exhaust steam if automatic valves for dealing with water of condensation are fitted. The initial pressure of the ether vapour is 25 atmospheres, and the steam pressure 8 to 10 atmospheres. With the boilers evaporating 420 kilos. of water per hour the motor gave 50 actual horse power.—R. B. P.

*A Recuperative Gas Furnace.* C. Bigot. Monit. Scient. 1893, 7, 519.

See under XXIII., page 174.

*Filter for Diffusion Juice.* Bouvier. Zeits. Zuckerind. Böhmen, 1893, 18, 64.

See under XVI., page 167.

*Brown Paper from Wood.* Papier Zeit. 18, 2653.

See under XIX., page 170.

#### PATENTS.

*A Method of Lining Spigot and Socket, Flange or Snug Pipes with Enamel, Glass, Composition, or other Material.* F. Herbert, Birtley, near Newcastle. Eng. Pat. 22,682, December 13, 1892.

See under VIII., page 154.

*An Improved Furnace Pan and Concentrator for the Boiling of Fish Livers and the like.* C. B. Lotherington and I. Spencer, Hull. Eng. Pat. 23,542, December 21, 1892.

See under XII., page 162.

*Improvements in Filters.* E. Delhotel and E. Moride, Paris. Eng. Pat. 23,830, December 24, 1892.

In ordinary sand filters for the purification of water the upper surface of the sand soon becomes coated with the impurities contained in the water, whereby the action of the filter is considerably impeded.

In order to avoid this, the inventors give to the incoming water a gyratory action by making the inner end of the inlet pipe annular, and providing it with a number of angular branches, thus causing the water to issue tangentially therefrom, and to being kept in constant rotation above the surface of the sand.

Through the centre of the casing passes a vertical pipe, having an outlet below closed with a tap, the upper end reaching nearly under the cover of the casing and finishing with a bell-shaped mouthpiece.

When the rotary mass of water above the bed of sand has taken up a sufficient amount of impurities, the tap of the vertical outlet is opened and the impure water driven out through the vertical pipe, either by the pressure of the unfiltered water, or by the current of filtered water reversed for the purpose.—B.

*Improvements in the Heating of Surfaces for the Drying, Heating, or other Treatment of Paints, Oxides, or other Matters by the Aid of Electricity, and in the Means or Apparatus employed therein.* R. E. B. Crompton and H. J. Dowsing, London. Eng. Pat. 259, January 5, 1893.

See under II., page 138.

*Improvements in Apparatus and Appliances for Obtaining more perfect Combustion of Fuel in the Furnaces of Steam Generators or other Furnaces.* S. Taylor, Sheffield. Eng. Pat. 587, January 11, 1893.

See under II., page 139.

*Improvements in or applicable to Brine-evaporating Pans.* J. Parks, Northwich. Eng. Pat. 2400, February 3, 1893.

See under VII., page 151.

*Improvements in Apparatus used in the Concentration of Sulphuric Acid.* I. Levinstein, Manchester. Eng. Pat. 2476, February 4, 1893.

See under VII., page 152.

*Improvements in Thermostats.* A. Shields, Glasgow. Eng. Pat. 3057, February 11, 1893.

THE invention has reference to that class of thermostats in which the expansion and contraction of a sensitive fluid such as mercury, spirit, or the like, operate a diaphragm connected with the controlling apparatus. The apparatus consists of a chamber divided into two parts by a flexible diaphragm, the upper part being connected by a rod or link to a valve for regulating the supply of heating or cooling fluid; and the lower part communicating by means of a branched pipe with a reservoir of the sensitive fluid. The expansion of the fluid moves the flexible diaphragm and connecting link, thereby operating the controlling valve, while a spring brings back the diaphragm when contraction takes place. To prevent great expansion from bursting the apparatus, a safety diaphragm chamber is provided with its lower part communicating with the sensitive fluid and the upper face of the diaphragm pressed down by a powerful spring or by gas under pressure. When the expansion in the regulating chamber has reached the maximum amount, the safety diaphragm will begin to lift against the pressure above it, and thus all danger of bursting the apparatus will be avoided. The reservoir and the safety and regulating chambers are connected up by piping and provided with suitable gas cocks and valves; they are mounted on one casting.—E. G. C.

*A Process and Apparatus for Converting Non-Gaseous Hydrocarbons and Materials Containing them into Combustible Gas.* F. Siemens, London. Eng. Pat. 4022, February 23, 1893.

See under II., page 139.

*Improvements in or relating to Apparatus for Galvanising Sheet Metal.* J. V. Lagnesse, Grevegnée, Belgium. Eng. Pat. 4298, February 27, 1893.

See under X., page 158.

*Improvements in Gas Producers.* E. J. Duff, Glasgow. Eng. Pat. 7467, April 12, 1893.

See under II., page 140.

*An Improved Method of and Apparatus for Filtering.* J. P. Prat, Bordeaux, France. Eng. Pat. 7644, April 14, 1893.

THIS refers principally to filters in which conical filtering bags are employed, which in this case are stretched over skeleton frames made in the shape of double cones, one central cone surrounded by an annular cone joined at their bases. The small end of the bag is tied to the small end of the central cone, the bag then pressed into the annular valley between the two cones, and the larger end covering the outer surface of the larger cone, being finally tied at the largest diameter. The full length of the bag is thus made use of in an apparatus the height of which is about one-third only of the length of the bag.—B.

*Improvements in the Method of and Apparatus for Manufacturing Cyanides, Ammonia, and Fuel Gas.* A. J. Boulton, London. From E. Cowen, New York, U.S.A. Eng. Pat. 13,069, July 4, 1893.

See under II., page 140.

*Improvements in the Manufacture of Ammonia, Sodium Hydroxide, and Chlorine, and in Apparatus therefor.* C. Kellner, Vienna. Eng. Pat. 13,722, July 14, 1893.

See under VII., page 152.

*A Method of and Apparatus for Effecting the Absorption of Carbonic Acid.* A. Müller, Nieder Schonweide, near Berlin. Eng. Pat. 13,928, July 18, 1893.

See under VII., page 152.

*Improvements in Apparatus Employed in the Manufacture of White Lead.* J. V. Walton, Darlington. Eng. Pats. 3116, February 11, 1893, and 16,703, September 5, 1893.

See under XI., page 163.

*Improvements in or relating to the Treatment of Fish and Fish Offal and in Apparatus therefor.* J. C. W. Stanley, London. Eng. Pat. 17,205, September 13, 1893.

See under XV., page 165.

*Improvements in Apparatus for Illuminating and Heating by Means of Petroleum.* E. de Pass, London. From J. Leemana, St. Gallen, Switzerland. Eng. Pat. 18,660, October 5, 1893.

See under II., page 141.

*Improvements in Chamber Kilns.* F. H. Jung, Salisbury. Eng. Pat. 18,829, October 7, 1893.

See under VIII., page 154.

*Improvements in Filters.* J. N. S. Williams, Glasgow. From L. Spalding, Kauai, Hawaiian Islands. Eng. Pat. 20,006, October 24, 1893.

IN carrying out this invention a tank is employed through which passes a horizontal pipe at a convenient height from the floor. From both sides of this pipe branch off a number of smaller pipes closed at their outer ends, but perforated throughout their length. Suspended from each branch pipe is a frame of meshed wire, covered with a woven cloth bag, which also entirely encloses the branch pipe. The liquid to be filtered is maintained in the tank at a level above the pipes, from which it is discharged after having passed through the filtering bags.—B.

*An Improved Apparatus for Sterilising or Disinfecting Liquids.* J. H. W. Ortmann and C. W. C. Herbst, Hamburg, Germany. Eng. Pat. 19,760, December 23, 1893.

THE apparatus is intended for heating uniformly, liquids contained in bottles (Pasteur's method). The bottles are placed on a plate resting on blocks a short distance above the bottom of the bath. This plate has a series of perforations close to the edge, and there is a space of about 10 mm. between the outer edge and the sides of the bath. The plate has also a large central opening connected with a discharge pipe, which passes through the bottom of the bath. In the bottom of the bath there are two inlet pipes, about half way between the centre and the ends.

The bath is first filled with cold water. Hot water is then admitted through the two pipes into the space between the two bottoms. The heated water rises at the edge of the false bottom, and through the perforations, whilst the displaced colder water is discharged through the central pipe. This pipe has a special opening below the false bottom for the discharge of any cold water between the two bottoms.

The water becomes thus slowly but uniformly heated, and the desired temperature may be maintained as long as required by slow additional heating.—N. H. J. M.



## II.—FUEL, GAS, AND LIGHT.

*The Amount of Cyanogen in Gas-purifying Material and its Influence on Regeneration.* Dr. Burschall. J. für Gasbeleuchtung, 36, 7—10.

THE author points out that the Prussian blue formed during the purification of coal gas can be recovered almost without additional cost, and should form a source of extra profit.

He describes at length his own experiments on the subject, and summarises them as follows:—

The presence of ammonia during the purification causes a twofold formation of sulphocyanide, the amount of which, though it cannot be diminished in the purifier, nor its formation during the regenerating process entirely avoided, may in the latter process be diminished, first, by avoiding excessive heating of the mass; secondly, by getting rid of the ammonia as quickly as possible; both of these conditions are promoted by sifting the material as soon as possible after its removal from the purifier, when it contains the greatest amount of moisture, and also by spreading it out in as even a layer as possible, for regeneration. It must not be overlooked that old purifying material does not fix as much water as fresh material, and that the water formed during the absorption of sulphuretted hydrogen may cause disturbances. By rapid sifting a speedy regeneration takes place, and if a thorough airing does not at the same time ensue, the temperature rises and aids the formation of the sulphocyanide. The formation of Prussian blue, in spite of the ease with which the sulphocyanide is formed from cyanogen in the presence of ammonia and hydrogen sulphide, is explained by the fact that a not inconsiderable quantity of cyanogen escapes absorption in the first purifier, where most of the ammonia and hydrogen sulphide are caught, and consequently a fairly large quantity of cyanogen passes to those parts of the purifying plant where there is no hydrogen sulphide and only traces of ammonia present. Here the fresh purifying material permits a reaction to take place between the cyanogen and the oxide of iron, the compounds resulting from which cannot be transformed into the sulphocyanide at ordinary temperatures by ammonia and hydrogen sulphide, but require higher temperatures, such as occur during the further process of regeneration. It is these portions of the cyanogen which can be recovered as useful products by proper subsequent treatment of the purifying material, even in works where the gas is insufficiently washed.—R. B. P.

*Oxy-Oil Gas (Tatham's Process) and its Use for Purposes of Enrichment.* L. T. Thorne. Read before the Southern District Association of Gas Managers, November 23rd, 1894.

SOME time ago the author described (Incorp. Gas Inst. Trans. 1890, 143) the results of preliminary experiments to test the accuracy of the claims put forward by the patentee of this process, and expressed a favourable opinion thereon. In the present paper he describes further experiments on oxy-oil gas and gives the practical results obtained with it at Huddersfield, where it is now manufactured and used for enriching the town gas. The Huddersfield plant is designed to give an output of 200,000 cub. ft. of oxy-oil gas per day, but when the author's experiments were made there a portion only of the plant, producing about 40,000 ft. per day, was completed.

The essence of the Tatham process lies in the addition to heavy hydrocarbon gases, from whatever source obtained, of considerable quantities of free oxygen, this addition being preferably effected soon after the gases leave the retorts and whilst they are still hot. At present various low-priced petroleum and shale oils, which can be obtained in bulk in England at 3l. per ton, have been employed for making the oil gas. When oxygen is added to a heavy oil gas whilst the latter is still hot the illuminating power of the gas is raised. The maximum increase is obtained when from 12 to 18 per cent. of oxygen (according to the quality of the oil gas) has been added. Further addition of oxygen up to about 16 to 24 per cent. causes no further

change in the actual illuminating power, but renders the flame stiffer, whiter, and brighter to the eye, reduces the size of the dark zone, and improves the general shape and appearance of the flame. Still further addition of oxygen gradually reduces the illuminating power of the flame. The increase of illuminating power with very heavy oil gas is 25 per cent. or even more, with lighter oil gas rather less. As a rule the most suitable percentage of oxygen in the oxy-oil gas is about 20 per cent. if the oxy-oil gas is to be burned *per se*, about 15 per cent. if it is to be used for the enrichment of coal gas, &c. An oxy-oil gas showing an illuminating power of about 100 candles can be made, but taking into account both quantity and quality the most economical working in practice will probably be to produce an 80-candle gas, of which at least 25,000 cub. ft. would be obtainable per ton of oil used. Experiments have shown that oxy-oil gas is exceedingly stable. For instance, 500 ft. of oxy-oil gas kept in a 3,000-ft. holder for a week during varying frost and wet and stormy weather in November only lost 5.5 per cent. in illuminating power. When used as an enricher it is found that oxy-oil gas actually increases the stability of poor coal gas. A number of tests were made in which oxy-oil gas was added to unenriched coal gas and the mixtures then subjected to severe cold for some days. The mean of the results showed that, under like conditions, the unenriched coal gas lost 1.35 candles, that containing 5 per cent. of oxy-oil gas lost 0.95 candle, and that with 10 per cent. oxy-oil gas only 0.5 candle.

Experiments were made at Huddersfield extending over some time to determine the enriching power of the oxy-oil gas. The readings were all taken with a 2-candle Methven screen on a 60-in. photometer, the gas being in all cases burned from a standard argand burner. The following results were obtained:—

Gas.	Average Candle Power.	Number of Readings Taken.	Percentage of Oxy-Oil Gas in Tested Gas.	Gain of Illuminating Power Per Cent. of Oxy-Oil Gas.
Unenriched	14.42	600	..	..
Enriched...	17.05	480	2.96	0.89
" ...	18.07	120	4.51	0.81

thus showing a gain of over four candles for a use of 5 per cent. of the enriching gas. For comparison, oil gas was made under identically similar conditions (except of course the addition of oxygen), and enrichment tests made with this. The average of 150 tests gave an increase of 1.87 candles for 3.05 of oil gas or 3.05 candles for 5 per cent. enricher. The gas enriched with oxy-oil gas has a whiter and brighter appearance and a prettier-shaped flame than that enriched with cannel or with oil gas. The author estimates the cost of enrichment of coal gas by oxy-oil gas, at the present price of petroleum, at about one-third of a penny per candle per thousand cubic feet.—L. T. T.

### PATENTS.

*Improvements in the Heating of Surfaces for the Drying, Heating, or other Treatment of Paints, Oxides, or other Matters by the Aid of Electricity, and in the Means or Apparatus employed therein.* R. E. B. Crompton and H. J. Dowsing, London. Eng. Pat. 259, January 5, 1893.

THESE improvements are intended for the utilisation of electric currents for heating surfaces for drying or heating or roasting different materials in a cleanly manner, free from products of combustion, or which require to be heated in the presence of certain gases, and for this purpose electric conductors are embedded in refractory insulating material in the manner described in Eng. Pat. 17,091, 1892, which is preferred for the roasting of ores. Inclined drying shelves are shown arranged in a vertical chamber



or furnace, so that the material to be heated, fed into the chamber by an archimedean screw at the top, falls from one shelf to another into a collecting chamber at the bottom. The temperature of these shelves may be separately varied by resistances. The wires for high temperatures should be of steel, or alloys of nickel and steel, known as nickeline, embedded in ganister or silicious sand.—J. C. R.

*Improvements in Apparatus and Appliances for Obtaining more Perfect Combustion of Fuel in the Furnaces of Steam Generators or other Furnaces.* S. Taylor, Sheffield. Eng. Pat. 587, January 11, 1893.

This invention has for its object the introduction of air through a hollow fire bridge provided with suitable air in- and outlets. A number of metal projections, the object of which is to conduct heat to the air passing through the fire bridge, project horizontally from the front plate of the fire bridge inside. There is a second solid fire bridge behind the front one, which becomes highly heated by the fire and on which the heated air from the first or hollow bridge impinges, and causes a more perfect combustion. The air inlet to the hollow bridge can be opened or closed by two sliding plates, one of which is for regulating the amount of air passing through, and the other for automatically opening and closing it. The latter is controlled by a system of levers and rods in such a manner that when the fire door is opened for stoking, the air inlet is opened and admits air to the bridge; it remains open for a sufficient time after the door is closed to obtain a perfect combustion, the time being regulated by a cataraet cylinder attached to the mechanism.—R. B. P.

*A New and Improved Method for Saponifying or Mixing Tars, Oils, Hydrocarbons, and similar Materials, and for using the same for Heating, Lighting, and other Purposes.* C. A. Sahlstrom and E. Parr, both of London. Eng. Pat. 2155, January 31, 1893.

In one vessel the oil or other material is heated from 70° to 110° F. by passing through it a current of hot air or steam from a coil situated at the lower part of the vessel, and perforated with a series of fine holes. In another vessel water containing from  $\frac{1}{2}$  per cent. to 3 per cent. of alkali is heated to the same temperature. This solution is then run in a fine stream into the oil in the first vessel, which is continuously stirred during the mixing and for some 5 to 15 minutes afterwards. In this way the oil or other material can be made to take up as much as 150 per cent. of water. The resulting mixture can be used for lighting and heating alone, or it can be made into solid fuel by the admixture of ground peat, spent tan, sawdust, coal dust, or similar materials, and subsequently pressed into blocks.—R. B. P.

*Improved Apparatus for Vaporising and Burning Mineral Oil.* B. Grosche, London. Eng. Pat. 2901, February 9, 1893.

This invention has for its object the production of a cheap and efficient burner for mineral oil. In it the oil first passes through a "trap" filter and is then vaporised in a specially-shaped generator, whence the vapour passes upwards and strikes a "spreader" placed over the mouth of the burner, where it burns.—R. B. P.

*A Process and Apparatus for Converting Non-gaseous Hydrocarbons and Materials containing them into Combustible Gas.* F. Siemens, London. Eng. Pat. 4022, February 23, 1893.

This invention relates to the production of combustible gas from hydrocarbons such as petroleum, paraffin, naphtha, fats, tars, resins, wax, naphthalene, and the like, by igniting the surface of the hydrocarbon in a suitable chamber, and, after ignition, limiting the amount of air admitted. The heat

of combustion converts the hydrocarbon into combustible gas, which, together with the products of combustion, are led away for subsequent use.

Fig. 1 shows a vertical section through a suitable form of apparatus, and Fig. 2 a horizontal section on the line X-X.

Fig. 1.

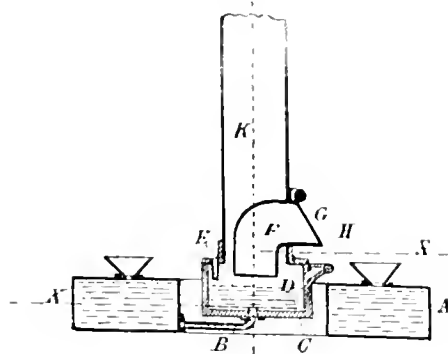
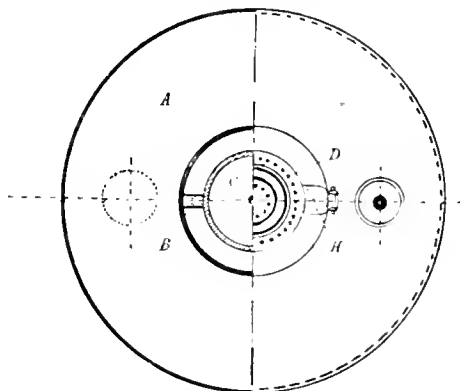


Fig. 2.

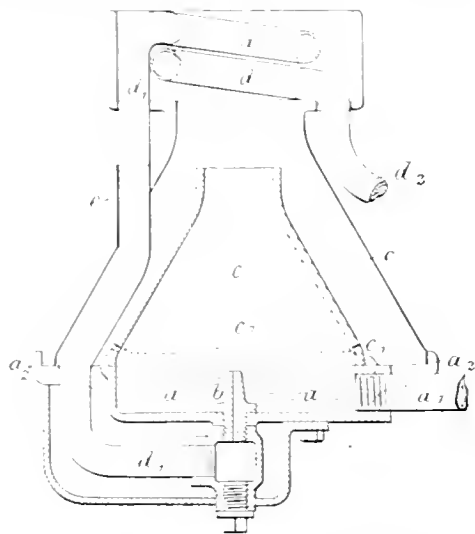


A is an annular reservoir supplying the liquid hydrocarbon, or the hydrocarbon liquefied by heat, to the central combustion chamber C through the pipe B. Through the cover of C are pierced a number of small holes D, admitting air to the surface of the hydrocarbon. An elbow pipe F, the lower end of which is covered by a perforated plate, and the upper end by a hinged door G, serves, by opening or closing G, to admit a further quantity, greater or less as required, of air to the chamber C. A lighted taper is introduced through H to ignite the hydrocarbon. Combustible gas is then produced, which ascends through K to a gas-holder.—R. B. P.

*An Improved Method of and Apparatus for Burning Hydrocarbons for Lighting and Heating Purposes.* F. B. Hill and R. D. Brett, both of London. Eng. Pat. 4211, February 25, 1893.

The object of this invention is to facilitate the combustion of heavy hydrocarbons, such as crude petroleum or tar. This is effected by using a jet of vapour of a lighter hydrocarbon, such as paraffin, in place of the usual jet of air, or of air and steam mixed. The figure shows the construction of the lamp: a is the receptacle for the heavy oil, the level of which is kept constant by a small cistern, fitted with a ball-cock, placed between the reservoir and a; b is the jet, and is in connection with the vaporising coil d. The latter receives the supply of light hydrocarbon through d<sup>2</sup>; e is a hood, perforated round its lower edge with air-supply holes e<sup>1</sup>; e is the outer casing, which is supported on lugs a<sup>2</sup> in such a manner as to admit air to the space between e and c. The

reservoir for feeding the lamp has two compartments, a large one for the heavy, and a smaller one for the light hydrocarbon. A pipe passes down to near the bottom of each, the one leading to the intermediate cistern for controlling the level of the heavy hydrocarbon in *a*, and the other supplying *d* with light hydrocarbon. The reservoir



is fitted with an air-pump for producing a pressure in its upper part to force up both the heavy and light hydrocarbon. In operating the lamp, the heavy hydrocarbon in *a* is ignited. The air entering through *c* is sufficient to produce a partial combustion, whereby an inflammable gas is produced, which mixes with the vapour of the light hydrocarbon issuing from *b*, and is carried upwards with it through the top of *c*; it here meets the air passing between the casing *e* and the hood *c*, and burns with an intense flame.—R. B. P.

*Improvements in Gas Producers.* E. J. Duff, Glasgow.  
Eng. Pat. 7467, April 12, 1893.

In order to reduce the cost of construction, the producer is made rectangular in form. It is built up of plane, flanged, cast-iron plates, and lined with fire-brick.

The air for combustion enters from a lower casing through perforated cast-iron plates, which slope upwards from the sides and meet at a ridge along the centre. Air is injected and fuel fed in in the usual manner. The ashes descend on each side of the ridge and fall through suitable openings along each side into a trough, which is partially filled with water, thus sealing the lower part of the producer.

—R. B. P.

*An Improved Fuel.* D. W. Nightingale and S. G. Wicking, both of London. Eng. Pat. 8040, April 20, 1893.

THIS is a mixture of 1 part gas tar, 4 parts petroleum, and 20 parts pulped waste material. The latter may be of any kind, such as old fibrous or textile material. The ingredients are incorporated and then pressed into blocks.

—R. B. P.

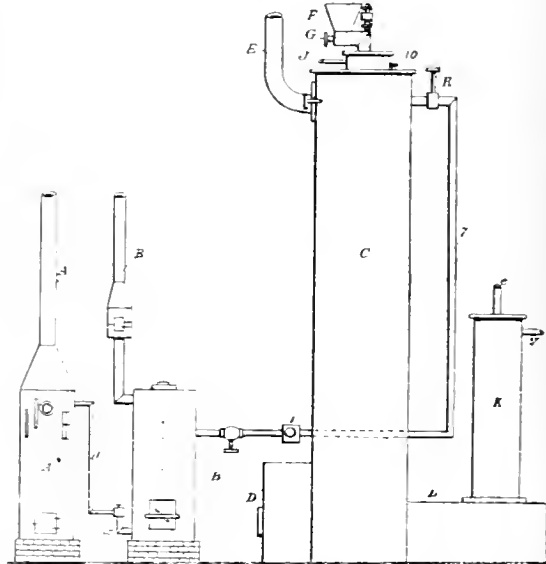
*Improvements in the Method of and Apparatus for Manufacturing Cyanides, Ammonia, and Fuel Gas.* A. J. Boulton, London. From E. Cowen, New York, U.S.A.  
Eng. Pat. 13,069, July 4, 1893.

THIS invention has for its object the rapid and economical production of compounds of nitrogen suitable for agricultural and industrial arts, and simultaneously the production of fuel gas.

Fig. 1 shows the general arrangement of the plant, in which A is a steam generator, B a gas producer, and C an apparatus for the production of cyanides, &c. By means of an injector or other similar appliance, *x*, a mixture of

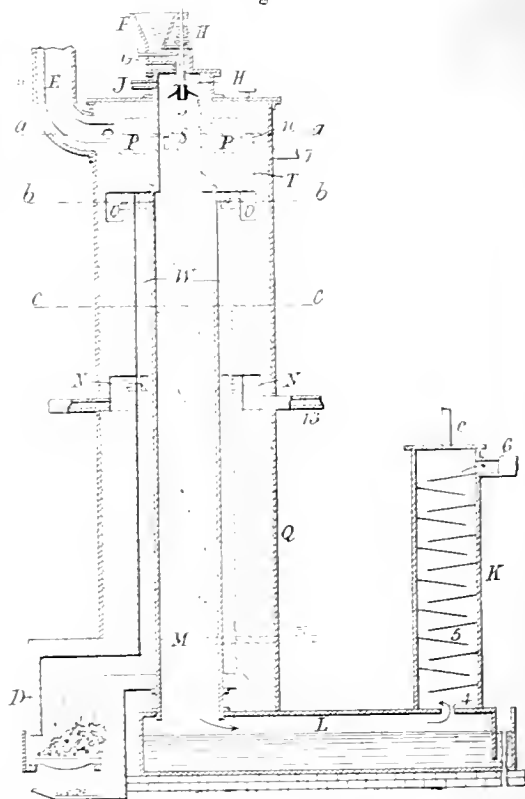
steam from A, and air is passed through a mass of incandescent fuel in B. In B, carbon mon- and di-oxide are formed, whilst hydrogen is freed, the resulting gas consisting of a mixture of these bodies with a large volume of nitrogen

Fig. 1.



in a condition suitable for the formation of cyanides. The construction of C is shown in Fig. 2. It consists of a stack of refractory material, at the upper end of which is the feed

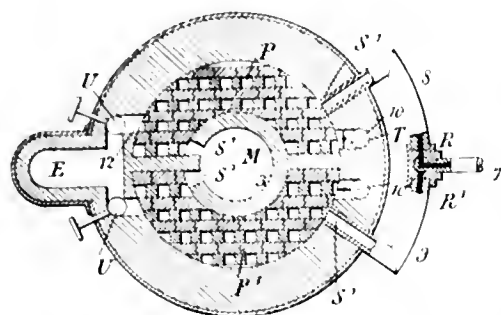
Fig. 2.



mechanism. M is a vertical retort, of refractory material also, which communicates with a tank L and a washer K. P

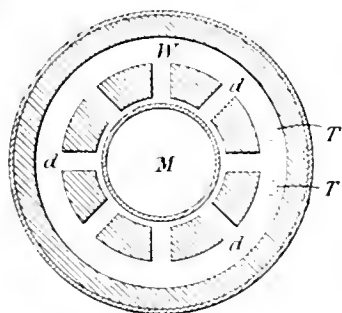
and  $P^1$  (Figs. 2 and 3) are superheaters. The arrangement of the various ports, passages, fire, &c. is shown in the

Fig. 3.



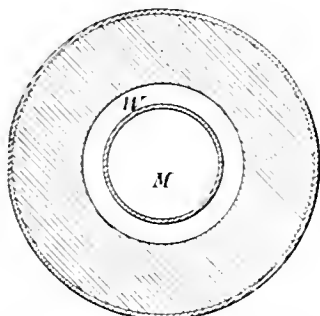
figures. Assuming that the apparatus, including one of the superheaters, has been brought to a sufficiently high temperature by the fire D, communication is established so that gases from B (Fig. 1) flow through the hot superheater and  $S^1$  into the feed opening, where they meet the material falling from the feed apparatus and pass down therewith through

Fig. 4.



the retort M. Simultaneously, a suitable volume of hydrocarbon gas or vapour, or a mixture of both, is introduced through J, which mixes with the gases from B. The material referred to above, which is fed in through the feeder at the top, consists of a suitable volume of alkali or alkaline earth. In passing through M, the gases being subjected to a high

Fig. 5.



temperature are split up and new compounds are formed. The various reactions going on are described, which are said to result in the formation of cyanogen and its salts, ammonia, and fuel gas which passes out over the water in the tank L, up through the washer K, and out through the pipe O. Various modifications in working, for varying the relative proportions of the bodies produced, are described.—R. B. P.

*Improvements in Apparatus for Illuminating and Heating by means of Petroleum.* E. de Pass, London. From J. Leemann, St. Gallen, Switzerland. Eng. Pat. 18,660, October 5, 1893.

This invention relates to improvements in the apparatus described in Eng. Pat. 13,681, August 30, 1890. The improvements aim at obtaining a more perfect combustion. The burner consists of two flat zig-zag passages formed of parallel tubes screwed into suitable top and bottom fittings; these are placed opposite each other at a small angle, and surrounded on four sides by metal plates, thus forming a hollow wedge-shaped box open at the top and bottom. Oil is fed into each of the zig-zag passages and is vaporised therein. The vapour passes into the wedge-shaped box at its lower, or wider end, and issues at the upper narrower end, where it burns, heating the burner and vaporising the oil. Air is pumped into the oil reservoir so that it bubbles up through the oil. The lamp is started in the usual way.—R. B. P.

*Improvements in Apparatus for Carburettling or Enriching Gas or Air.* A. Love, Barking, Essex, and D. Weston, London. Eng. Pat. 20,860, November 17, 1893.

The method of enriching gas adopted in this invention is to inject a spray of the enriching medium into a chamber through which the gas to be treated is passing, the latter at the same time being thoroughly stirred by means of paddles revolving in the said chamber. Briefly the apparatus consists of three parts: (a) a chamber for receiving the enriching medium; (b) a plunger pump for producing the spray; and (c) the enriching chamber for treating the gas in.

These three parts are shown superposed: (a) is at the top, and consists of a simple chamber into which the enriching medium is fed by forcing compressed air into the top of the air-tight casks in which it is stored. Any vapour given off from the enriching medium collects in the upper part of the chamber (a), and passes thence to the gas main, where it mixes with and helps to enrich the gas. From (a) the enriching medium passes through a "sight-feed" tube, fitted with a regulating cock to the suction pipe of the plunger pump (b). The suction pipe is so placed that the plunger shuts it off from the pump barrel except just at the end of its out stroke. In this latter position the enriching medium flows into the pump. The plunger then descends, cuts off the suction inlet, and forces out the enriching medium that has entered the pump, driving it through a perforated diaphragm, in the form of a spray, into the lower chamber (c). This is a cylindrical casing fitted with removable end covers, which carry by means of glands a rotating shaft, to which are attached the paddles. The latter consist of a light framework covered with wire gauze. The chamber (c) is arranged as a by-pass to the gas main in such a manner that either the whole or only a part of the gas passing through the main can be sent through it. The gas in (c), by the stirring action of the rotating paddles, is brought in intimate contact with the enriching medium injected as a spray, and also with a film of it that forms on the paddles, and thus becomes thoroughly enriched or carburetted.—R. B. P.

*Improvements in the Manufacture of Incandescent Bodies for Incandescent Gas Lighting.* R. Langhans, Berlin, Germany. Eng. Pat. 22,396, November 22, 1893.

THE object of this invention is to render the oxides employed as a skeleton for incandescent gas lighting more durable in regard to their fragility, and consists, mainly, in employing, instead of a cotton fabric, a tissue or plaiting of very fine platinum wires, which are covered or coated by electrolysis, with the oxyhydrates, carbonates, tartrates, or oxalates, by connecting the platinum tissue with the negative pole of a source of electricity (corresponding to a cathode), immersed in a saline solution, or a mixture of saline solutions of the suitable earthy metals, until a sufficiently thick layer of the oxyhydrates and other insoluble compounds is deposited upon the metallic foundation or base.

Advantages are said to be derived from adding ammoniacal, rather than sodium or potassium, salts to the electrolyte, hydrates of the latter being inseparable from the precipitates. These precipitates are slowly dried, and then heated until nothing remains on the platinum wires but pure oxides, either alone or in combination.

It is also proposed to wrap cotton wool, &c. on the platinum wire, or to wrap platinum on a core of yarn, in various ways, and then treat electrolytically; and other methods and materials are mentioned for obtaining metallic and non-metallic foundations, as aluminates and certain silicates, in conjunction with conducting carbonised organic textile fibres; and aluminium covered electrolytically with oxides, and then itself converted into a hydrate by immersion for three or four days in a strong solution of peroxide of hydrogen, which is said to produce a fixed hydrate without deformation.—J. C. R.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*The Alkali Test for Kerosene Naphtha Products, &c.* K. Lissenko and A. Stepanow. *Dingl. Polyt. J.* **290**, 139.

See under XXIII., page 177.

#### PATENT.

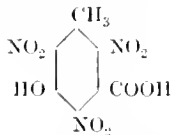
*New or Improved Treatment of Hydrocarbons (or of Analogous Substances Composed mainly of Hydrogen and Carbon) to Decompose them and Obtain Various Products.* W. J. Wisse and A. Schneller, The Hague, Holland. Eng. Pat. 21,343, November 23, 1892.

See under XI., page 160.

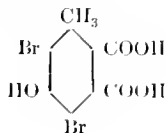
### IV.—COLOURING MATTERS AND DYES.

*The Colouring Matter of Cochineal.* W. v. Miller and G. Rohde. *Ber.* **26**, 2647.

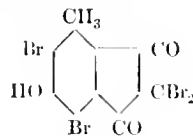
THE first important result regarding the constitution of the colouring matter of cochineal was obtained by Warren de la Rue, who discovered nitro-cochineic acid amongst the products of the oxidation of carminic acid with nitric acid. Liebermann and Dorp showed this acid to be trinitrocresotinic acid, the constitution of which, according to Kostanecki and Niementowski, is:—



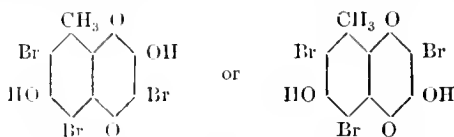
The observation of Hlasiwetz and Grabowski that carminic acid is a glucoside capable of decomposition into glucose and carmine red, has been shown by various observers to be erroneous. Of the greatest interest was the discovery by Will and Leymann of two bromine derivatives of carmine red, which on further treatment yielded a methyl-dibrom-hydroxyphthalic acid—



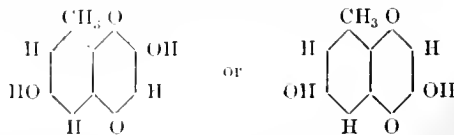
The formation of this acid is due, as the authors of the paper show, to the decomposition of a bromine derivative (a) of carmine red of the formula—



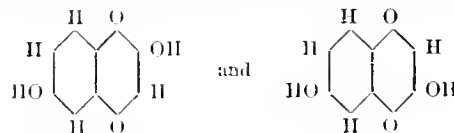
by means of a solution of sodium carbonate. The other (b) of the two above-mentioned bromine derivatives of carmine red appears to stand intermediate between carmine red and the  $\alpha$ -derivative referred to, as the latter is easily converted into the former by oxidation with bromine in a solution of sodium carbonate. This fact strongly points out the naphthoquinone nature of  $\beta$ -bromocarmine, and indeed it was found that  $\beta$ -bromocarmine, on reduction with zinc dust in an alkaline solution, yields a hydroquinone, and from this fact one may infer that  $\beta$ -bromocarmine is a derivative of an  $\alpha$ -naphthoquinone and possesses the formula—



The conclusion is arrived at that carminic acid, which is identical with carmine red, is itself a derivative of methyl-dihydroxy- $\alpha$ -naphthoquinone of the formula—



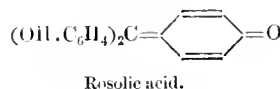
With regard to the future synthesis of carminic acid, experiments to produce the dihydroxynaphthoquinones—



would be of very great interest.—C. O. W.

*Constitution of Hydrocyanorosaniline and Fuchsin.* E. Fischer and W. L. Jennings. *Ber.* 1893, **26**, 2221—2225.

THE hydrocyanorosaniline prepared by E. and O. Fischer (*Annalen*, **194**, 275) has the constitution  $\text{CN} \cdot \text{C}_6\text{H}_4 \cdot \text{NH}_2$ . Rosenstiehl believes that the coloured salts of *p*-rosaniline have an analogous constitution, from which it might be supposed that the compounds would bear a great resemblance to one another, instead of which they are in every way dissimilar. Rosenstiehl's formula does not explain the relation of *p*-fuchsin to rosolic acid, which relation is clearly shown by Nietzki's quinonoid formulae, thus:—



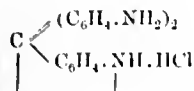
The analogy which exists between quinone compounds and the rosaniline dyes was pointed out by Graebe and Caro (*Annalen*, **179**, 184). Rosenstiehl's chief objection to the older formulae for the salts of *p*-rosaniline is that

these formulæ do not explain the combination of three additional molecules of acid; this, however, finds its analogy in the combination of quinone with hydrogen chloride. The constitution of the salts of rosaniline containing 4 molecules of acid is not, however, definitely settled.

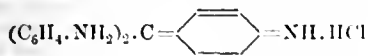
The authors have effected the conversion of pararosaniline into triphenylcarbinol by employing the Sandmeyer-Gattermann diazo method; the views of E. and O. Fischer (*loc. cit.*) as to the relation of these compounds are therefore confirmed.—A. R. L.

*Pararosaniline and its Salts.* A. Miolati. Ber. 1893, 26, 1788—1790.

THE change which takes place when *p*-rosaniline  $\text{OH} \cdot \text{C}(\text{C}_6\text{H}_4 \cdot \text{NH}_2)_3$  is converted into its coloured salts is at present a moot point. E and O. Fischer regarded the constitution of *p*-fuchsin as—



whilst Nietzki in 1889 (Chem. d. organ. Farbstoffe, S. 87) assigned to it the formula of a substituted quinone-imide derivative—



and Rosenstiehl and von Richter suggest that it may be formed by simply substituting chlorine for hydroxyl in *p*-rosaniline, which leads to the formula  $\text{Cl} \cdot \text{C}(\text{C}_6\text{H}_4 \cdot \text{NH}_2)_3$ . Now if the latter hypothesis be correct, *p*-fuchsin, like other analogous compounds, must be a non-electrolyte, whereas by either of the first two formulæ, which represent it as the hydrochloride of a base, it must conduct electricity.

The author has proved experimentally that the solution of the salt does conduct electricity, its molecular conductivity being about 46.5, or about half that of quinaldine (J. Walker, Zeit. physik. Chem. 4, 333), *p*-rosaniline is accordingly a very strong base. *p*-Fuchsin must therefore undergo electrolytic dissociation in aqueous solution, and cannot have the constitution assigned to it by Rosenstiehl and von Richter.—A. R. L.

*The Formamides of Alizarin.* M. Prud'homme and C. Rabaut. Bull. Soc. Ind. Mulhouse, 1893, 223—224.

By the action of formic acid the amidoalizarins yield formamides.

*β-Amidoalizarin.*—One part of *β*-amidoalizarin, dried and reduced to fine powder, and 10 parts of formic acid (20° B.), are heated together for 7—8 hours. The product of the reaction diluted with water, is thrown on to a filter and washed with distilled water. Caustic soda or potash dissolve it to a bluish-violet, ammonia to a red-violet solution. These solutions do not deposit lakes like those of amidoalizarin. The formamide in the dry state has a yellow colour, whereas *β*-amidoalizarin is brick-red. Treated with concentrated sulphuric acid at 150° it yields *β*-amidoalizarin with liberation of  $\text{CO}_2$ . Heated on a water-bath with glycerol and sulphuric acid it is transformed into alizarin blue.

This product is the dihydroxyanthraquinone formamide of the formula  $[\text{OH}:\text{OH}:\text{N}:\text{CHO}=1:2:3]$ . It is slightly soluble in water. With alumina it yields an orange, with chromium and iron mordants browns are obtained. If calcium acetate be present in the dye-bath, alumina gives brownish-red; chromium, brown; iron, black or violet shades.

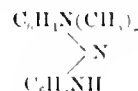
*α-Amidoalizarin.*—The author has obtained the dihydroxyanthraquinone formamide  $[\text{OH}:\text{OH}:\text{N}:\text{CHO}=1:2:4]$ . The colour of its alkaline solutions differs little from those of *α*-amidoalizarin. In the dry state this product is yellowish-brown. Concentrated sulphuric acid transforms it first into *α*-amidoalizarin, then into purpurin. With glycerol and sulphuric acid it yields the corresponding alizarin blue. It

is slightly soluble in water. The shades given with the various mordants are very similar to those yielded by alizarin. The addition of calcium acetate has little effect.

—R. B. B.

*A New Direct Grey.* Report by T. Rammann on a Paper by M. Petzold. Bull. Soc. Ind. Mulhouse, 1893, 221—222.

A MIXTURE, in molecular proportions, of aniline hydrochloride and hydrochloride of nitrosodimethylaniline is slowly heated to 60°. The yellow mass becomes in turn darker yellow, yellowish-green, and black, finally violet-blue. When this point is reached all the nitrosodimethylaniline will have entered into combination. The reaction is complete when a small portion of the product placed on filter paper and moistened with boiling water no longer gives a yellow ring. The colouring matter is then precipitated with common salt and zinc chloride. After being dried and ground it appears as a black powder, dissolving easily in cold water to a violet-blue solution. The author believes that this colouring matter may be considered as the hydrochloride (or double zinc salt) of an indamine, the base having the constitution—



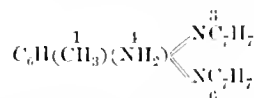
This formula is somewhat doubtful, considering the instability of the indamines.

The product is a basic grey, dyeing cotton, wool, and silk directly. It can be applied to either fibre as a steam colour. It is dyed on cotton either direct or with tannin mordant. When applied with tannin and antimony salts it resists the action of sulphuric acid, chlorine, soap, and light.

Generally speaking, it behaves like other basic greys, *e.g.*, nigrosine, nigramine, and methylene grey.—R. B. B.

*The Oxidation of Paratoluidine.* A. G. Green. Proc. Chem. Soc. 1893 [129], 237—238.

THE red crystalline substance obtained by Barsilowsky in 1873 by oxidising paratoluidine with potassium ferrocyanide has been variously considered as a polymeric paratoluene, as a tritoluylenediamine, and a complex amidoazo compound. The author has re-examined this remarkable substance. He arrives at the conclusion that the substance is a diparatolylimide represented by the formula—



derived from amidoaliquinone. On reduction, it takes up two atoms of hydrogen, affording a stable, colourless, leuco base. This latter is readily re-oxidised to the original substance, has very slight basic properties, and is not decomposed by acids or further reduced by boiling with stannous chloride. The leuco base condenses readily with benzyl, forming a coloured azonium compound; and it yields colourless, non-oxidisable anhydro compounds with formic and acetic acids, thus showing all the characteristics of a mono-substituted orthodiamine. This result, taken in conjunction with the presence of two paratolylimido groups in the original substance, as shown by its decomposition by acids, proves that the leuco base is a diparatolyltriimidotoluene. This formula would represent the leuco base as an amido derivative of diparatolylparatolylenediamine. The diparatolylparatolylenediamine was accordingly prepared by heating hydrotolquinone with paratoluidine and zinc chloride and submitted to comparison with the leuco base. A marked similarity in appearance and properties was observable, entirely in accordance with the view that the latter is the amido derivative of the former. Moreover, the diparatolylparatolylenediamine was converted by oxidation into toluquinonediparatolylidiamine, and this substance showed a striking similarity in its appearance and in all its properties to Barsilowsky's base. There could, therefore,

be little doubt that the tolylamido groups in the leuco base and the tolylimido groups in the original compound occupy the para position to each other, and that consequently the constitution of the Barsilowsky base is that given above, whilst its leuco compound must be represented by the formula  $C_6H_2(CH_2)(NH_2)(NHC_6H_4)_2$  [1 : 4 : 3 : 6]. In agreement with this conclusion, the synthesis of Barsilowsky's base was effected by oxidation of a mixture of orthamidometaparatolylamine and paratoluidine in acetic acid solution, whilst its lower homologue was prepared in a similar manner from orthamidometaparatolylamine and aniline.

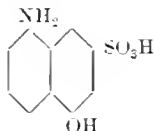
*The Colouring Matter of the Indian Dyestuff "Tesu."*  
J. J. Hummel and W. Cavallo. Proc. Chem. Soc. 1894 [132], 11.

THIS yellow dyestuff consists of the dried flowers of *Butea frondosa*. The dyeing power of the flowers as sold is comparatively slight, but is increased by boiling with diluted acid, the glucoside of the dyestuff becoming hydrolysed.

### PATENTS.

*The Manufacture and Production of New Sulpho-Acids of certain Naphthalene Derivatives and of New Colouring Matters obtainable therefrom.* J. Y. Johnson, London. From "The Badische Aniline and Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 2370, February 2, 1893.

THE substances referred to in this specification are derivatives of 1'4'-naphthylamine sulphonic acid. This yields a new  $\alpha$ -naphthylamine disulphonic acid and a new amido-naphthol sulphonic acid. The colouring matters obtained from these new substances are also described. Hitherto it has not been possible to sulphonate 1'4'-naphthylamine sulphonic acid, but by converting it first into the acetyl derivative the sulphonation proceeds easily, and the acetyl group can be readily split off subsequently. By fusing the  $\alpha$ -naphthylamine disulphonic acid with alkalis one of the sulphonic acid groups is replaced by hydroxyl, forming a new amido-naphthol sulphonic acid having the following constitution:—

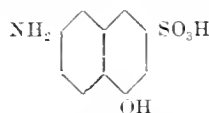


This acid is especially valuable, as it combines with diazo and tetrazo compounds to form colouring matters and can itself be diazotised, and, moreover, certain of the azo-dyes derived from it can be diazotised and combined on the fibre giving brownish-black, violet, and black shades. For preparing the acetyl derivative of 1'4'-naphthylamine sulphonic acid, about 5 kilos. of the acid are mixed with 30 kilos. of glacial acetic acid, 3 kilos. of fused sodium acetate, and 5 kilos. of acetic anhydride, and boiled under an inverted condenser for about eight hours until a test no longer yields a diazo compound with nitrous acid. The excess of acetic acid and acetic anhydride is then distilled off and the residue is sufficiently pure for further use. In order to purify it, it is dissolved in just sufficient water and precipitated with sodium sulphate, when it forms a white crystalline powder. The same compound can also be produced by sulphonating  $\alpha$ -acetonaphthalide, but the product then contains certain isomeric acids. In order to obtain the disulphonic acid the acetyl compound produced as above is mixed with 40 kilos. of fuming sulphuric acid containing about 30 per cent. of  $SO_3$ . During mixing, the temperature is kept between  $0^\circ$  and  $5^\circ$  C., and the melt is afterwards kept at about  $20^\circ$  to  $30^\circ$  C. for 12 to 15 hours, until a sample is completely soluble in cold water. The mixture is then poured on to 50 kilos. of ice and the whole boiled in order to split off the acetyl group. On cooling, a crystalline precipitate separates out, which may be purified by recrystallisation from brine. The same acid may be obtained, but

mixed with other isomers, by sulphonating  $\alpha$ -acetonaphthalide with five times its weight of the fuming sulphuric acid employed above. The compound is obtained as the acid sodium salt, and is converted into the amido-naphthol sulphonic acid as follows:—About 150 kilos. of caustic soda and 50 litres of water are heated to  $170^\circ$  C., and 50 kilos. of the acid sodium salt are added. The temperature is kept between  $160^\circ$  and  $220^\circ$  C. for about an hour, until successive tests dissolved in water and acidified with hydrochloric acid show no increase in the amount of precipitate. The melt is then allowed to cool, broken up, and added to a mixture of 450 kilos. of ice and 600 kilos. of 30 per cent. hydrochloric acid. The amido-naphthol sulphonic acid separates out as a greyish-white crystalline precipitate, which can be purified by recrystallisation as sodium salt or by dissolving it in soda and precipitating with acetic acid. It dissolves with difficulty in cold water, and on treatment with nitrous acid yields a diazo compound in orange-yellow needles. A violet-blue for un mordanted cotton is produced by preparing the tetrazo compound from 18.5 kilos. of benzidine, pouring this into an ice-cold dilute soda solution, and immediately adding a solution of 48 kilos. of the new amido-naphthol sulphonic acid. After stirring about four hours, filter-press and dry. The colouring matter thus obtained gives shades fast to washing and light and tolerably stable to acids. It can be diazotised on the fibre and combined with amines and phenols and their derivatives, giving brownish-black, violet, and black shades, some of which are very fast to washing, light, and acids. The combinations can, of course, be varied in a similar manner to the methods usually employed for the preparation of disazo dyestuffs.—T. A. L.

*The Manufacture and Production of New Azo-Dyes, and of a Component for Use in their Manufacture.* J. Y. Johnson, London. From "The Badische Aniline and Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 2614, February 6, 1893.

ARMSTRONG and WYNNE (this Journal, 1890, 800—801, and Proc. Chem. Soc. 1890, 128) describe the preparation of a  $\beta$ -naphthylamine disulphonic acid by sulphonating 2'4'-naphthylamine sulphonic acid. By fusing this disulphonic acid with caustic alkalis a new amido-naphthol mono-sulphonic acid is obtained having the formula—



About 50 kilos. of the sodium salt of the  $\beta$ -naphthylamine disulphonic acid are fused with 150 kilos. of caustic soda and 30 litres of water at about  $180^\circ$  C. for an hour, or until successive tests show no increase in the amount of precipitate when samples are dissolved in water and treated with hydrochloric acid. The melt is then worked up as in the preceding patent. The new amido-naphthol sulphonic acid forms a greyish or white crystalline powder, soluble in hot, sparingly soluble in cold water, almost insoluble in alcohol. It combines readily with diazo and tetrazo compounds to form azo dyestuffs, and certain of these can be diazotised on the fibre and combined with amines and phenols and their derivatives. The combinations of the amido-naphthol sulphonic acid with diazo and tetrazo compounds can take place in either acid or alkaline solutions, in the former case giving redder or yellower, and in the latter blue or more violet shades.—T. A. L.

*The Manufacture or Production of Derivatives of Hydroxyanthraquinones.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 2695, February 7, 1893.

THE specification describes the preparation of the  $\alpha$ - and  $\beta$ -nitro-alizarins and of their isomers and derivatives. The direct nitration of alizarin in sulphuric acid does not give a good yield of  $\beta$ -nitro-alizarin. This substance is, however,

readily formed by nitrating the boric ether of alizarin and subsequently hydrolysing the product. About 10 kilos. of crystallised boric acid are stirred into a solution of 10 kilos. of alizarin in 200 kilos. of sulphuric acid of 66° B. After cooling, 3 litres of nitric acid of 42° B. are added at about 0° C. The melt is allowed to stand several hours, and is then poured into cold water, the precipitate filtered off and dissolved in soda-lye. The alkaline solution, after having been heated to boiling, is decomposed with hydrochloric or sulphuric acid, when the  $\beta$ -nitro-alizarin separates in a pure state. In a similar manner the  $\beta$ -nitro derivatives of flavopurpurin, anthrapurpurin, and alizarin bordeaux may also be obtained. By employing the arsenic acid ethers, however (Eng. Pat. 12,580 of 1892; this Journal, 1893, 596), the nitration proceeds differently, resulting in the formation of the  $\alpha$ -nitro derivatives. About 10 kilos. of alizarin are dissolved in 200 kilos. of sulphuric acid and 10 kilos. of solid arsenic acid are stirred into the solution. After cooling, 3 litres of nitric acid are slowly added at about 0° C., allowed to stand for several hours, and then poured into cold water. The precipitate is filtered off, dissolved in soda lye, boiled, and decomposed with an excess of sulphuric acid, and the boiling is continued until the whole of the arsenic ether of the  $\alpha$ -nitro-alizarin is decomposed, which is the case when a sample dissolves in soda-lye with the characteristic violet colour of  $\alpha$ -nitro-alizarin. The precipitated dyestuff is then filtered off and washed. By applying the same method to flavopurpurin, anthrapurpurin, or to alizarin bordeaux, the corresponding  $\alpha$ -nitro derivatives of these compounds may be obtained.

—T. A. L.

*The Manufacture and Production of New Acid Colouring Matters.* J. Y. Johnsen, London. From "The Badische Anilin and Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 3487, February 16, 1893.

THIS is an extension of Eng. Pat. 4476 of 1888 (this Journal, 1889, 280) and of Eng. Pat. 10,619 of 1891 (this Journal, 1892, 514), both of which refer to colouring matters of the "Nile blue" series. The present specification describes the preparation of acid dyestuffs of the same series by condensing the nitroso derivatives of dimethyl- and diethyl-*m*-amidophenol or cresol with  $\alpha$ -naphthylamine monosulphonic acids and with certain  $\alpha$ -naphthylamine disulphonic acids. Of the latter, the 1:4:2', the 1:4:3', and the 1:2':4' appear to have been used. The dyestuffs obtained from the monosulphonic acids vary according to the conditions under which the condensation is performed. These differences are best illustrated by an example. About 70 kilos. of nitroso-diethyl-*m*-amidophenol hydrochloride, 50 kilos. of 1:3'-naphthylamine sulphonic acid, and 32 kilos. of crystallised sodium acetate are thoroughly mixed together, and 150 kilos. of 60 per cent. acetic acid are then added, and the mixture is heated on the water-bath for 12 hours or until no further formation of colouring matter takes place. The melt is then diluted with 3,000 litres of water, and sufficient soda is added to dissolve the colouring matter, filtered, salted out, and collected. The colouring matter so obtained is easily soluble in water to a blue solution and dyes wool a blue colour fast to light and alkalis. A different product is obtained by decreasing or omitting the sodium acetate and using glacial acetic acid in the above melt. These dyestuffs are referred to as type A, and differ from the previous ones, which are known as type B by their instability to alkalis. Dyes of the type A are readily soluble in the form of alkaline salts, but the solutions are precipitated by acetic acid. They dye wool greenish-blue shades, which, however, are changed to reddish-violet or red by the action of alkalis. Dyes of the type B are readily soluble in water in the form of their alkaline salts, and these solutions are not or only partially precipitated by acetic acid. As already mentioned, these dyestuffs are fast to alkalis. Certain of the  $\alpha$ -naphthylamine sulphonic acids yield both types of dyes, as for instance the 1:3' and the 1:2' acids. On the other hand, the 1:3, 1:4, and 1:4' always yield colouring matters of the type B, whilst the 1:2 and 1:1' acids give dyes of the type A only.—T. A. L.

*The Manufacture or Production of Alkylated Hydroxy-naphthalene Sulphonic Acids and Colouring Matters therefrom.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 3495, February 16, 1893.

THE 1:1'-dihydroxy-naphthalene mono- or di-sulphonic acids in the form of their basic salts can be alkylated in one hydroxyl group with the usual alkylating agents by simply heating at about 105° C. If the acid salts be employed they can be alkylated in a similar manner by adding sufficient alkali to convert them into the basic salts. One molecular proportion of the acid sodium salt of 1:1'-dihydroxy-naphthalene 3:3'-disulphonic acid (so-called chromotrope acid) is heated with lime in order to convert it into the basic salt. The solution obtained is heated with one molecular proportion of ethyl bromide for eight hours to 105° C. The resulting sodium calcium salt of the 1-ethoxy-1'-naphthol 3:3'-disulphonic acid separates out, leaving any unaltered chromotrope acid in the solution. All these alkyl derivatives are to be employed in the manufacture of azo-colouring matters, and the combinations are performed in the well-known manner.—T. A. L.

*Improvements in the Manufacture of Indigo.* H. H. Lake, London. From Kalle and Co., Biebrich-on-the-Rhine, Germany. Eng. Pat. 4257, February 27, 1893.

By heating *o*-nitro-phenyl-lactoketone with a solution of sodium bisulphite to about 50° C., it is readily dissolved and converted into a soluble product. When this compound is printed on cloth, indigo is developed on the fibre by passing it through a caustic alkali bath. About 10 kilos. of *o*-nitro-phenyl-lactoketone and 15 kilos. of a solution of sodium bisulphite containing 35 per cent. of NaHSO<sub>3</sub> are heated at 50—55° C. The ketone dissolves readily at this temperature, and on cooling the new compound separates out in needles or plates. In order to produce indigo, the cloth is printed with the compound and passed through a caustic soda-lye of 18°—25° B. The results obtained are superior to those hitherto produced with artificial indigo. The goods are evenly dyed and do not rub. (This Journal, 1893, 908 and 988—989.)—T. A. L.

*Manufacture of New Colouring Matters Derived from Dioryldiphenylmethane.* J. C. L. Durand, D. E. Huguenin, and A. J. J. d'Andiran, St. Fons, Rhone, France. Eng. Pat. 20,979, November 4, 1893. Date under International Convention, April 13, 1893.

AN extension of Eng. Pat. 9182 of 1893 (this Journal, 1893, 756). The preparation of the following colouring matters is now claimed:—1. That from 1 molecule of dihydroxydiphenylmethane on 2 molecules of diazo combinations, of which one at least is the intermediate product from 1 molecule of tetrazo-diphenyl or tetrazo-ditolyl, and 1 molecule of salicylic acid. 2. The yellow colouring matters from 1 molecule of dihydroxydiphenylmethane and 2 molecules of the intermediate compound from 1 molecule of tetrazodiphenyl or ditolyl and 1 molecule of salicylic acid. 3. The colouring matter from 1 molecule of dihydroxydiphenylmethane, combined with 1 molecule of the intermediate compound from equimolecular proportions of tetrazodiphenyl and salicylic acid, and 1 molecule of the intermediate compound from equimolecular proportions of tetrazodiphenyl and sulphanic acid. 4. The colouring matter by combining dihydroxydiphenylmethane first with 1 molecule of the intermediate compound from equimolecular proportions of tetrazodiphenyl and salicylic acid, and then with 1 molecule of a salt of diazobenzene. All these colouring matters dye unmordanted cotton yellow.—T. A. L.

*An Improvement in the Manufacture of Printing Ink.* J. and J. Bibby, Liverpool. Eng. Pat. 12,563, June 30, 1893.

FOR the preparation of black, blue, and "analogously coloured" printing inks, the inventors claim the use of

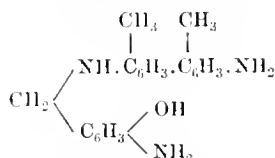


cotton-seed oil foots as a substitute for the linseed oil and the soap ordinarily employed. In this way they considerably cheapen the varnish, and for many purposes, owing to the bluish-black colour of the foots, they state that any addition of pigments becomes unnecessary. The foots are boiled with open steam, and sulphuric acid added until the whole is neutral to litmus; the fatty acids are then skimmed off and freed from water. For a common ink, 75 lb. of the foots so prepared are heated with about 38 lb. of dark rosin and 20 lb. of lamp black ground in, in the usual manner. For better-class work, 75 lb. of the foots are mixed with about 48 lb. of dark rosin and 15 lb. of cotton pitch, and a mixture of about 10 lb. of carbon black, 18 lb. of vegetable black, 10 oz. of Prussian blue,  $2\frac{1}{2}$  lb. of "imitation" Prussian blue, and 4 oz. of Indian red, ground in as before.

The inventors also claim the *partial* substitution of the usual amount of linseed varnish by the cotton-seed foots, as described.—F. H. L.

*Manufacture of New Unsymmetric Diamido Bases and of Tetrazo Colouring Matters Derived therefrom.*  
J. C. L. Durand, E. Huguenin, and A. J. J. d'Andiran,  
Bâle, Switzerland. Eng. Pat. 20,790, November 2, 1893.

This is a further extension of Eng. Pat. 12,701 of 1893 (this Journal, 1893, 824; see also Eng. Pat. 20,301 of 1893, this Journal, 1894, 32). The new base referred to is produced by heating together equimolecular proportions of formaldehyde, tolidine, and *o*-amido-phenol hydrochloride. The resulting compound is said to have the constitution—



The new base is only slightly soluble in alcohol and is insoluble in ether and benzene. The aqueous acid solutions of its salts show a green fluorescence. It begins to melt at 75° C., and decomposes about 100° C. The tetrazo compound forms brownish-yellow flakes, and the nitrite required to produce it nearly corresponds to two amido groups. If combined with naphthionic acid it produces a red colouring matter, which dyes un mordanted cotton. By substituting dianisidine for the tolidine in the above condensation a base having similar properties is produced, of which the formula is the same as above if the two  $\text{CH}_3$  groups be replaced by  $\text{OCH}_3$  groups.—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Research on the Hygroscopic Properties of Various Textile Materials.* M. T. Schloesing, jun. Bull. Soc. d'Encouragement, 1893, 718—733.

THE water retained by a hygroscopic substance, in equilibrium with the moisture of the surrounding air, depends upon the degree of saturation of the air, and the temperature, and experiments conducted without reference to these variables cannot yield precise results. In the experiments described, two methods have been employed. The first of these methods is similar to that employed by the author in a prior research upon tobacco (Comptes rend. 102). The sample of wool or silk, for instance, containing a previously ascertained proportion of moisture, which may be adjusted to the requirements of the experiment, is placed in a metal cylinder, 1.25 metres in length and 50 to 60 mm. in diameter, immersed in a water-bath, of which the temperature is kept constant to within two or three hundredths of a degree, while a slow current of air

is passed through the cylinder. An equilibrium of moisture is thus established between the air and the material at an exactly known temperature. The air thus passed through the material is received in an absorber containing concentrated sulphuric acid, and is thence aspirated to a gasometer for exact measurement. The moisture in the material is then ascertained. The bath employed accommodates four tubes, each connected with an absorber and a gasometer.

An example is given in which three samples of Indian cotton, having an average percentage of moisture of 5.75, were found to be in equilibrium of moisture with air at 31° 88', having a fraction of saturation of 0.447. In the formulae given  $f_\theta$  expresses the fraction of saturation, and—

$$f = \frac{11m}{1 + V \frac{11 - h - Ft}{p(1 + \alpha t)} 0.8047 760}$$

in which  $f$  is the tension of the vapour in the air that has passed through the material;  $f_\theta$ , the maximum tension of water vapour at  $\theta^\circ$ ;  $11m$ , the mean barometric pressure reduced to zero;  $11$ , the same at the end of the experiment;  $\theta$ , the mean temperature of the bath during the experiment;  $V$ , the volume of air aspirated into the gasometer;  $h$ , the excess of external pressure over that in the gasometer at the same moment;  $Ft$ , the maximum tension of aqueous vapour at temperature  $t$ ;  $p$ , the weight of water absorbed by the sulphuric acid;  $\alpha$ , the coefficient of dilatation of the gas. 0.8047 represents the weight of a litre of vapour at  $0^\circ$  and 760 mm. pressure. The special values for these experiments, leading to the result stated, are given.

The second method of experimenting consists in placing the material in contact with air of known content of aqueous vapour for a considerable time. This may be done in various ways, but that preferred is similar to the method employed by Regnault in verifying the hygrometric properties of air. It is based on the property of sulphuric acid, of certain dilutions, to absorb or give off moisture in proportions determinable by independent trials at given temperatures. In the instance given, a few cubic centimetres of sulphuric acid of the composition  $\text{SO}_3 + 18\text{H}_2\text{O}$  are placed in a litre flask, and over the acid are suspended the samples of material under trial, the flask being closed with a rubber stopper, and immersed in a bath kept rigorously at a constant temperature of 24° C. for five days. This method was used as a control upon the first, and the results obtained were pretty closely accordant. Three samples were tried, of American, Egyptian, and Indian cotton respectively, and yielded 10.97, 11.28, and 11.85 per cent. of moisture, the "fraction of saturation" of the air being 0.83 under the conditions stated. Series of experiments were made, of which the results are given in five pages of tables, for temperatures varying between 12° and 35°, and for different degrees of saturation of the air, on samples of the three kinds of cotton named; on double-milled wool (croisées) of Buenos Ayres; on merino wool of Port Phillip; on raw ungnunged silk, and other samples washed, of the Cévennes, and of China. The results obtained, reduced to the three temperatures of 12°, 24°, and 35°, are also shown by curves, in which the abscissæ are taken for the percentage of moisture in the material, and the ordinates for the fractions of saturation of the air in which the experiments were made. (See Figs. 1 and 2.)

It will be observed that all the curves have the same general trend. Also that the three kinds of cotton tested show marked differences. The ungnunged raw silk, owing to removal of gummy matters, comes out as less hygroscopic than the raw *fera* silk (i.e., raw silk which at most has been submitted to washing with or without soap, and to bleaching). The two specimens of wool give sensibly equal results.

The influence of temperature upon the moisture absorbed for the same fraction of saturation of the air, is but slight. This has been found to be true for other hygroscopic substances, such as tobacco, and also, according to Regnault and Sorel, solutions of sulphuric acid. It is pointed out, as

Fig. 1.

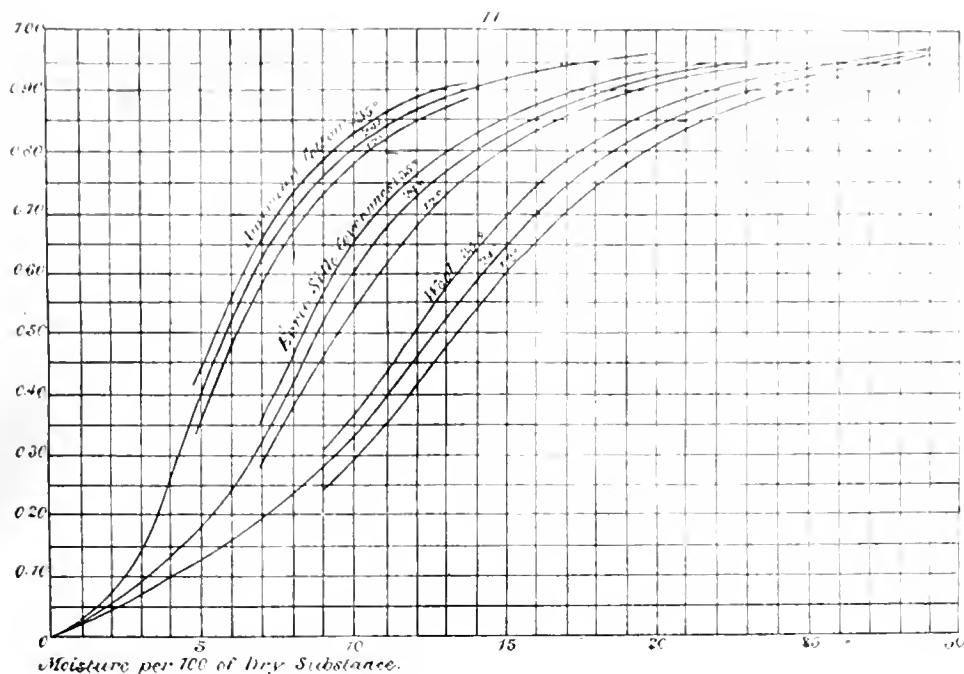
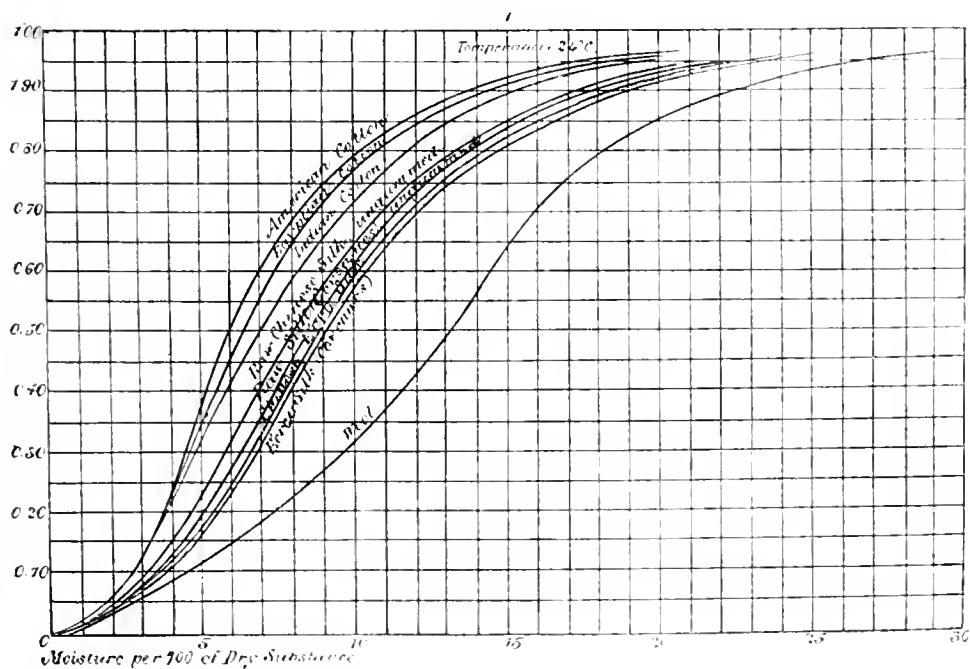


Fig. 2.



a result of these researches, that the degree of humidity of the air in which such operations as spinning and weaving are conducted, is of importance in relation to the condition of the textiles treated.—E. S.

Vegetable Silks, and particularly the Kapok of India (*Bombax pentandrum* and *Eriodendron anfractuosum*). J. J. Arnaudon. Monit. Scient. 7, 1893, 695—697.

UNDER the names vegetable silk, silk grass, silk cotton, &c. are grouped in commerce several products consisting of the bristly hairs or down surrounding the seeds of various plants. The author enumerates these plants, and states

that somewhat similar products may be obtained from poplars, willows, and certain species of acacia. Kapok is the name given to the bristly fibre which surrounds the seeds of the plants mentioned in the title. The properties of this fibre have been known since the year 1600. The trees commence to bear fruit and to produce the fibre when three years old, and they grow best at an altitude of 6,000 feet. They attain to the height of 100 feet; the first leaves are oval, and are succeeded by compound leaves of three to nine leaflets; the capsular fruit is as large as a small pear, and contains seeds the size of a small pea, and of a brown colour, surrounded by the fibrous envelope. The plant cannot thrive in a cold climate.

The best kapok is grown in Java; other qualities come from Sumatra, India, and Ceylon. It is only utilised to any extent in Australia, where 8,600 bales were imported in 1886, of which 7,991 came from Java. It is used for making wadding and hats, also for stuffs to imitate beaver, and mixed with rabbits' fur for costly fabrics, such as are made into turbans, mantles, &c. It has been tried also for dressing wounds.

This fibre may replace cotton in the manufacture of a fulminating powder, prepared by the action of nitric acid upon it. The nitrated kapok can be so treated as to yield a "vegetable silk" perhaps capable of being spun and mixed with animal silk.

The kapok thread measures 135–150 millimetres in length. Treated with alcohol and dried at 100° C, it loses 12 per cent. of its weight; of this 7 per cent. consist of substances soluble in alcohol. If the alcoholic extract be evaporated and the residue again treated with alcohol, a viscous mass is obtained consisting of oily globules mixed with a substance which yields volatile acicular crystals.

If the kapok fibre is treated with hot water it acquires an increased affinity for colouring matters. Indigo extract, azo scarlets, and bordeaux only yield very fugitive colours. Magenta dyes it better, and Bleu de Lyon, Malachite green, Metanil yellow, Phosphine, &c., give fairly fast shades.

—R. B. B.

#### *The Tendering of Cotton Fibre by Tartaric Acid.*

A. Scheurer. Bull. Soc. Ind. Mulhouse, 1893, 248.

See under VI., page 148.

### PATENTS.

*Improved Manufacture of Sheet Material, specially suitable for use as Label Cloth and Bookbinders' Cloth.* E. J. Goodwin, Manchester. Eng. Pat. 22,566, December 8, 1892.

A TEXTILE fabric or cloth, of close texture, is subjected to the action of sulphuric acid of about 115° T., so as partly to parchmetise the fibres and more or less completely close the interstices without destroying the texture of the cloth. The excess of acid is removed by washing, with or without previous treatment with alkali, and the washed material is subjected to pressure between calendering rolls, whereby a finished appearance is imparted, and the closing of the interstices completed. The material may be suitably dyed if necessary.—E. G. P. T.

*A New or Improved Method of Treating Canvas, Shirting, Silks, Paper, and like Substances for the Purpose of Rendering the same Sensitive to Light.* G. J. Junk, Berlin. Eng. Pat. 21,249, November 8, 1893.

See under XXI., page 173.

## VI.—DYEING, CALICO PRINTING, PAPER-STAINING, AND BLEACHING.

*A New Direct Grey.* Report by T. Baumann on a Paper by M. Petzold. Bull. Soc. Ind. Mulhouse, 1893, 221.

See under IV., page 143.

*The Quinone-oximes in Steam Colours.* M. Prud'homme. Bull. Soc. Ind. Mulhouse, 1893, 230–231.

DINITROSORESOREINOL, and the quinone-oximes generally, can be dyed by means of iron, copper, nickel, or cobalt mordants, i.e., by salts of metals which form more than one oxide; whereas they do not form insoluble alumina lakes. Hitherto these compounds have found no use in steam colours, the lakes with the above-mentioned salts being too quickly produced.

Solutions of potassium ferro- and ferricyanide heated to boiling with dinitrosoresoreinol, liberate hydrocyanic acid and deposit a green precipitate containing iron, but in which the ordinary tests for iron do not serve for the detection of that metal.

At the author's request, A. Scheurer has made certain experiments with steam colours. The colour consisted of fixed quantities of dinitrosoresoreinol and gum tragacanth, with varying amounts of ferro- or ferricyanide. The ferricyanide gives dark green shades, and this salt is to be preferred in practice. The ferrocyanide yields browner shades. One hour is the best time for steaming. The colour is fast to soap; the addition of calcium acetate, acetic acid, or sodium bicarbonate to the printing colour gives dull reddish shades not fast to soap.

Owing to the fact that dinitrosoresoreinol is somewhat insoluble, the colours have rather a dull and streaky appearance. Possibly this defect might be obviated by first heating together the gum and dinitrosoresoreinol, allowing to cool, and then adding the ferricyanide.—R. B. B.

#### *The Tendering of Cotton Fibre by Tartaric Acid.*

A. Scheurer. Bull. Soc. Ind. Mulhouse, 1893, 248–250.

In 1858, Grace Calvert noticed that the destructive action of organic acids on cotton is greater when dry heat is subsequently applied than when the material is steamed. (Bull. Soc. Ind. Mulhouse, 29, 208).

The author's experiments confirm this view, in so far as they show the energetic action of dry heat. A first series of experiments in which the printing colour contained—

	Grms.
Tartaric acid .....	75
Gum solution .....	925

gave no very definite results, merely a general indication that the tendering effect varies with the humidity of the air in the drying chamber. The tendering was in no case as great as was expected from previous results.

In a second series of trials the cotton, before drying or steaming, was immersed for half an hour in a boiling solution of sodium carbonate (10 grms. per litre). The tendering effect was thus greatly increased. The results of dynamometric tests are set forth in the form of a table, and the principal conclusions to be drawn are: (1) drying for quarter of an hour on a roller at a temperature of 110° weakens the fibre very little less than steaming for 1½ hours at 98°–99°; (2) drying at 40°–50° does not perceptibly tender the cotton more than drying in the cold; (3) the amount of tendering after steaming is shown to be about 25 per cent.—R. B. B.

*Tendering of Cotton Goods by the Iron Mordants used in Dyeing.* A. Scheurer. Bull. Soc. Ind. Mulhouse, 1893, 245–248.

EXPERIMENTS were made with two mordants, pyrolignite of iron alone, and pyrolignite of iron together with aluminium acetate. Dynamometric trials were made of the

resistances, after suspension for 12 hours in moist heat, followed either by a simple washing with water, or by "cleansing" with solutions of phosphate or silicate of soda or with lime. A second series of samples was prepared in the same way and then steamed for one hour. The results of the trials are given in the form of a table, and certain conclusions are deduced therefrom:—(1) the tendering does not vary directly, as the concentration of the mordant; (2) the presence of aluminium acetate has little effect; (3) the tendering effect with the iron mordant employed is approximately as follows:—

	Warp.	Weft.
	Per Cent.	Per Cent.
After fixing (ageing).....	15	15
After cleansing with silicate .....	20	50
After cleansing with phosphate.....	50	25
After cleansing with lime, at 50°.....	20	30
After cleansing with lime, boiling.....	30	40

The difference between warp and weft is explained by the greater tension of the latter, whereby a resistance may be offered to the penetration of a thick colour. The phosphate result is probably due to an error.

In another series of experiments the tendering in each case is shown to be about 25 per cent. (warp) whatever the cleansing agent employed.—R. B. B.

*The Dyeing of Indigo Blue combined with other Colouring Matters.* M. Polonovsky and J. Nitzberg. *Monit. Scient.* 7, 1893, 685—689.

Owing to the brilliancy and comparative fastness of the shades produced, indigo is preferred to all other colouring matters for dyeing blue on cotton, and this, in spite of certain defects, notably its property of "rubbing," and the fact that it is not perfectly fast to soap nor to atmospheric agencies. As the cost of indigo is somewhat great, especially for deep shades, many efforts have been made to combine this colouring matter with others less expensive, and by this means to produce equally good and fast colours at a decreased cost. The authors, who have had the opportunity of studying and applying several of these combinations, give a critical *résumé* of the devices most largely employed.

Two kinds of processes may be distinguished according as the supplementary colour is applied before ("bottoming") or after ("topping") the indigo vat. Also in one case, viz., the indigo-indophenol vat, the two colouring matters are applied in one operation. In this article the various processes are briefly described, commencing with the least rational.

1. *Topping with Magenta or Methyl Violet.*—The cotton is taken from the indigo vat, brightened with sulphuric acid, dyed in a cold solution of magenta or violet, aired, wrung out, and dried. The basic colours are very fugitive, and are removed from the fibre by washing.

2. *Bottoming with Catechu.*—The cotton is worked for one hour at 90° C. in a bath containing 3 per cent. catechu, the steam turned off, and the cotton allowed to remain in the bath over night. The next day it is taken out and worked three-quarters of an hour at 70° with 1 per cent. sodium bichromate, dried, and entered into the indigo vat. The ground of catechu is fast to washing, alkalis, and acids, but does not to any extent increase the depth of shade, and its yellow tint renders the blue dull and greenish.

3. *Bottoming with Oxide of Iron.*—The cotton is worked in ferrous sulphate solution, and then in very dilute bleaching powder solution. Ferric oxide is precipitated on to the fibre; this ground is cheaper than catechu, possesses all the defects of the latter, and is much less fast to acids.

4. *Bottoming with "Half black."*—The "half aniline black," produced as described below, has a bronze tint, but is transformed by passing through an alkaline bath into a deep blue, similar to vat blue. The alkaline bath is here replaced by the passage through the alkaline indigo vat. To dye 50 kilos. cotton: to 700 litres cold water add 1.25 kilos. aniline, 5 kilos. hydrochloric acid, 2.5 kilos. sodium bichromate, and 0.75 kilo. ferrous sulphate. Work 40 minutes in this bath, wash to remove every trace of acid, steam, and top with indigo. There is a saving of cost, and the shade is equal to pure indigo. But, like aniline black generally, it "greens" on exposure to air, is not fast to acids, and has a tendency to be uneven.

5. *Topping with Indigo Substitute.*—Indigo substitute is a preparation of logwood extract oxidised by sodium bichromate. After dyeing a medium shade in the indigo vat, the cotton is treated with dilute acid to remove lime, and washed free from acid. It is then mordanted with stannous chloride and dyed in a cold bath containing 12 kilos. of colour for 700—800 kilos. water; this bath serves for a large number of dyes, being replenished with the colour after each lot. By this treatment very dark shades are obtained at a relatively small cost. But the dyeing is apt to be uneven, the colour is very sensitive to acids, and becomes duller on washing.

6. *Topping with Benzozaurin R.*—If this direct cotton colour is used the cotton is washed on coming from the vat, and dyed in a bath containing 1.2 per cent. benzozaurin R and 4 per cent. sodium carbonate, and the shade may be rendered faster by a subsequent passage through very dilute copper sulphate solution. Good dark shades cannot be obtained by this method, and there is considerable loss of indigo in the dye and copper sulphate baths.

7. *Bottoming with Paraphenylene Blue.*—To the dye-bath heated to 60—80° C. add 1.5—2.5 per cent. paraphenylene blue and 10 per cent. salt, and work the cotton one hour. The bath is not completely exhausted, and for each subsequent lot one-third of the above weight of colour and half the weight of salt are sufficient. The colour is fixed by passing through a hot solution containing 1.5—2 per cent. potassium bichromate and 4 per cent. copper sulphate, and the cotton, after washing, is entered into the indigo vat. This ground is fast to alkalis and acids, but less fast to washing than indigo, and gives dull shades.

(8.) *Indigo-Indophenol Vats.*—The fact that indophenol can be applied by the same method as indigo, together with its less cost and its fastness, has caused some dyers to replace pure indigo vats with the mixed indigo-indophenol vats. Either hydrosulphite, ferrous sulphate, or zinc powder vats may be employed. The mixed vats as used by M. A. Vuillemin at Colmar are thus prepared:—50 kilos. indigo are ground to a paste with 15—18 kilos. indophenol and 200 litres water. The paste obtained serves for the preparation of 20 vats, each containing 700 litres water. Into a vessel of 200 litres capacity is put one-twentieth part of the paste, with 12 kilos. of lime previously slaked in 50 kilos. hot water; the mixture is stirred and allowed to settle for 24 hours.

The dye-vat is then prepared as follows:—It is filled three-quarters full of water, and the contents of the last-mentioned vessel are added, the whole well stirred, and allowed to settle for 6 hours. The vat is then ready for dyeing 25 kilos. cotton yarn. After 6 hours' rest another 25 kilos. can be dyed, and so on, until the vat is exhausted. The process with zinc powder vats is very similar.

In the opinion of the authors the economic advantages of the mixed vat are not sufficiently certain to compensate for its defects, viz., the sensitiveness of the colour to acids and to chlorine. The colour is extracted by cold alcohol.

All the processes described have certain defects. The requirements for a dyestuff to replace indigo are fastness, brightness of shade, and even dyeing, and, as indigo tends to dye a greenish-blue, the necessary addition must be a red, pink, or violet.

*Red Colouring Matters.*—The direct cotton reds are not sufficiently fast. St. Denis red is not fast to light. Alizarin

red is too costly, and too much indigo is required to cover its red colour.

*Pink Colouring Matters.*—Alizarin pink has been employed. A paste is made containing alizarin, aluminium hydrate, neutralised sulphated castor oil, calcium acetate, acetic acid, and pyrolignite of iron. The cotton is worked in a bath made up with this paste, and the bath gradually heated to 90° C. A fall fast pink is obtained, but is too costly for general use as a ground for indigo.

*Violet Colouring Matters.*—*Alizarin Violet.*—This the authors declare is by far the most satisfactory addition to vat indigo, although it is as yet little known to dyers. Great care is required in conducting the operations, as regards proportion of materials, time of dyeing, &c. One method, said to produce a shade identical with a deep vat blue, is described in detail. The cotton is first worked in a bath containing 1,600 litres water at 50° C. and 6 kilos. sumac extract. For each subsequent lot 3 kilos. extract suffice. After steaming, the cotton is passed through pyrolignite of iron at 1·4 B., again steamed, and worked three-quarters of an hour in chalk water, washed thoroughly, and is then ready for dyeing. The dye-bath contains 7 per cent. "alizarin for violet." Work three-quarters cold, raise to 70–75° C. in 2 hours; the bath is then exhausted. Rinse, air, and work in indigo vat until the desired shade is obtained. The quantity of indigo required is one-third of that necessary to give the same shade alone.

If the sumac extract is replaced by sulphated castor-oil neutralised with ammonia, the shades are more brilliant, but the cost of production is greater.

The blue "bottomed" with alizarin violet possesses all the best qualities of pure indigo blue. It is almost perfectly fast to alkalis, acids, and light. The authors state also that by adopting this process a saving of 40 per cent. of the cost may be effected.—R. B. B.

#### *Improvements in Turkey-Red Dyeing.* J. Mullerus. Chem. Zeit. 1893, 316–317 and 374–375.

THE author discusses the technical value of the processes of dyeing Turkey-red by means of soluble alizarin compounds, devised respectively by Schlieper and Baum (this Journal, 1884, 61), Baldensperger (sealed note of May 1886, opened 1891, Soc. Ind. Rouen; this Journal, 1892, 237), Erban and Specht (Ger. Pat. 54,057), and Schaeffer (Eng. Pat. 20,910 of 1891; this Journal, 1892, 237).

In the first of these, which was the earliest of the kind to be published, a bath of lime-water is employed for the conveyance of the alizarin to the alumina-mordanted cotton. The author questions the efficacy of this, seeing that it is generally held that the calcium alizarate, which is thus formed, is insoluble in water. He declares that the process, so far as he can ascertain, has not been adopted by any Turkey-red dyers other than its originators, and he is of opinion that, if it, as it has been described, forms the basis of a successful method of dyeing, an important part of it has been withheld from the published account.

The process of Baldensperger, which in order of discovery comes next, and which is probably the first in which a soluble (alkali) alizarin compound has been employed, utilises ammonia to dissolve the alizarin. The lime essential for the production of the red colour-lake is, in this process, added in the form of a soluble calcium salt (calcium acetate, for example) to the dyebath in small quantity only, to avoid precipitation. The author cannot speak favourably of the results obtained by this method.

Erban and Specht employ alkalis as solvents for the alizarin, and reverse the usual order of the principal dyeing operations (namely, first mordanting and then dyeing), making the application of the dye precede that of the mordant. Their process consists in passing the cotton to be dyed through an alkaline solution of alizarin, drying, passing through a bath of a mordanting salt, again drying, and finally completing the combination of the mordant and

colouring matter upon the fibre by steaming. Thus, for example, the cotton is taken through a bath of—

500 grms. of alizarin,  
2 litres of water,  
125 cc. of ammonia solution,  
240 cc. of Turkey-red oil (75–80 per cent.), and  
8 grms. of sodium stannate,

dried in an ageing room (*i.e.*, by hot air), padded in a bath of—

3,100 cc. of water,  
550 cc. of aluminium acetate at 16° B., and  
85 cc. of calcium acetate at 21° B.,

again dried, steamed for two hours (one hour under one atmosphere's and one under 1½–2 atmospheres' pressure), and, finally, soaped and washed. Following this process the author succeeded in dyeing short fents tolerably well, but, on a larger scale, entirely failed to get a passable colour, this being browner, duller, looser, and more uneven than that obtained on the smaller scale. Using aluminium "bi-sulphite" instead of aluminium acetate in the mordanting bath a rather better colour was produced.

Excepting that the solution of the alizarin is effected with borax instead of with ammonia, the process of Schaeffer appears to be identical with that last described. A slightly richer and brighter red than is obtained by that method is, however, producible by it.

Experiments were further made with a sodium carbonate solution of alizarin, and with an ammoniacal solution of the same dye, to which sodium aluminate was added, but the results were in both cases more or less unsatisfactory.

The author concludes that it is impossible to obtain by the above processes a colour which can compete with the red which is ordinarily dyed. The processes may find advantageous employment in the dyeing of cloth which is to be subsequently printed dark colours or black. But probably the only application of any importance which can be made of them, is in the dyeing of fabrics for discharge printing, the red colour-lake in the partially-formed state in which it exists before steaming being discharged with especial ease. Borax and soda are to be preferred to ammoniacal solvents for the alizarin in the dyeing of the red for this purpose.

Incidentally it is mentioned that very bright and fast alizarin pinks and other colours may be produced by Erban and Specht's method, and that the proprietors of one of the German dye-manufacturing concerns have considered the process sufficiently valuable to acquire the patent rights of it.—E. B.

#### *Improvements in Turkey-Red Dyeing.* Erban and Specht. Chem. Zeit. 1893, 542–543.

IN reference to the preceding paper of Mullerus' (see preceding abstract), it is pointed out that the present authors have patented the employment, in their process of dyeing, of the following compounds as solvents of the alizarin:—Ammonia, ammonium carbonate, alkaline hydroxides, and carbonates; alkali salts having an alkaline reaction, such as sodium phosphate, borax, sodium silicate, sodium aluminate, soaps, Turkey-red oils, &c.; and organic bases (amines). It will thus be seen that the process of Schaeffer, to which allusion is made in the paper in question, is closely related to that of the authors.

Mullerus' assertion that he was unable with the authors' process to obtain on a larger scale results equal to those he obtained on a small scale, is severely criticised.—E. B.

#### PATENTS.

##### *Improvements in or relating to Calico Printing.* L. Lang, Manchester. Eng. Pat. 2009, January 30, 1893.

THE object of this patent is to produce coloured designs with a less number of engraved rollers than heretofore, and with a better effect. One roller carries the outline of the subject, and on this roller the shades and shadows are also produced by stippling or line engraving. The outline and the shades are thus printed at one impression, and the

various colours are afterwards applied by separate rollers, which are only engraved just sufficiently to receive the colour and impart it to the fabric.

In producing a coloured design according to this invention, it is necessary that the colour of the outline and the first colour afterwards applied should match, e.g., black outline and slate colour to follow, or brown outline and chocolate to follow.—R. B. B.

*Improvements in the Process of Dyeing.* O. P. Amend, New York, U.S.A. Eng. Pat. 18,232, September 28, 1893. (Under Internat. Convention.)

The fibre is treated with a cold solution of chromic acid together with some other acid which must be of a non-oxidising character. The product is described as an insoluble chromate of the fibre. It is further subjected to the action of a reducing agent in solution, e.g., ferrous sulphate, cuprous chloride, or a bisulphite. The prepared fibre may be finally dyed by any well-known process.

The invention is described as that of the production of "a fast mordant on fibrous substances."—R. B. B.

*Improvements in and connected with Varnishing in Colours.* J. Perl, Berlin, Germany. Eng. Pat. 21,455, November 10, 1893.

See under XIII. B., page 164.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Oxidation of Nickel Sulphide.* P. de Clermont. Comptes rend. 1893, 117, 229—231.

When recently-precipitated nickel sulphide which has been well washed with hot water is exposed in a thin layer on filter-paper to the air, it gradually loses its moisture and at the end of 24—48 hours changes colour from black to apple-green. The product is detached from the filter, extracted with boiling water until nothing more is taken up, and the insoluble residue treated with dilute hydrochloric acid, which leaves a residue of sulphur undissolved. The hydrochloric acid solution contains the basic sulphate,  $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ , the constitution of which has been determined by Fremy, and which has been described by Tapputi, Berzelius, and Habermann. The green aqueous solution contains principally the basic sulphate, together with the normal sulphate and a little free sulphur.

When nickel sulphide is covered with a layer of water the oxidation extends over several months, and more normal sulphate and less basic sulphate are produced than in the case last cited.

In conclusion, the author states that nickel sulphide cannot be completely oxidised in the air, but the residual sulphide enters into a stable combination with the normal sulphate.—A. R. L.

*Liquid Chlorine.* M. Fribourg. Bull. Soc. d'Encouragement, 1893, 706—707.

Liquid chlorine is prepared on an industrial scale at M. Pechiney's works at Salindres, where, by means of a pump, the body of which is formed as a U-tube, in cast-iron lined with lead, having sulphuric acid and petroleum respectively in its two limbs, acting as pistons, the gas is compressed into iron or steel cylinders, each weighing about 100 kilos., and capable of containing 50 kilos. of the liquid chlorine. Two bronze stopcocks are provided to each cylinder, one serving for delivery of the gas, and the other, connected to a tube reaching to the bottom of the vessel, for charging, and also for discharging if required, the liquid chlorine. It is stated that neither iron, copper, nor lead are attacked by dry chlorine, and that vessels for containing the liquid have suffered no deterioration after two years' use.

The density of liquid chlorine is about 1.33, and 50 kilos. give nearly 15,000 litres of the gas. The pressure of the liquid at 35° is 10 atmospheres, and the containing cylinders are tested up to a pressure of 100 atmospheres. The author describes a vessel for containing liquid chlorine, suited for laboratory use, consisting of a wrought iron bottle, with double stopcock, from which the gas or the liquid may be obtained at pleasure, means being provided for connecting one of the stopcocks to a tube conveying the gas. Reference is made to Faraday's original experiment in liquefying chlorine; and the advantages of liquid chlorine over bleaching powders and liquids, as to convenience in application and facility in transport, are stated.—E. S.

*The Separation of Volatile Fatty Acids.* M. Wechsler, Monath. f. Chem. 14, 162.

See under XXIII., page 178.

## PATENTS.

*Improvements in Electrolytic Apparatus.* F. Andrioli, Brixton, Surrey. Eng. Pat. 1661, January 25, 1893.

See under XI., page 160.

*Improvements in or applicable to Brine evaporating Pans.* J. Parks, Northwich. Eng. Pat. 2400, February 3, 1893.

In this invention brine is evaporated by means of steam instead of heating the pan direct by fire, and with a view to economy of fuel a second pan is built directly over the first and heated by the steam issuing from the lower.

The accompanying illustrations show the construction of the pans in longitudinal and cross sections.

*a a* is a vessel containing water only, heated direct for the purpose of raising steam which is to boil the brine contained in the pan above. The vessel is built over a fire-grate and flues, and is provided with water pockets for increasing the heating surface. The issuing steam heats the brine pan *e e*, above which the second brine pan *f f* is situated. The two pans are separated by a diaphragm *g*.

Fig. 1.

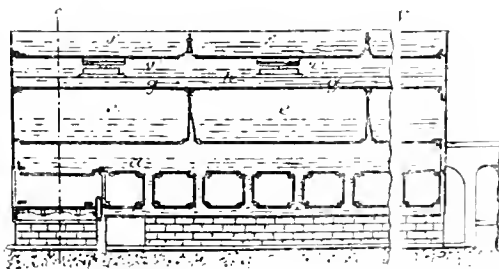
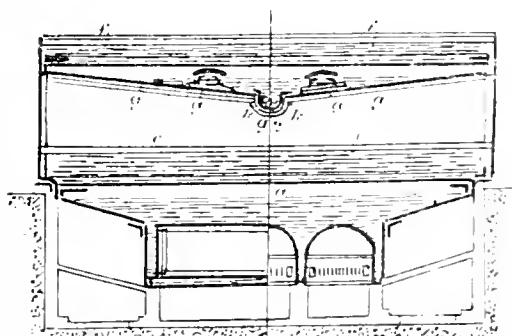


Fig. 2.



which is deeper along the middle than on the sides, and is provided with passages or shafts,  $g^1 g^1$ , through which the steam from the lower pan ascends.

The condensed water is collected in the central channel  $g^2$ , and is used for feeding the lower evaporating vessel with pure condensed water. Through the channel  $g^2$  are also led the brine inlet pipes K, whereby the brine is heated before reaching the evaporating pans.

In order to facilitate the removal of the salt without the loss of the whole of the steam between the two brine pans, vertical partitions are introduced in the pans, which cut them up into sections, any one of which can be emptied without interfering with the others, and suitable means are provided for regulating the brine, the water, and the issuing steam.—B.

*Improvements in Apparatus used in the Concentration of Sulphuric Acid.* I. Levinstein, Manchester. Eng. Pat. 2476, February 4, 1893.

APPARATUS for the concentration of sulphuric acid, consisting of a descending series of glass vessels exposed to external heat, in which the acid is led from the spout of one vessel into the bottom of the next lower vessel through a glass tube, is described in Eng. Pat. 19,213, 1892 (this Journal, 1893, 156); but the inventor has found that these glass tubes are liable to displacement, in which case the lighter acid flows on to the surface of the acid in the next lower vessel, impairing the working of the process, besides having other inconveniences, sometimes involving the loss of the vessels. Such defects and losses are avoided by flattening the upper end of the glass tube, and so cutting away or notching one of the flattened sides that the cut-away part may fit closely against the upper vessel immediately below the spout. Or the upper end of the tube may be moulded to fit as described. A portion may be cut away sideways from the lower end of the glass tube, so that clogging may not take place.

The glass vessels are shown resting in metal basins having opposite vertical wings cast to each basin, to form shields or defences against the eddying hot gases that might otherwise come into contact with them and occasion breakage.—E. S.

*Improvements in the Method of and in Apparatus for Recovering and Concentrating Waste and Diluted Sulphuric Acid, and for Separating and Recovering other Acids in Mixture therewith.* I. Levinstein, Manchester. Eng. Pat. 2835, February 9, 1893.

THE method of concentration employed is that described in Patent No. 19,213, 1892 (this Journal, 1893, 156), according to which a series of glass vessels receives the acid to be concentrated from the "receiving tank," into which it is run from a large lead or stone tank. The glass vessels, on successively lower levels, are fed one from the other by tubes (see abstract of Patent 2476, 1893, in this Journal), and are contained within an oven, the trunk of which, according to the present invention, is formed at its end into two branches, one leading to a lead tower, and the other to an earthenware tower, both towers being filled with coke or the like, and connected to a chimney or draught-producing device. When the sulphuric acid to be concentrated contains nitrous acid, the damper in the branch leading to the earthenware tower is opened, the other branch being closed, and water is caused to trickle down the tower. The nitrous acid in the vapours is recovered as nitric acid, and may be pumped up to the top of the tower for use instead of water. When the concentration is carried out by two stages, the nitrous acid is removed and recovered as nitric acid in the first stage; but during the second concentration the vapours are led through the second branch to the lead tower. The arrangements admit of modifications according to circumstances. Thus, two concentrating ovens may be used, one connected to an earthenware tower, and the other to a lead tower. When sulphuric acid works are near, the earthenware tower may be dispensed with, the branch or trunk being put into communication with the Glover tower of such works.—E. S.

*Improvements in and relating to the Manufacture of Paints, Sulphuric and Sulphurous Acid from Protosulphate of Iron.* M. N. D'Andria, Stretford, Lancaster. Eng. Pat. 4342, February 28, 1893.

See under XIII., page 163.

*Improvements in the Method of and Apparatus for Manufacturing Cyanides, Ammonia, and Fuel Gas.* A. J. Boulton, London. From E. Cowen, New York, U.S.A. Eng. Pat. 13,069, July 4, 1893.

See under II., page 140.

*Improvements in the Manufacture of Ammonia, Sodium Hydroxide, and Chlorine, and in Apparatus therefor.* C. Kellner, Vienna. Eng. Pat. 13,722, July 14, 1893.

SODIUM chloride solution is pumped from a reservoir into a closed vessel containing a horizontally arranged series of carbon or platinum plates serving as anode to a source of electricity, while mercury at the bottom of the vessel serves as cathode. As the sodium chloride is electrolysed, an amalgam of sodium is formed, and chlorine is set free, which escapes by a tube in the cover and is conducted away for use. As the bottom of the vessel is depressed towards the centre, the amalgam flows by gravity through an aperture into a funnel tube, which conveys it on to the inclined bottom of a second vessel receiving sodium nitrate solution from an elevated reservoir. The inclined bottom is lined with corrugated iron, above which is arranged a wire grating, gilt, silvered, or platinum-coated, serving as one electrode, the amalgam being the other electrode; the electric energy developed by the pair is conducted away, preferably for relieving the electric generator connected to the first vessel. The sodium amalgam decomposes the sodium nitrate solution with formation of ammonia (which is conducted away and collected) and of sodium hydroxide. The mercury, now freed from sodium, issues from the lower end of the bottom to be passed through a cooling coil, and to be returned by a force pump to the electrolytical decomposing vessel, which it enters near the periphery of the depressed part, and follows a spiral path by a channel formed for the purpose so as to attain a whirling motion, under which conditions it becomes recharged with sodium, and the process goes on continuously until the sodium nitrate is decomposed. Provision is made for returning the sodium chloride solution for enrichment to its reservoir. It is an important condition in the apparatus that the carbon anode should present a much larger area than the mercury to which it is opposed.—E. S.

*Apparatus for the Electrolytic Production of Bleaching Liquid.* C. Kellner, Vienna, Austria. Eng. Pat. 13,723, July 14, 1893.

See under XI., page 161.

*A Method of and Apparatus for Effecting the Absorption of Carbonic Acid.* A. Müller, Nieder-Schönweide, near Berlin. Eng. Pat. 13,928, July 18, 1893.

THE gases containing carbonic acid, such as arise from the combustion of fuel or from roasting, are led to a vacant space at the bottom of an air-tight sheet-iron casing, within which a number of equidistant vertical nettings, preferably of iron wire, are placed. The netting is woven so close that it may contain, for instance, 49 small square holes per square inch. The sheets of netting are separated by a series of slats, beside which the upward current of gases has to pass, so as to bring them into strong frictional contact with the surfaces of the nettings. The alkaline liquid acting as an absorber is forced by a pump into a tube having branches communicating through small perforated pipes with spaces between clamps securing the upper part of the nettings, whereby it becomes distributed over the whole length of all the nettings in such manner that every little square is occupied by a film of the liquid. Thus the passage of the alkaline liquid is opposite in



direction to the flow of the gases. There is a pipe in the bottom of the casing to conduct away the liquid as it drips from the nettings. The spent gases, practically freed from carbonic acid, are led off from the top of the casing by connection to a chimney or other draught producer. It is stated that in this apparatus the liquid is discharged after it has been in contact with the gases for four minutes. Details of the devices used in securing the nettings and of other arrangements are given.—E. S.

*A New Cleansing Composition or Powder.* S. E. Mott, G. W. Wort, S. Wort, and E. W. Wort, London. Eng. Pat. 18,169, September 27, 1893.

The cleansing powder is composed of soda-ash and finely-ground soda crystals in various proportions.—K. E. M.

*Improvements in Apparatus for the Electrolytical Decomposition of Salt Solutions.* C. Kellner, Vienna, Austria. Eng. Pat. 19,542, October 17, 1893.

See under XI., page 162.

*A New Process for Reducing Sulphuretted Ore into Oxides and Sulphur.* L. Bemelmans, Brussels. Eng. Pat. 19,606, October 18, 1893.

The finely-crushed ores are treated in a modified Hasenclever furnace, which is divided into sections by vertical partition walls. They are at first exposed to the action of sulphurous vapours produced at the earlier stages of the process, by which means arsenic, antimony, and phosphorus are eliminated if present, and some sulphur is given off. They then pass on to the roasting zone proper, where they meet with highly-heated air, free from oxygen, and finally they descend to the hottest oxidating zone, from whence they are discharged in a completely oxidised state. A certain proportion of non-sulphuretted ore may be used if coal is added in certain proportions. The heated air necessary for the process is supplied alternately from the two chambers of a recuperator, the free chamber of which is re-heated by the combustion of some gaseous fuel, so that neither the oven nor the ore require any other heating.

The gases from the upper or disillating zone consist of nitrogen, sulphurous acid, and vapour of sulphur, together with carbonic acid and the sulphurets of arsenic and antimony in some instances. They are injected, together with hot producer gas and steam, into a reducing apparatus or recuperator, by which the sulphurous acid and free sulphur are converted into sulphuretted hydrogen. This is then cooled down, dried, and mixed with something less than half its volume of sulphurous acid, which also has been dried when necessary, but is preferably taken from the ore-roaster at a lower level. The mixture is led through heated flues, and finally injected into a condenser by means of steam jets, where sulphur separates in a solid or liquid form and water-vapour passes off.—J. H. C.

## VIII.—GLASS, POTTERY, AND ENAMELS.

*Chemical Considerations on the Pottery Industries of the United States.* K. Laogenbeck, Amer. Chem. Soc. 15, 651—655 and 695—703.

THE pottery industries of the United States are conducted on an empirical basis; the potters, on the one hand, are generally ignorant of chemistry, and the chemists, on the other, seldom have opportunities of working in or having access to the manufactories. Thus the chemists, as a rule,

have no knowledge of the potters' needs, and the collecting and analysing of thousands of new varieties of clay, which is done every year under the United States Geological Survey department, bears no industrial fruit as far as the potters' industry is concerned. The present papers were written with a view to give such a description of the chief branches of the American pottery industry as would show the chemists how to make the work of the Geological Survey available for this industry.

The most important products of the pottery industry in America are fire-clay goods, terra cotta, red ware, yellow ware, stone ware, cream-coloured ware, and white granite ware. The first two being unglazed wares, the manufacturer is less dependent upon the aid of chemistry, and they are not dealt with by the author.

*Red ware* is ordinarily formed from the same materials as are used for making bricks, and is used for door knobs, milk crocks, cooking vessels, &c. There is a practice, much to be reprehended, of using litharge or galena alone for producing the glaze, which takes up alumina and silica from the ware. The glaze thus formed is basic on the surface and hence is liable to be attacked by the acids present in cooking operations. A usual glaze is one having the following composition: 1.0 PbO, 0.09 Al<sub>2</sub>O<sub>3</sub>, 0.03 Fe<sub>2</sub>O<sub>3</sub>, 1.4 SiO<sub>2</sub>. In order to be safe against chemical action when the vessels are used for cooking, it would be better to use a glaze containing a higher proportion of acid. In burning this ware an oxidising fire must be maintained throughout to ensure a bright red colour and prevent reduction of the lead in the glaze.

*Yellow ware* is a higher class ware, and requires a higher temperature for burning. Accordingly the glaze is applied to once baked (or biscuit) pieces, and is then finished in a second fire less hot than the first. The material used belongs to the class known commercially as second-class fire clays, such as the "buff" or "blue" clays of the coal measures. The following is the composition of a typical clay of this kind, as expressed in a "rational analysis":—Clay substance, 76.05 per cent.; quartz, 19.54 per cent.; feldspathic detritus, 4.28 per cent. The material is prepared more carefully than that of the "red ware" potter, being "slipped" in a vat with mechanical plungers, then passed through a 60-mesh wire sieve or a No. 8 silk lawn, and brought to the condition of a plastic clay by evaporation or by a filter press. A class of clays known as "flint clays," which occur in rocky masses having a conchoidal fracture, are perfectly suitable in composition for making yellow ware, but are not used for the purpose owing to their intractable nature. The machinery required for wet-grinding the flint clays would not be specially expensive, but the author is of opinion that in so conservative an industry the introduction of such machinery would meet with great opposition. If a chemist wishes to introduce a new clay into the pottery industry he must be able to inform the manufacturer at what degree of heat the clay will attain "good biscuit" condition, in order that the potter may know if he can put it in the kiln along with his old ware. For the purpose of these tests a *Seger pyrometric cone* is used. These cones are made of orthoclase, marble dust, quartz, and kaolinite, and are numbered 1. to VIII. according to the proportion of the different materials employed, which determines the temperatures they will resist before "crooking over" and melting down. A common glaze for yellow ware is 1.0 PbO, 0.2 Al<sub>2</sub>O<sub>3</sub>, 2.0 SiO<sub>2</sub>. "*Rockingham ware*" differs from yellow ware only in being covered with a brown manganiferous glaze, e.g., 0.85 PbO, 0.15 MnO, 0.18 Al<sub>2</sub>O<sub>3</sub>, 1.8 SiO<sub>2</sub>. It is used for tea and coffee pots, pitchers, &c. When experimenting with clay to ascertain its uses in pottery, the chemist may make the clay into rings, which are set upright in the furnace, a little "slip" being used to make them stick. These rings can be withdrawn from time to time with an iron wire, and the action of the fire can be ascertained, at the same time that the temperature is measured by the Seger pyrometric cones. The fired pieces of clay are then removed from the muffle, covered on one side with the glaze, and baked a second time to melt the glaze, the temperature in this case being noted by the melting of a gold-silver alloy. The tint of the clay under

the glaze, and the behaviour of the glaze upon the clay, should be carefully noted and accurately described for the information of the potter.—V. C.

#### PATENTS.

*Artificial Stone, Bricks, Tiles, and the like.* J. Gaskell, Rutherglen, and W. Robinson, Windle. Eng. Pat. 22,784, December 12, 1892.

See under IX., page 154.

*A Method of Lining Spigot and Socket, Flange, or Saug Pipes with Enamel, Glass, Composition, or other Material.* F. Herbert, Birtley, near Newcastle. Eng. Pat. 22,682, December 13, 1892.

The pipes, having been moulded or cast in the usual manner, are cleaned from sand, &c., the cores are removed, and the pipes are placed upon a "gantry" and caused to revolve in a furnace. A perforated tube, containing the enamel or glaze, is passed through the interior of the pipe and made to revolve, thereby distributing the enamel or glaze evenly upon the interior surface of the pipe. The heat of the furnace causes the enamel to melt and fix itself upon the surface of the pipe.—V. C.

*Improved Means for Producing Crucibles or Melting Pots.* E. J. T. Digby, Liverpool. Eng. Pat. 3661, February 18, 1893.

See under X., page 158.

*Improvements in Chamber-Kilns.* F. H. Jung, Salisbury. Eng. Pat. 18,829, October 7, 1893.

This invention has for its object the production of an intense heat with a small amount of expense and labour, the rapid drying of the bricks without discolouring them, and increasing the output of the kiln. These objects are attained by the arrangement of the heating flues and their accessories.—R. B. P.

*New or Improved Process of Etching on Glass, Porcelain, and the like.* J. Retzlaff, Steglitz, Prussia. Eng. Pat. 20,533, October 31, 1893.

In the usual method of etching upon glass surfaces, a layer of tin foil is affixed by means of paste, the pattern is then cut out in the tin foil, the exposed paste removed by means of turpentine, and the exposed surface of glass is then acted upon in the well-known manner. The process of cutting out the pattern from the tin foil is slow, requiring care and skilled workmanship. In the new process this operation of mechanically cutting out the pattern is replaced by a chemical action. Thus, in order to etch a design in relief, the method would be to transfer the design on to the tin foil in a grease pigment and then to eat away the tin foil round the greased portions by means of an acid. The tin foil in this process is attached to the glass surface by a layer of asphalt. Besides giving perfectly sharp contours to the pattern, the new process has the advantage of being specially adapted for producing large quantities.—V. C.

*Improvements in the Manufacture of Bricks, Terra Cottas, and the like.* C. Schlickeyen, Berlin. Eng. Pat. 22,280, November 21, 1893.

See under IX., page 154.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### PATENTS.

*Artificial Stone, Bricks, Tiles, and the like.* J. Gaskell, Rutherglen, and W. Robinson, Windle. Eng. Pat. 22,784, December 12, 1892.

The above are made from the waste sand, glass, and sulphate of iron produced in glass grinding and polishing, with or without the addition of a flux. If the moistened material be pressed hard, solid bricks can be produced by heating without the addition of extraneous material. On the other hand, various fluxes may be added according to the class of article required. Barytes, sulphate of lime, granite, and clay are the materials most commonly used for mixing with the waste material.—V. C.

*Improvements in the Manufacture of Bricks, Terra Cottas, and the like.* C. Schlickeyen, Berlin. Eng. Pat. 22,280, November 21, 1893.

In the manufacture of bricks, facing blocks, terra cottas, and the like, it has hitherto been almost impossible to prevent the appearance of white or yellow spots. Sometimes after burning it is found that the facing surface is so much discoloured that the block cannot be used unless it be first chemically treated, at considerable expense, or coated with colour.

The present invention prevents the formation of these spots by treatment during drying, that is to say, after the objects have been moulded and before they have been put into the kiln. On leaving the press mould the brick is cut at about  $\frac{3}{8}$  in. from the bottom, so that the slab, which has been nearly cut off adheres slightly to the brick. The brick is placed upon a drying board, which must be absolutely clean, and is preferably covered with paper free from salt or acids. The surface of the brick which is downwards is the facing surface. In the drying process the upper layers are more exposed to the action of the air, and hence the moisture, with the salts, &c., is drawn up from the lower layers. Before the article is placed in the kiln it is freed from the slightly adhering upper slab, which now contains the salts.

It is not absolutely essential to cut the brick and subsequently remove the slab as here described, as by the above process of drying, all the salts, &c. collect in the upper portion (which will be the back of the brick), leaving the facing surface free from anything which can cause a spot or stain.—V. C.

*Certain Improvements in the Preservation of Wood.* A. A. Hely, Westminster, Middlesex. Eng. Pat. 3356, December 16, 1893.

The wood is impregnated through its pores, under any well-known process, first with a strong solution of calcium bisulphite and then with a corresponding solution of caustic lime. A monosulphite is formed, which is subsequently oxidised by the action of the air to calcium sulphate, and becomes practically part of the ligneous structure.

—N. H. J. M.

## X.—METALLURGY.

*Researches on the Properties of Alloys.* W. C. Roberts-Austen. Second Rep. to Alloys Research Com. Inst. Mech. Eng. 1893.

The main object in view was to extend a research the author had previously made upon the application of the "periodic law" of Newlands and Mendeleeff to the mechanical properties of metals. The law, as originally expressed,

states that the properties of the elements are a periodic function of their atomic weights." It had already been shown that the effect of impurities added to gold is nearly proportional to their atomic volumes, the larger the volume of the atom the greater being its effect; and the question was, does this hold good for other metals?

It was evident that there is a direct connection between the properties of a mass and the volume of its atoms. In all probability, therefore, the introduction of free molecules of an added element must create a disturbance, the nature and magnitude of which will bear some relation to the volume of the disturbing atoms. In the present inquiry, which is conducted essentially from the engineer's point of view, great prominence is given to mechanical tests, upon the results of which the employment of materials depends. A series of experiments was made on copper, showing the influence of arsenic, antimony, and bismuth on that metal.

The passage of iron from one allotropic modification to another is accompanied not only by a change of heat capacity, but also by a change of volume. When a mass of iron cools down from a temperature of 1,000° C., or 1,800° F., it also contracts, until at the point of recalescence it suddenly expands; after which it again contracts, as it cools to atmospheric temperature. An experiment described in the previous report indicated the probability that pressure lowers the critical points of iron and steel. By compressing a piece of steel in a hydraulic press, it should be possible to obtain recalescence at a lower temperature than would be the case if the pressure were not applied. Observations were made by the eye in order to see whether such a lowering of the recalescence point could be detected in a piece of compressed steel; but it was found that lateral flow of the test piece rendered the experiment difficult, by preventing the application of a sufficient stress until after the metal had passed through its allotropic change, and had become more or less rigid. Moreover, the orifice in which the thermo-junction was placed in the specimen became obliterated by the lateral flow of the metal. One such experiment, however, showed that when the mass was heated to 1,000° C. without pressure, that recalescence occurred at 650° C., but at 620° C. with a pressure equivalent to nine tons per square inch.

Three sets of experiments in all were made, and from a study of these it was evident that the recalescence point is lowered by pressure; but it was found that this lowering is not effected unless the load is applied at a temperature well above that at which recalescence takes place.

It has long been known that when a triple alloy of bismuth, lead, and tin, which bears Newton's name, is cooled down from the molten state, there occurs, long after the metallic mass has solidified, a remarkable rise in its temperature. Experiments made show that this evolution of heat occurs generally at 46° C., or 115° F. An evolution of heat also takes place if the solid mass is cooled rapidly by quenching in water. This effect, however, disappears after the third or fourth heating, but may be restored by remelting the mass, or by raising it to a temperature just below its melting point. A suitable alloy for the experiment is the one containing 50 per cent. bismuth, 31½ per cent. lead, and 18½ per cent. tin. The appearance of the fractured surface of the mass, if broken before the evolution of heat, is plate-like, crystalline, and almost vitreous, but after the thermal change the fracture is grey, dull, and finely grained. It will be evident that the changes this alloy undergoes, present certain analogies to those which take place in steel during the hardening by rapid cooling from a high temperature, and when the metal cools slowly from bright redness.

It became interesting in the investigation to see whether the application of pressure would cause the change to take place at a lower temperature than that at which it would occur without pressure.

The result of the experiment showed that under a pressure of one ton per square inch the temperature at which the thermal change took place was lowered as much as 4° C., or 7° F., that a pressure of about four tons per square inch was sufficient to obliterate the thermal disturbance altogether. The effect of pressure in lowering the critical point in this alloy is evident, and it was interesting to ascertain whether

the compression of the mass had really prevented the alloy from undergoing the molecular change from the vitreous to the grey variety, or whether the recalescence had been so gradual that there was no sudden augmentation of temperature in the cooling mass, such as would be indicated by a marked discontinuity in the autographic curve. The die was accordingly made in halves, so that it could be rapidly opened when the pressure was released. The solid cylinder of compressed alloy was removed and fractured with as little delay as possible, but it was found that the change, though not complete, was far advanced. It is impossible to say whether the change occurs directly the pressure is removed, or during the time it has been applied.

It may be asked, what evidence has been gathered as to the mode of action of added elements? And does it appear that the atomic volume of the added element has a dominating influence on the mechanical properties of the mass in which it is hidden? The true action of an added element may readily be masked by its action as a de-oxidiser. Notwithstanding this, it is undoubtedly proved that bismuth, potassium, and tellurium—all of which have large atomic volumes—greatly lower the tenacity of copper. Arsenic, which has a larger atomic volume (13·2) than copper (7·1), confers strength on copper; but it is very certain that the limit of elasticity and the ductility of a metal are greatly influenced by the presence of such of the elements with large atomic volumes as have thus far been studied; and this fact may be of more molecular significance than the diminution of tenacity, to which, for the sake of simplicity, attention was mainly directed when the early experiments on gold were made.

The very important question now arises, may the effect of an added element mainly depend on its power of forming either a fusible or an infusible compound with the mass in which it is hidden? This is the view held by le Chatelier, who also suggests that the deleterious action of an element is due to its forming a fusible compound with the mass, whilst, on the other hand, the presence of an element which forms an infusible compound with the mass promotes the formation of a fine grain and gives strength. The whole tendency of recent work is to show that when small quantities of elements are added to a metallic mass, they behave as a gas would, and remain free. It remains to be seen at what point in the cooling of a highly heated fused mass of a metal forming a solvent an impurity would enter into combination with a portion of the mass, and what amount of impurity of a given kind has to be present in order that it may lose its individuality.

With regard to the important molecular change which may take place in a solid metal or alloy, and of which the recalescence of steel is the most striking example, it is no small gain to have ascertained that the temperatures at which such changes occur are altered by pressure; and this seems to be a conclusion which it is safe to deduce from the experiments given in the present report.

The profound change which is produced in the properties of metals by alloying them is well shown by some recent experiments by Professors Dewar and Fleming. They have examined the effect of very low temperatures on the electrical resistance of pure metals and alloys, and the results tend to prove that, if pure metals could be reduced to the absolute zero of temperature, they would offer no resistance to the passage of an electric current. This is true of soft, pliable metals like gold, and rigid ones, such as nickel. Impure metals and alloys behave very differently: the diminution of resistance by exposure to cold is very marked, but the direction of the curves representing resistance and temperature appears to indicate that the resistance at the absolute zero of temperature would still be considerable.

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*The Chlorination of Gold Ores.* L. D. Goddall. Eng. and Mining J. 1894, 5.

THE questions to be determined in the laboratory for the treatment of an ore in the raw state are the amount of chemicals required, the time of exposure to chlorine necessary, and the fineness to which the ore must be crushed to obtain the best results.

It is very difficult to determine in the laboratory the exact amount of chemicals required in the mill; however, by making a series of comparative tests on the same sample before and after roasting, some idea may be obtained as to the relative consumption of chlorine or bromine in the raw and roasted samples.

The degree of fineness required in the crushing should be very accurately determined by making a series of experiments on 20, 40, 60, 80, and 100 mesh samples of the same ore.

*Treatment of Roasted Ore.*—The laboratory work previous to the treatment of an ore requiring roasting should be very thorough. A good plan is to make a preliminary test as follows:—A sample, carefully taken, is passed through an 80-mesh screen and assayed; 150 grms. of the ore are then roasted in the muffle in two 4-in. roasting dishes, each containing 75 grms. of ore, using the same temperature and time for each. The temperature should be the same as in ordinary oxidising roasting, starting with a dark red heat and gradually increasing to a light red at the end of the operation. From one to two hours is sufficient for most ores if properly stirred. After the roasting is completed the ore is allowed to cool, is weighed, and the loss in weight noted. The sample is now again assayed to determine the loss of gold, if any, by volatilisation. The sample is then chlorinated as usual and the tailings assayed.

This test, if properly carried out, should give a fair idea as to the applicability of chlorination to the ore when subjected to an oxidising roasting. If the extraction in the above experiment has been reasonably good, further tests should now be made with regard to the fineness of the ore required to ensure the most successful treatment. The experiment should now be repeated on 20, 40, 60, 80, and 100 mesh samples of the same ore. This is very important, as results will probably show the extraction on the different sizes to be entirely dependent on the physical characteristics of the ore. If very dense and close-grained, even if the gold is comparatively free, or if associated with pyrites, and these occur in almost microscopical crystals, then fine crushing is almost always necessary; but if the ore is naturally very porous, or capable of being made so in the roasting, then crushing to a 16-mesh to the linear inch may give as high an extraction as crushing to 80-mesh.

After determining the most suitable mesh of screen to use, the next step will be to find the most suitable temperature in roasting. Some ores containing a large percentage of sulphur, like concentrates, must be subjected to the usual slow, gradually increasing oxidising roasting. Ores containing less sulphur, or such as are partially oxidised, can be roasted in a very much shorter time, but require a higher temperature throughout.

Some ores entirely free from sulphur require a slight roasting. This is due to the fact that the gold is associated with the hydrated oxides of iron. This roasting is more properly called a calcination, as the heat used is just sufficient to expel the combined water.

The proper temperature in roasting may be determined by taking different samples of the same ore, the proper fineness having been previously determined. Three experiments will suffice to give a good idea as to the amount of heat to use. The first experiment should be conducted at a low heat from the start, gradually increasing to a light red at the end; the second started at a moderate heat, gradually increasing to a light red at the end; the third started at a light red heat, and kept so throughout. The time required for the three roastings should be the same—about two hours—to make sure that the first sample is thoroughly roasted. After roasting, each of the samples should be weighed, and the loss in weight noted, then assays made to determine the loss in gold, if any, by volatilisation. The samples are then chlorinated and tailings assayed. If no loss of gold has occurred by increasing the temperature at the start, and the extraction was equally good, then there is just one more point to be determined, the time required for the roasting. Five samples are taken and roasted, respectively, for one-quarter, one-half, one, one and one-half, and two hours. The

roasted samples are then again assayed for loss of gold by volatilisation, chlorinated, tailings assayed, and results compared.

The results obtained will have now shown the proper fineness required in crushing, and the best method of roasting (if oxidising roasting has been successful).

There is one more factor to be determined, the least amount of chemicals required. It is a very difficult matter to decide accurately on so small a scale as a laboratory experiment, since the actual condition of the working scale is not easily obtained with small quantities of ore. Indeed in actual working the consumption of chemicals seems to depend to a considerable extent on the size of the charge of ore, the larger the charge the smaller being the consumption of chemicals per ton of ore. This may be accounted for by the fact that an excess of chlorine or bromine is always necessary, and this excess will treat three or four tons just as well as one.

*Treatment of Ores subjected to a Chloridising Roasting.*—Some ores, when given a perfect oxidising roasting cannot be successfully chlorinated, because they require too large an amount of chemicals to produce an excess of chlorine or bromine. This is due to the presence of certain oxides and sulphates of base metals in the ore, such as zinc, copper, calcium, magnesium, and others, absorbing the chlorine as fast as it is produced. When such is the case, the difficulty may be overcome by the addition of a certain percentage of salt in the roasting. The manner in which the salt is added depends upon the nature of the gold and other metals in the ore; but, as a rule, it is best to add it near the end of the roasting.

In a chloridising, as in an oxidising roasting, the same questions should be determined with regard to fineness of crushing, temperature of roasting, and time of exposure of ore in furnace, together with the amount of salt, and when and at what temperature to add it. In addition to the foregoing experiments it is always advisable, although not absolutely necessary, to make a quantitative analysis of each ore treated. In practical work, where large quantities of ore from the same mine are constantly being treated and the ore is of a uniform nature, a part of the average sample may be laid away each day until, at the end of a week or a month, when these samples can be carefully mixed and an analysis of the resulting sample made. Where a number of different ores are continually being treated, a comparison of these analyses, with chemicals used and results obtained, could not fail to be of great interest.

The foregoing looks like a formidable mass of work to be expended on any one sample, but it must be remembered that the ore from any one mine, and frequently from one district, acts in a very similar way with any definite metallurgical process, and when such problems are once satisfactorily solved, they will cause very little trouble thereafter.

The practical operations in the mill may be conducted in several different ways, but in this paper reference is made only to the improved system of barrel chlorination.

In the case of ores not suitable for concentration where chlorination must be applied directly to the ore either in the raw or roasted state, the obstacles are more difficult to overcome. Such ores are of common occurrence in many parts of this and other countries, but their successful treatment by chlorination has been rare. The most notable, and probably the only instance in this country where such success has been attained, is in the Black Hills, in South Dakota, where the Golden Reward Company has demonstrated that the process is a practical one.

The ore, after being crushed, is roasted in Bruckner and Howell-White furnaces. After it is roasted the ore is charged into lead-lined barrels, having a capacity of from four to five tons of ore, where a sufficient quantity of bleaching powder, acid, and water is added. The barrels are then revolved for about an hour and a half, at the end of which time the gold is supposed to be chloridised and dissolved. The barrel is then stopped and the gold solution removed from the ore by means of pressure, and a filter arranged inside of the barrel, the outlet of which is connected with a pipe leading to the precipitating

tanks, or, in some cases, to the slime-settling vats, where, after standing a sufficient length of time, the clear gold-bearing solution is decanted into the precipitating tanks.

The gold-bearing solution, containing a large amount of free chlorine, is then treated with  $\text{SO}_2$ , until all the free chlorine is destroyed; when this point has been reached, the  $\text{H}_2\text{S}$  is turned on and the gold precipitated. The precipitated gold sulphide is collected in filter-presses, dried, roasted, and the gold melted into bars. Such, in brief, is the process followed at present by the most successful chlorination mill in this country, where ore is chloridised without previous concentration. Five prominent features may be mentioned in the process, as described. They are: Crushing, roasting, chloridising, leaching, and precipitating. It seemed impossible to leach very fine ore by the system employed in the Golden Reward Mill. In 1888, while assistant to Mr. T. K. Rose, now of the Royal Mint, of London, the author experimented with the ores of the Black Hills district, and found it a comparatively easy matter to roast and chlorinate them when crushed sufficiently fine to 30-mesh. In January 1893 the writer was again called upon by the Black Hills Milling and Smelting Company to demonstrate, if possible, the cause of their inability to chlorinate the ore. Rolls were used to crush the ore, and screens of 10 meshes to the linear inch. The roasting was accomplished by means of Bruckner and Howell-White furnaces, two of each kind being in operation.

*The Chlorination of Gold Ores.* L. D. Godshall. Eng. and Mining J. 1894, 32—33.

THE Rapid City works used bromine almost altogether, instead of chlorine. The author has recently been informed that bromine can now be obtained in quantity for as little as 18 cents per pound; ore well roasted should not require more than  $1\frac{1}{2}$  lb. of bromine per ton of ore; in fact, considerably less than this was used at Rapid City at times; whereas with chlorine it takes an exceptionally well roasted ore to bring the cost to 50 cents per ton of ore, taking western prices of chemicals as a basis.

That it is possible to extract a very high percentage of the gold from the refractory ores of the Black Hills by means of either chlorine or bromine when such ores are crushed to pass only an 8 or 10-mesh screen, is extremely doubtful, but with fine crushing it becomes at once simple and practical. The only difficulty yet remaining is the crushing. To crush a hard ore in the dry state so as to pass through a 30-mesh screen is a difficult thing to accomplish by means of rolls, while pulverisers of all kinds are still more to be dreaded.

The most practical and at the same time the most economical method of fine dry crushing is, in the opinion of the writer, by stamps. The desirability of using stamps instead of rolls will unquestionably be contrary to the convictions of many engineers. The writer, however, has seen and heard of so many breakdowns and so much trouble in mills where rolls were used, that the conviction has been forced upon him that stamps, notwithstanding their increased initial cost, and power required, are more reliable and more economical. An objection frequently brought against the use of stamps is the fact that a great part of the ore is crushed very fine and apt to produce slimes, but where pressure leaching is used these slimes are of no serious consequence. In fact, with many ores the finer the crushing the higher the extraction, and pulverisation rather than granulation should be the aim.

The furnace best adapted for roasting such fine ore depends somewhat upon the character of the ore; if very low in sulphur, the Howell-White will answer, although large dust chambers should be provided with this furnace. Much of the flue dust would also have to be roasted a second time. The Bruckner cylinder is better adapted for ores in general than the Howell-White, as the charges can be controlled better in this furnace, while the amount of flue dust produced is comparatively small. The Pearce turret furnace is good for ores moderately low in sulphur. This furnace has the good points of the old-fashioned reverberatory, and is mechanical in every respect, producing

also a comparatively small amount of flue dust. For concentrates or such ores as are high in sulphur or where a chloridising roasting is desired at the end the Pearce turret may answer, but a reverberatory or a mechanical furnace in conjunction with a reverberatory is to be preferred.

Where an ore contains iron pyrites or where the gold is associated with a hydrated oxide, as in the Victor mine, roasting becomes necessary. It is a curious fact that not a single ore was found in the camp that would not yield readily either in the raw or roasted state to the process of chlorination. However, the majority of the ores of the camp contain more or less tellurium combined with the gold, making the roasting a more delicate operation than it would otherwise be.

Various figures have been given as to the actual cost of treating a ton of ore by this process. The cost of treatment by this process, or any other, depends on a variety of circumstances, such as the price of labour, fuel, freight, capacity of mill, &c.

Estimate of the cost of treatment per ton by barrel chlorination based on Colorado prices, treating 100 tons of ore per day of 24 hours. The labour required is: 6 men unloading ore and fuel at 2 dols., 12 dols.; 5 men tramming ore and fuel at 2 dols., 10 dols.; 2 engineers at 3.50 dols., 7 dols.; 2 firemen at 2.50 dols., 5 dols.; 2 millmen at 3.50 dols., 7 dols.; 1 oiler, 2 dols.; 2 men at Blake crusher, at 2 dols., 4 dols.; 1 blacksmith, 3.50 dols.; 1 carpenter, 3 dols.; 1 machinist, 3.50 dols.; 1 helper, 2 dols.; 2 men at chlorinating barrels, at 3 dols., 6 dols.; 2 helpers at 2 dols., 4 dols.; 6 roasters at 3 dols., 18 dols.; 6 wheelers at 2 dols., 12 dols.; 2 foremen at 4 dols., 8 dols.; 1 clerk, 3.50 dols.; 1 assayer, 4 dols.; 1 helper, 1.50 dols.; 2 extra men for odd jobs at 2 dols., 4 dols.; 1 superintendent, 10 dols. The total cost will be as follows:—

	Total.	Per Ton.
Labour.....	Dols. 130	Dols. 1.30
Fuel.....	100	1.00
Repairs and supplies.....	50	0.50
Chemicals and assay office supplies.....	75	0.75
Or total cost of treatment per day..	355	3.55

For a plant to treat 25 tons per day of 24 hours, 26 men would be needed, and the cost would be:—

	Total.	Per Ton.
Labour.....	Dols. 75	Dols. 3.00
Fuel.....	40	1.60
Chemicals and assay supplies.....	25	1.00
Repairs and mill supplies.....	25	1.00
Total.....	165	6.60

In both of the above calculations the estimates are high, and should be considerably lowered in actual practice by having discharging waggons; for instance, the cost of unloading is practically done away with. If bromine can be used the cost of chemicals will also be much less, and this item should even be considerably less when chlorine is used. It was thought better to state the costs a little too high than too low. The greatest economy in milling ores by this process or by any other, lies in running the mill to its utmost capacity. It is impossible, as may be seen by the above figures, to treat ore in a 25-ton mill as cheaply as in one treating 100 tons daily, but it is still far worse to build a 100-ton mill and treat only 25 tons a day in it. Under such circumstances the cost of milling

may run up to 8 dols. or 10 dols. a ton. With most ores a minimum net extraction of 90 per cent. can be obtained by this process, but on low-grade ores it is safer to calculate upon a minimum net extraction of 85 per cent. of the gold contents of the ore.

*The Estimation of Phosphorus in Steel containing Silicon, and in Pig-iron.* J. Spüller and S. Kalman. Zeits. Anal. Chem. 32, 1893, 538.

See under XXIII., page 175.

#### PATENTS.

*Improved Process for the Manufacture of Litharge, Red Lead, and Cement from Galena or other Lead Ores, or from Sulphate of Lead.* W. S. Kilpatrick, Glasgow. Eng. Pat. 23,548, December 21, 1892.

THE galena or other lead ore is roasted or sublimed so as to form a mixture of sulphate and oxide of lead. This mixture, or the sulphate itself, is mixed with the necessary proportion of oxide or carbonate of the alkalis or alkaline earths or any other metal, and is calcined in a reverberatory furnace till the mass consists of lead oxide. The sulphate is then removed by grinding and washing if litharge is required, or allowed to remain for cement, and the litharge may be converted into red lead by the ordinary process. Oxygen may be used at any stage of the process if desired.

—F. H. L.

*Coating or Covering Paper with Aluminium.* W. Ibbotson, London. Eng. Pat. 23,603, December 22, 1892.

See under XIX., page 170.

*An Improved Process for the Manufacture of Aluminium Sulphide.* C. T. J. Vautin, London. Eng. Pat. 751, January 12, 1893.

METALLIC aluminium is melted in proper proportions with lead or other suitable metallic sulphide at a bright red heat in a neutral atmosphere, in a crucible or reverberatory furnace with basic lining, and preferably gas-fired.—J. H. C.

*Improved Means for Producing Crucibles or Melting Pots.* E. J. T. Digby, Liverpool. Eng. Pat. 3661, February 18, 1893.

THE materials used are as follows:—

	Parts.
Plumbago, in 17-mesh granules .....	17
"      in 80-mesh " .....	2
Russian clay .....	5
China stone .....	5
Gas carbon .....	2

The three last are separately ground to a fine powder; the others are then added in a suitable mixing machine and kneaded together with a little water. The vessels are then formed, dried at 150° F., and finally baked in a kiln.

—J. H. C.

*Improvements in or relating to Apparatus for Galvanising Sheet Metal.* J. V. Lagnesse, Grevegué, Belgium. Eng. Pat. 4298, February 27, 1893.

THE apparatus consists of two frames provided with hooks, connected with and by rods or links, and so arranged that they can be operated at a distance from the galvanising bath. One of these serves for immersing the plate and the other for withdrawing it and causing it to pass through sand without necessitating the use of rollers or other like appliances.—J. H. C.

*Improvements in the Manufacture of Metallic Oxides and Compounds.* E. de Pass, London. From H. Thofehn, Paris. Eng. Pat. 10,096, May 20, 1893.

THE metals to be oxidised are melted and made to flow in a thin stream through blasts or jets of steam, gas, air, water, acids, or other suitable reagents, which have been previously heated, if necessary. In this way the metals are pulverised or sprayed into little drops or particles, none of which can escape the action of the reagent employed. Illustrations are given of the form of apparatus recommended by the inventor.—J. H. C.

*Process for Avoiding or Considerably Diminishing the Choking and Contraction of the Mouth of the Retort in the Manufacture of the "Thomson" Steel.* A. Raze, Ongrée, Liege. Eng. Pat. 16,857, September 7, 1893.

A NUMBER of openings are formed around the mouth of the retort, and immediately above the level of the molten metal. Through these openings air is blown during the operation of conversion or casting, so as to obtain a more perfect combustion of the matters which ordinarily condense around the mouth.—J. H. C.

*A New Process for Reducing Sulphuretted Ore into Oxides and Sulphur.* L. Bemelmans, Brussels. Eng. Pat. 19,606, October 18, 1893.

See under VII., page 153.

*A New or Improved Metallic Alloy.* J. Cox, Birmingham. Eng. Pat. 22,461, November 23, 1893.

THIS alloy has the appearance of silver, receives and retains a high polish, does not tarnish, can be rolled into sheets or drawn into wire, and is cheap to manufacture. It is composed of copper, nickel, spelter, antimony, tin, and lead, and is prepared as follows:—

Mixture number one is first prepared by melting 78 lb. of copper and adding first 20 lb. of nickel and then 12 lb. of spelter, 1 lb. of antimony, and 1 lb. of tin. 64 lb. of this mixture is then melted and 22 lb. of spelter, 2 lb. of tin, and 8 lb. of lead are added, thus forming the improved alloy.—J. H. C.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

*A New Accumulator.* K. Lenz. Mitth. K. K. Tech. Gewerbe-Museums in Wien. 1894, 84—89.

THE author states that Sigmund Randnitz has constructed an accumulator in which neither pure lead nor pure lead oxides are primarily used as materials, but instead a lead compound the nature of which is kept secret. By charging, this becomes converted into a stone-like peroxide without buckling or altering in shape. The result is a large increase in capacity for a given weight. A cell of three plates (one +, two —) weighed 1.09 kilos. (each plate having a surface of 6 × 16.5 cm.), and had a capacity of 32.8 ampère-hours, or 30.1 per kilo.; the acid strength was 26—27 per cent. The advantages and disadvantages of the systems of Planté, Faure, Tommasi, Tudor, and Randnitz are thus contrasted, the capacities being stated in ampère-hours per kilo. of plates.

*Planté.*—Requires a long period for formation; plates separated by intermediate filamentous layer. Capacity, 3—4.

*Faure.*—Short period of formation. Porous intermediate layer to prevent erosion and breaking up of plates. Capacity, 10—15.



*Tommasi*.—Diminution of tendency to break up by introduction into perforated acid-proof frames. Capacity, 22·5.

*Tudor*.—Production of stable active plates from massive lead plates. Capacity, 3·5—4.

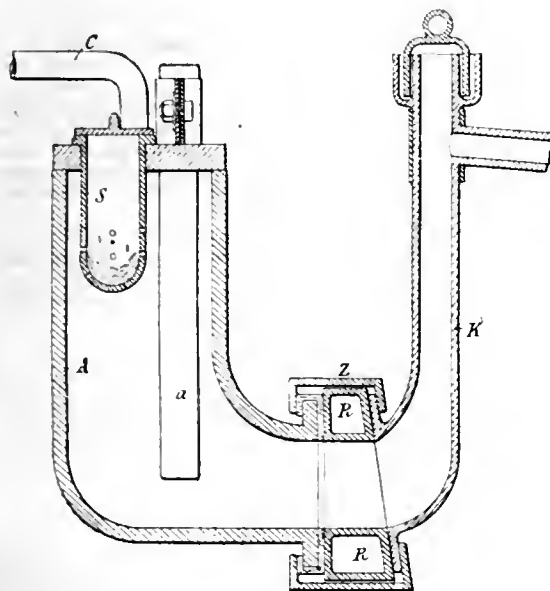
*Randnitz*.—Unalterability, cheap renewal, large capacity. Capacity, 30·1.—C. R. A. W.

*New Apparatus for the Manufacture of the Metals of the Alkalis and Alkaline Earths by Electrolysis.* W. Borchers. *Zeits. f. ang. Chem.* 1893, 16, 486—489.

In the construction of apparatus for the manufacture of the alkali metals by the electrolysis of the fused haloid salts of the latter, the following rules must be observed:—1. The alkali metal must be deposited at a metallic (iron) kathode. 2. The kathode space must permit of the collection and removal of the metal without the latter coming into contact with reducible substances. 3. The anode must be of carbon. 4. The anode space must permit of the ready removal of the halogen, and its walls must be constructed of material capable of withstanding the action of halogens and haloid salts. 5. No metallic particles should be present in the melt between the poles. 6. All the parts of the apparatus must be constructed of fire-resisting material.

These conditions are partially fulfilled by Grabau's apparatus (1889), and also by an apparatus described by the author in 1891. The whole of the difficulties have only been surmounted, however, by the construction of the apparatus shown in Figs. 1. and 2. It consists of two

Fig. 1.

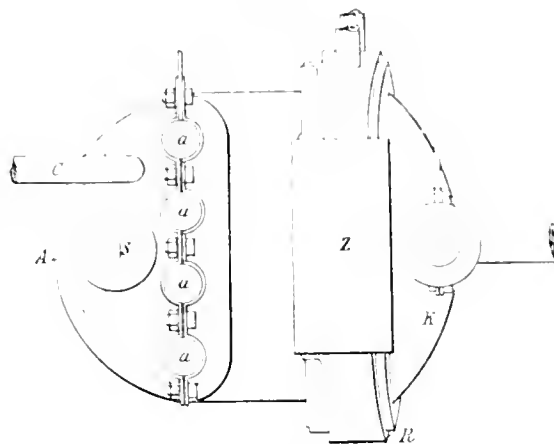


COOLING WATER INFLOW.

communicating vessels, of which one, the kathode space K, is of iron; the other, the anode space, is of chamotte. A jacket is furnished for the anode a, by means of which no chlorine can reach the kathode, and the outside of which is out of contact with the melt and with the strongly reducing alkali metals. The employment of a tubular kathode for the collection and discharge of the metal is also possible, on the inner side only of which the metal can be deposited, as the outer side does not as a rule come into contact with electrolytes. The two vessels are bound together by a hollow metallic band R and two clamps Z above and below it; a stream of water passed through the hollow band, locally solidifies the melt, which effects the luting. The

flange between the vessel A and the band R is packed with asbestos, which acts as a damper in order to prevent cracking. A tube C serves to lead off the halogen. Dropping the salt directly into the anode vessel is apt to cause the latter to crack; the salt is therefore introduced in small lumps through S, the bottom of which is covered with a layer of asbestos.

Fig. 2.

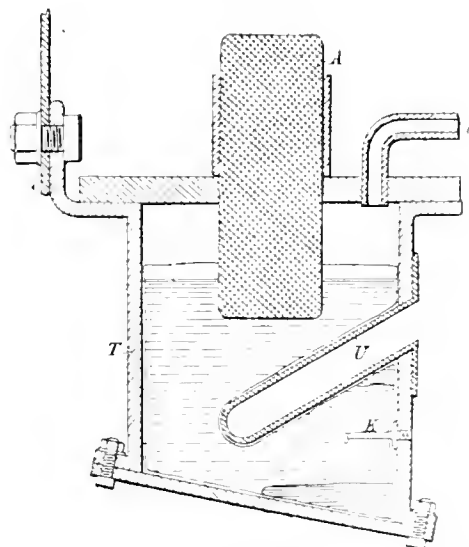


The current density at the kathode should be about 5,000 ampères per square metre. An advantage of the new apparatus is that with a current density remaining constant at the kathode, the current density at the anode may be reduced, which means a great saving of energy.

The electrolytic preparation of the alkaline-earth metals presents many more difficulties than that of the alkali metals. The mixtures of salts melt only at a red heat, and the metals themselves at still higher temperatures. The melting point of barium, for example, is near to that of iron; then follows strontium, calcium having the lowest melting point of the three.

A current density at the kathode of 1,000,000 ampères per square metre is necessary. Fig. 3. shows an improved

Fig. 3.



apparatus for this purpose. A wrought-iron crucible T serves for the decomposition vessel; a U-shaped piece of sheet iron is inserted into it at U, and below this pins K which serve as kathode, are screwed into the walls of the



vessel. The size of the pins is so regulated that during the electrolysis there shall be a current density of one ampère per square mm. of exposed surface. A strong bar of carbon A serves for the anode, or a bundle of thin rods or plates of the same material; a tube C is furnished to lead off the halogen.

The *modus operandi* is as follows:—The fused haloid salts of the alkaline-earth metals are poured into a cold crucible, the heated bar of carbon immediately dipped into the melt, and connected with the current. The walls of the crucible are covered with a crust of the solidified melt, which isolates them completely; this is therefore maintained during the operation by cooling either by air or by water. The crust is only quickly re-melted at the kathode. The resistance of the melt must be great enough that the current causes a sufficiently strong heating to keep the electrolyte molten. In consequence of the great current density at the kathode, this is heated far above the surrounding temperature. The metal here deposited may agglomerate to a molten mass, which falls or rises, and finally solidifies under U. The great current density causes much of the metal to be swept into the anode space, where, naturally, it is oxidised; a portion of the metal, after dropping from the kathode, is also dissolved by the haloid salts, forming basic compounds. The highest yield of metal realised is 20 per cent. of the theoretical; this, however, shows a great advance, for with the arrangements at hand heretofore only a 5 per cent. yield is attainable.—A. R. L.

#### PATENTS.

*New or Improved Treatment of Hydrocarbons (or of Analogous Substances Composed mainly of Hydrogen and Carbon) to Decompose them and Obtain Various Products.* W. J. Wisse and A. Schneller, The Hague, Holland. Eng. Pat. 21,343, November 23, 1892.

ACCORDING to this invention, hydrocarbon oils, such as kerosine oil or its residue, are split up electrically in a closed iron vessel in which are immersed electrodes supplied with electrical energy of high electromotive force—about 30,000 volts—from an alternating current transformer, or high potential continuous currents may be employed; the oil undergoing decomposition with deposition of carbon, which it is proposed to collect for the manufacture of lampblack by subsiding or filtration. Other products are collected from the oil so treated, some better adapted for lubricating purposes than the original oil; these, as well as the vapours escaping from the vessel, containing anthracene and other products, may be collected and separated by distillation. The inventor proposes to treat resin, tar, bitumen, vegetable oils, fats, alcohols, &c., to obtain lampblack and by-products analogous to those obtained in the treatment of mineral oil.

—J. C. R.

*New or Improved Galvanic Batteries.* C. A. J. H. Schroeder and H. E. R. Schroeder, England. Eng. Pat. 23,101b, December 15, 1892.

EACH cell of these primary batteries consists of a chamber divided by a porous partition into two compartments, each supplied by two tubes with different mixtures of gases. Each compartment contains two platinum conductors, which, on being heated and brought into contact with each other, cause a chemical combination of the gases in the different compartments, and generate an electric current. The porous partition may be replaced by a flat porous pot filled with a conductive liquid, such as dilute sulphuric acid, &c. In battery No. 2 the outward application of heat is employed, and two single gases are used instead of two gas mixtures. The galvanic action in battery No. 3 depends on one liquid being an electro-positive element dissolved in a conductive liquid, and the other an electro-negative element also dissolved in a conductive liquid. To dissolve these substances an alteration of temperature is required in some cases, and a high pressure in others. The positive liquid in

this battery may be replaced by immersing amalgamated zinc or one of the alloys given in Eng. Pat. 22,956, 1893, in dilute sulphuric, hydrochloric, or nitric acid.—G. H. R.

*Improvements in Electric Smelting and in Furnaces therefor.* R. Niewerth, Berlin, Germany. Eng. Pat. 23,773, December 24, 1892.

THESE improvements relate to the construction of an airtight electric furnace, with electrodes passing through stuffing boxes at the top of the furnace, the cathode being at the bottom, and shown as a flooring to be in contact with the metal to be smelted, for which arrangement advantages are set forth.

The smelting is proposed to be effected by an electric arc or arcs out of contact with air, to avoid oxidation, which is said to render electric smelting expensive. Inlet and outlet pipes are provided for gases, with a hopper with water or other seal at the top of the furnace to supply fresh material. In the case of removing carbon from aluminium and iron, it is proposed to conduct the operation in the presence of hydrogen, which will remove the carbon as hydrocarbon gases, and assist the action of fusion.

When smelting copper, it is proposed to introduce carbon monoxide into the furnace to avoid oxidation, and if oxides are present as impurities reducing gases are admitted.

—J. C. R.

*Improvements in Electrolytic Apparatus.* E. Andreoli, Brixton, Surrey. Eng. Pat. 1661, January 25, 1893.

THIS invention (relating to further improvements on Eng. Pat. 18,597, 1891) refers to the employment of what is termed a conducting diaphragm, constituting a cathode, but only taking a small proportion of the whole current passing through the electrolyte, which is mainly carried by a separate cathode, between which and the anode the diaphragm is placed. This diaphragm is constructed of gauze and granular conducting material, which is protected from corrosion by the slight electrolytic action taking place upon it while the electrolysis current is passing, and when disconnected from that source of supply, to attain the same object, it may be joined up with a primary or secondary battery in the same manner.

Apparatus is described for the electrolytic production of chlorine and caustic soda, employing an anode of carbon and a cathode of iron; circulating and cooling the electrolyte, and decomposing hypochlorites in the anode compartments by acids or salts, being also referred to.—J. C. R.

*A New Galvanic Battery.* C. A. J. H. Schroeder and H. E. R. Schroeder, Balham, Surrey. Eng. Pat. 2241, February 1, 1893.

A BOLT of slate, porcelain, agate, or other suitable material is provided at each end with threads, lubricated with black lead; on one end of the bolt is screwed a nut of the same or of either of the above-mentioned materials. The bolt is then reversed in position, and an amalgamated zinc plate is threaded on it. This is followed by a plate of vulcanite, slate, or other suitable insulating material, which in its turn is followed by a carbon plate, a zinc plate, an insulator, and so on alternately until the last plate is in position, when they are firmly pressed together, and held in place by a second nut screwed on the other end of the bolt. The complete battery cell when in use is placed in a suitable receptacle of such shape that the cell fits closely in one direction, while a free space is preferably left at each side in the other. If the battery is required for continued action a reservoir is attached to it. The solution used may consist of any suitable exciting liquid, such as bichromate of potash, or that described in Eng. Pat. 23,101, 1892.—G. H. R.

*A Manufacture of Pharmaceutical Preparations, for Use more particularly in Cases of Anæmia and for Weak Throats.* W. Webster and T. Moore, Blackheath, Kent. Eng. Pat. 2884, February 9, 1893.

THIS invention relates to the production of weak solutions of iron, for pharmaceutical preparations, by placing electrodes of iron, to which battery connections are made, in an electrolyte of a weak solution of common salt, thereby obtaining by electrolysis oxides and carbonates in solution, which may be under pressure of carbon dioxide generated within the vessel, or separately treated with that gas to form aerated chalybeate water. An apparatus is shown in which the cathode of iron forms the lower part of the containing vessel, and an iron pipe the anode, which also answers for leading out the solution, as in a seltzogene, and can be renewed. The carbon dioxide is generated in a glass chamber fixed on to the top of the iron cathode. Glycerin may be added to the solution to prevent oxidation of the neutral salt of iron, and to reduce its astringent properties for internal use; and where carbon dioxide is applied direct to the electrolyte, common salt may be dispensed with.

—J. C. R.

*Improvements in Secondary Battery Plates or Elements.* A. Hough and O. March, London. Eng. Pat. 3731, February 20, 1893.

ACCORDING to this improvement the lead peroxide plates for secondary batteries are formed by filling the support with lead oxide mixed in a dry state with some suitable setting salt. The supports are of lead or lead alloy, either in the form of grids, or of troughs or channels, and are constructed in such a way as to yield without injury or distortion to the expansion of the active material which takes place during formation.—G. H. R.

*Improvements in the Preparation of Carbon Electrodes for Electrolytic Operations.* E. H. Liveing, London. Eng. Pat. 3743, February 20, 1893.

THE object of this invention is the removal of the undecomposed hydrocarbons from the retort carbon, before the latter is used as an electrode, so as to obviate the objections which may arise from their presence. This is effected by subjecting the carbon to a high temperature in a current or atmosphere of chlorine gas.—G. H. R.

*Improvements in the Construction of Carbon Electrodes.* E. H. Liveing, London. Eng. Pat. 3741, February 20, 1893.

WITH the object of overcoming the difficulties occasioned by the electrolysis of the chlorides of sodium and potassium, the inventor makes the following arrangement:—Broken pieces of gas-retort carbon are placed on a sieve, grid, or series of bars of slate or other non-conducting material. Electrical communication is made with the anode chamber, which is closed at the top by means of carbon rods or protected metallic conductors passing through stuffing boxes in its cover, and making contact with the carbon on the grid; and as the carbon rods will also waste at their ends they are made to slide through the stuffing boxes by means of a spring or weights. The number of rods necessary for a cell will be determined by the amount of current required.

—G. H. R.

*Improvements in the Construction of Plates for Accumulators or Secondary Batteries.* W. P. Thompson, London. From La Société Electric Phœbus, Paris, France. Eng. Pat. 4360, February 28, 1893.

ACCORDING to this improved method of construction the plates are cast with two or more grated faces held apart, and rendered solid by means of cross pieces, which are also cast at the same time with the plates, forming a grate, which, it is claimed, possesses great mechanical solidity. The plate, formed preferably of antimoniated lead, presents,

as a whole, two grated parallel walls provided with cross pieces on a median vertical line, and three horizontal ones. The positive and negative plates are separated from each other by means of any suitable insulating material placed in holes in them, and projecting beyond them, maintaining them at a suitable distance apart. G. H. R.

*Improvements in Electric Accumulators.* F. W. Ellermann, Vienna, Austria. Eng. Pat. 8952, May 1, 1893.

ACCORDING to this improvement the active porous material (preferably oxides of lead, such as litharge or peroxide of lead) is applied in a comparatively thick layer, the individual particles of which are connected electrically by means of mercury so as to reduce the resistance; while for the purpose of avoiding too rapid a discharge a finely-divided electrolytically inert body is added to the mass. The mercury compound added is the sulphate, which becomes reduced, and thus mercury becomes disseminated through the mass. The improved active mass is composed as follows:—

For the positive mass:

	Parts
Peroxide of lead.....	100
Sulphate of sodium.....	10
Sulphate of magnesium.....	5
Peroxide of manganese.....	3

For the negative mass:

	Parts
Litharge.....	100
Sulphate of sodium.....	10
Sulphate of magnesium.....	5
Sulphate of mercury.....	3

—G. H. R.

*Improvements in the Manufacture of Ammonia, Sodium Hydroxide, and Chlorine, and in Apparatus therefor.* C. Kellner, Vienna. Eng. Pat. 13,722, July 14, 1893.

See under VII., page 152.

*Apparatus for the Electrolytic Production of Bleaching Liquid.* C. Kellner, Vienna, Austria. Eng. Pat. 13,723, July 14, 1893.

THIS apparatus relates to a plan of constructing electrolytic apparatus for the production of hypochlorite of soda from a solution of common salt, by arranging a series of electrodes in a containing tank in such manner that their opposite sides act as different electrodes, but all contained in the same electrolyte, which circulates round their ends, taking a zig-zag course by spaces left for the purpose at the end of each alternate electrode. The electrolyte is supposed to undergo electrolysis on the opposite side of each electrode rather than travel by way of the passage of electrolyte around the ends of the electrodes from one extreme plate to the other, to which, only, terminal connection is made. Where carbon is used as electrodes, it is proposed to introduce a filter of glass wool between the decomposing apparatus and bleaching reservoir.—J. C. R.

*Improvements in the Method of and Apparatus for Electrically Heating or Working Metal or other Materials capable of Momentarily Withstanding the Heat of an Electric Arc.* W. P. Thompson, Liverpool. From C. L. Coffin, Detroit, State of Michigan, U.S.A. Eng. Pat. 18,990, October 10, 1893.

THIS invention consists essentially in acting upon the electric arc produced between electrodes by an electromagnet in such manner as to attract or repel the arc, and so cause it to play upon a larger area of the material being heated, than if permitted to concentrate its heat upon one point, it being rapidly moved from one point to another by the influence of the magnet, whereby an arc of greater power may be used than if it were stationary, and a correspondingly greater effect produced. Apparatus is described

and shown for carrying out this method, which is said to be applicable for heating other materials than metals, such as glassy sticite, or other silicious or boracious compounds in which it may be desired to produce incipient fusion.

—J. C. R.

*Improvements in Apparatus for the Electrolytical Decomposition of Salt Solutions.* C. Kellner, Vienna, Austria. Eng. Pat. 19,542, October 17, 1893.

This apparatus is for bleaching paper pulp or textile fabrics, and is termed a bleaching block. A number of plates are bound together, but insulated from each other, so as to act as electrodes in series, terminal connection being made to the end plates only. These blocks are immersed in the solution to be electrolysed, as common salt, for the production of hypochlorite of soda, and are shown for use in an ordinary rag engine containing paper pulp, and for bleaching textile fabrics. For the surfaces of the plates making up the block, the anode surface is of platinum foil, and the other, or cathode side, amalgamated with mercury, the plates being of sheet copper, tombac, or phosphor bronze. The apparatus being worked in the presence of the material to be bleached, which passes between the plates, economy is claimed.—J. C. R.

*Production of Ozone and Luminosity by Electrical Means.* E. Andreoli, Brixton, Surrey. Eng. Pat. 21,707, November 14, 1893.

This invention relates to the production of ozone and luminosity, by placing in a vacuum tube of glass, or other dielectric vessels, an electrode consisting of a carbon filament or a metallic wire or wires radiating towards the inner side of the glass tubes; and outside the tube, and in proximity to it, is fixed the other electrode, having points directed towards the centre of the tube. These electrodes being connected up with a Ruhmkorff coil or high frequency transformer, as soon as the current passes the inside of the tube becomes luminous; at the same time on each of the points of the electrodes the characteristic glow of the silent discharge appears, and is accompanied by a strong smell of ozone. It may be understood (although not stated) that the apparatus is worked in atmospheric air or oxygen. Eng. Pats. are referred to: 17,426 of 1891, 9631 of 1892, and 21,794 of 1892; this Journal, 1893, 452.—J. C. R.

*Improvements relating to Storage Batteries.* E. P. Usher, Massachusetts, U.S.A. Eng. Pat. 22,311, November 21, 1893.

The inventor describes an improved battery plate constructed of a frame having upright bars or arms vertically grooved in their outer faces, and formed with edgewise flanges extending into the space between the bars, in combination with non-conducting separators in contact with the opposite faces of the frame, and with a filling of material adapted to become active in the spaces. The plates and interposed separators are set in a shallow body of cement or gum, the several plates being supported at a slight distance above the bottom of the vessel containing the gum.—G. H. R.

*Improvements in Storage Batteries.* E. P. Usher, Massachusetts, U.S.A. Eng. Pat. 22,312, November 21, 1893.

In this battery the plates are formed of lead sheets separated by sheets of wood, earthenware, or any equivalent porous substance, and rest in spaces or end pieces provided with a slot their whole length to receive the edge of the lead sheet, and preferably having the sides which come against the surface of the separator sheet fluted or corrugated, which allows of the circulation of the acid. The cell is built up of several of these sets, and when put together the entire cell is placed in a shallow pan filled to the depth of a quarter or a third of an inch with hot, liquid gum, which sets hard when cold. The several chambers between the separators which contain the lead plates are filled with lead oxide.—G. H. R.

*Improvements in the Manufacture of Incandescent Bodies for Incandescent Gas Lighting.* R. Langhans, Berlin, Germany. Eng. Pat. 22,396, November 22, 1893.

See under 11., page 141.

*Improvements in Disinfecting.* E. Hermite, E. J. Paterson, and C. F. Cooper, London. Eng. Pat. 18,370, December 16, 1893.

See under XVIII. (C.), page 170.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*Valuation of Tallow by Determination of the Solidification Point of its Fatty Acids.* F. Wolfbauer. Mitth. k. k. Tech. Gewerbe-Museums in Wien, 1894, 57.

See under XXIII., page 181.

### PATENTS.

*An Improved Furnace Pan and Concentrator for the Boiling of Fish Livers and the like.* C. B. Lotherington and I. Spencer, Hull. Eng. Pat. 23,542, December 21, 1892.

DESCRIPTIONS and drawings are given of a closed, flat-bottomed pan with sloping sides, and fitted with an agitator. An outlet pipe carries off the noxious vapours evolved. This outlet pipe is connected (outside the pan) with a pipe with rose, through which water may be admitted in sprays, which condenses part of the vapours en route to the fireplace, whilst the rest are drawn to the ash-pit and the fire, and thus consumed. A suitable drain pipe passing vertically downwards to the drains, carries away any condensed water, &c., collecting in the lower part of the pipe from pan to fireplace, as it forms an elbow in bending round and proceeding horizontally to the grate of the fireplace.

—K. E. M.

*Improvements in and connected with the Separation of Cholesterin and Fatty Acids from Wool Fats.* L. Krause and J. Mayer, Wittenberg. Eng. Pat. 20,639, November 1, 1893.

The cholesterin and fatty acids contained in raw wool-grease are separated by distillation, for which purpose superheated steam or better still, direct heat is applied. The distillation is continued until the distillate, consisting of a yellow granular mass composed chiefly of fatty acids, begins to show a brown tint. The residuum is a black-brown product, consisting mainly of cholesterin.—K. E. M.

## XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

### (A.)—PIGMENTS, PAINTS.

#### PATENTS.

*An Improved or New Manufacture of Black Lead.* R. Ripley, Liverpool. Eng. Pat. 23,206, December 16, 1892.

To obviate the necessity for the large amount of friction required to give a polish to black lead when used in the

ordinary way, the inventor prepares a kind of paint consisting of black lead, beeswax, and turpentine in varying proportions, of which the following may be taken as the average:—1 part of wax, 2 of turpentine, and 24 of plumbago. The turpentine is replaceable by petroleum spirit, and the beeswax by any other wax which will give a similar polish.—F. H. L.

*Improvements in Apparatus employed in the Manufacture of White Lead.* J. V. Walton, Bollisheope, near Darlington. Eng. Pat. 3116, February 11, 1893.

This patent refers to the manufacture of white lead by the Dutch method, and has for its object to reduce to a minimum the handling of the product during some of the different processes involved. The stack is a brick building containing tiers of tank girders of L-section, the flanges of which act both as rails for the passage of the loading and unloading trucks, and also as supports for the perforated grids on which the pots of lead and acid stand. The webs of the girders are of such a height, that they project above the pots when *in situ*, so that on them may be laid a layer of flooring boards to carry the tan. On the outside of the building, corresponding with each pair of girders is provided a hinged platform supported by movable brackets, which, when erected, forms a continuation of the rails for the trucks. These trucks are provided underneath with hooks and lifting gear, so that they may carry the grids to their appointed place, drop them, and return to the outside platform, there to receive a fresh grid and pots raised from the ground by a crane. Doors are provided to close the different apertures, as well as skylights, sight-holes, ventilators, &c. On completion of the reaction, the stack is uncharged in a similar manner, the white lead removed from the pots and taken to the grinding mills and treated as usual. The drying stove consists of a building somewhat similar in design to the stack, having tiers of angle irons or ledges on the sides to carry the trays; on the floor is laid a line of rails for a truck, which has a platform raised by a long screw, so that the trays, when placed on the truck, may be made to coincide with any of the different shelves. In the drawings is shown a stove of two storeys, each with its line of rails, and two trucks of different heights are provided in case the one has not sufficient lift to reach every shelf.

—F. H. L.

*Improvements in Apparatus employed in the Manufacture of White Lead.* J. V. Walton, Darlington. Eng. Pat. 16,703, September 3, 1893. (Compare preceding abstract.)

This patent is the complement of the preceding one, also having for its object the avoidance of manual labour during the processes of grinding and packing the white lead made by the Dutch method. The white lead is transferred from the converting pots by means of suitable tools to boxes fixed on the floor of tipping trucks, which are run into the grinding shed. This is as far as possible an air-tight building, lighted from the sides and top and provided with a ventilating fan. The truck is tipped by gear actuated from outside the building over the mouth of a hopper, into which water is sprayed, communicating with the grinding rollers. After grinding and drying, the material is emptied into boxes fixed on exactly similar trucks and run into the packing room. This resembles the grinding shed, being air-tight and having its fan, and the trucks are tipped as before over a hopper fitted with a regulating slide, also worked from the outside. Doors are provided at opposite sides, through which the trucks carrying the casks are introduced and withdrawn. While charging, the cask is lifted off the truck by a counterweighted lever and hooks, to which a revolving toothed wheel gives a rapid succession of blows so applied as to lift it up and let it drop 20 or 30 times per minute, whereby the white lead is thoroughly consolidated without having recourse to the hand labour usually employed in packing.—F. H. L.

*Improvements in the Manufacture of Oxide of Iron, and in Means applicable for use in such Manufacture.* H. W. Hemingway, London. Eng. Pat. 4001, February 23, 1893.

In the ordinary processes for the manufacture of oxide pigments ("Venetian red," &c.) the crude ferrous sulphate is ground to powder before furnacing, which results in its becoming locally overheated and consequently spoilt in colour. To avoid this, the inventor, after evaporating down the copperas liquor, casts the sulphate into rectangular blocks of a size and shape corresponding with the retorts in which it is ignited. A convenient size is 12 inches in diameter and 1 inches thick, and they may advantageously be cast round a hook- or eye-ended metal bar to aid in their manipulation. The furnace for burning them consists of a number of vertical chambers, the alternate ones being filled with the blocks, while the furnace gases circulate in the others. The former are provided with a lid at the top to introduce the sulphate, and a hinged door at the bottom to allow the finished oxide to fall through on to barrows or other receptacles when opened. This device renders the process continuous and does away with the cooling of the furnace, to which the usual conduits for the removal of the acid fumes are attached near the top of each of the vertical retorts.—F. H. L.

*Improvements in and relating to the Manufacture of Paints, Sulphuric and Sulphurous Acid from Proto-sulphate of Iron.* M. N. D'Andria, Stretford, Lancaster. Eng. Pat. 4342, February 28, 1893.

THE ferrous sulphate is heated in retorts in the usual manner, the evolved gases being made to pass into a sulphuric acid chamber which is at work in the ordinary way. The anhydrous sulphuric acid is at once absorbed by the chamber acid, strengthening it, and thus materially reducing the cost of the subsequent concentration. As an alternative arrangement, the pipes leading from the retorts may be made to condense the sulphuric acid, and the remaining sulphurous oxide either led into the chamber to be oxidised as before by the nitrous gas or used for the manufacture of sulphurous acid or sulphites.—F. H. L.

*An Improved Paint.* E. Edwards, London. From J. W. Overton, Louisville, U.S.A. Eng. Pat. 9825, May 16, 1893.

This paint, which is said to be specially suitable for painting ships' bottoms, being unaffected by salt water, is prepared by grinding rutille (titanium dioxide) with coal-tar, bitumen, "or other hydrocarbon of the like kind." Any other compound of titanium may be used if preferred, and the proportions may vary from equal parts to  $3\frac{1}{2}$  parts of the pigment to one of the vehicle.—F. H. L.

*Improvements in the Manufacture of White Lead.* E. Waller, New York. Eng. Pat. 22,294, November 21, 1893.

This patent refers to improvements in the use of ammonium acetate for decomposing litharge, massicot, or native lead carbonate. The ore containing lead in the latter form is heated in a still with a 10 per cent. solution of ammonium acetate, about 40 gallons being employed for every 100 lb. of lead in the material to be treated. The evolved vapours are condensed and form a solution of ammonium carbonate, which is added to the contents of the still after the insoluble substances have been filtered off. A precipitate of white lead is thus obtained, and the supernatant liquid, consisting of ammonium acetate, may be used over again with the next batch of ore. The precipitation should be carried out at a high temperature, so that the product may be as fine as possible. If the ore contain copper or silver, metallic lead should be added in quantity sufficient to replace them, and they may be recovered from the filter sludge.—F. H. L.

## (E.)--RESINS, VARNISHES.

## PATENTS.

*Improvements in or relating to Compounds of "Blown" or Aërated Oils with Pyroxylin.* I. S. Fairfax, London. From W. D. Field, Milburn, New Jersey, U.S.A. Eng. Pat. 3469, February 16, 1893.

By combining a blown non-drying oil, containing unsaturated fatty acids, with pyroxylin or nitrocellulose a compound is produced, which is proof against the action of water and atmosphere. It is suitable as a coating for textile fabrics, paper, and leather, also useful as an insulating material. Drying oils or those belonging to the linseed oil group are not suitable for this purpose, whilst those belonging to the cotton-seed oil and olive oil group are well adapted. The blown oil is prepared by aërating a suitable oil at a temperature of about 200° C. until it has obtained the required degree of viscosity. Before mixing the pyroxylin with the oil the nitrocellulose is dissolved in a liquid, which is also a solvent for the oil, and for this purpose a mixture of amylacetate and benzine (petroleum spirit) is recommended.—K. E. M.

*Improvements in and connected with Varnishing in Colours.* J. Perl, Berlin, Germany. Eng. Pat. 21,455, November 10, 1893.

THE articles to be varnished, metal, bone, celluloid, &c., are coated in the usual manner with colourless varnish, thoroughly dried, and then coloured. The colouring bath consists of a solution of a vegetable, aniline, or alizarin colouring matter in a strong alcohol, e.g., methyl, ethyl, or amyl alcohol, or in ethyl acetate, amyl acetate, or acetone. In this solution the articles are momentarily immersed, freed from excess of liquid by shaking or by centrifugal action, and finally dried at a gentle heat.—R. B. B.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

*Glycerin as an Agent for Softening Leather.* Papier Zeit. 1893, 18, 2280.

GLYCERIN imparts considerable suppleness to leather, but soon sweats out in damp air. To fix it in the leather, it should first be incorporated with four times its weight of the buttery mass made by dissolving beef fat in warm cod-oil. Another method of rendering the glycerin a permanent constituent of the leather is to incorporate it with a small proportion of white of egg; this mixture may be applied alone or may be followed by the above described glycerin-oil. A solution of dextrin may be substituted for white of egg if this latter be too costly. By dissolving a tan-stuff in glycerin and mixing it with cod-oil and fat, a valuable stuffing for leather may be obtained.—A. G. B.

*Cements for Leather Belting.* Papier Zeit. 18, 2618.

THE following are given as suitable cements for making joints in leather driving belts:—

1. Equal parts of good hide glue and American isinglass, softened in water for ten hours, and then boiled with pure tannin until the whole mass is sticky. The surfaces of the joint should be roughened and the cement applied hot.

2. 1 kilo. of finely-shredded gutta-percha digested over a water-bath with 10 kilos. of benzol until quite dissolved, when 2 kilos. of linseed-oil varnish are stirred in.

3. 1½ kilos. of finely-shredded india-rubber is completely dissolved in 10 kilos. of carbon bisulphide by heating. Whilst hot 1 kilo. of shellac and 1 kilo. of turpentine is added, and the solution heated until the two latter ingredients are also dissolved.

4. 1 kilo. of best glue is dissolved at a moderate heat in 1½ kilos. of water, and thickened to the consistency of syrup. 100 grms. of thick turpentine and 5 grms. of carbo-holic acid are carefully stirred in whilst hot. The mixture is poured into flat tin pans and allowed to cool, then cut into pieces and dried in the air. The cement is made liquid with a little vinegar and applied to the joint with a brush. The two ends of the joint are then placed together and pressed between two iron plates heated to about 30° C.

5. Gutta-percha is dissolved in a mixture of 5 kilos. of carbon bisulphide and 1½ kilos. of turpentine oil until the resulting mass has the consistency of porridge. The surfaces of the joint must be freed from grease before this cement is applied and the joint kept under pressure until the cement is dry.

6. 250 parts of gold size, 60 parts of isinglass, and 60 parts of gum arabic are boiled in a water-bath until a homogeneous mixture is formed; 5 parts of Venetian turpentine, 6 parts of turpentine oil, and 10 parts of spirits of wine are then added. The joint, after cementing, is pressed between hot plates and allowed to get quite cold before the pressure is removed.—R. B. P.

## PATENTS.

*A Process for Clarifying or Decolourising Tannic Extracts.* A. and H. Sinan, Nantes, France. Eng. Pat. 22,480, December 7, 1892.

BLOOD or other gelatinous or albuminous matter (1 litre) is mixed with bicarbonate of soda (1 kilo.) and water (½ litre) and the mixture boiled by injecting steam for one hour. This liquor is poured into the ooze (20°–30° B.) through a sieve, and mixed therewith by the arms of a rotating shaft. The ooze, which need not be heated, is allowed to stand until the colouring matters have been carried to the bottom.

The product thus obtained is said to satisfy all the requirements of the tanning process.—A. G. B.

*An Improved Process to make Leather Soft, Durable, and Waterproof.* W. Becker, London. Eng. Pat. 4274, February 27, 1893.

THE tanned hide is shaved to the required substance and slicked on the grain until every appearance of this "becomes invisible." The hide is then dried, stretched in a frame with the aid of a stretcher (moon) until the hide is thoroughly soft. A second slicking on the grain follows, and the same side is covered with train oil (free from resin) and laid on the table. The flesh is now scoured and greased with a solution of one-half degrass, one-quarter tallow, and one-quarter train oil, previously boiled and allowed to stand for a few days. The hide is next air-dried and worked with the slicker, at first as usual, but afterwards with a sharp slicker in such a manner that the top of the two strata of grain is "abstracted" by the slicker and the underlying stratum left intact. After the grain is split it is blackened. The black constitutes one of the claims, and is prepared by soaking old iron in young beer or sediment of beer for about four weeks; this solution is applied to the hide after it has received a coat of logwood extract. Train oil, applied softly, serves to fix the black, and the hide is now dried. It is next laid on a blanket and rubbed with a "solution of 100 parts of vaseline oil and 30 parts of ceresin mixed with a quarter of an ounce of nigrosin," boiled together into an embrocation. Ironing, slicking, and glassing of the grain finish the process, except that a gloss is imparted to the grain by applying a size made by boiling a gallon of vinegar with half a pound of glue mixed with a quarter of an ounce of nigrosin, and allowing it to cool and stiffen.

The above is for shoe leather; saddlery leather is similarly treated, save that tallow is applied after the blacking.

—A. G. B.

## XV.—MANURES, Etc.

### PATENT.

*Improvements in or relating to the Treatment of Fish and Fish Offal, and in Apparatus therefor.* J. C. W. Stanley, London. Eng. Pat. 17,205, September 13, 1893.

THE fish or fish offal, with the addition of water, is placed in drums provided with air-tight lids, and these are placed in a tank of water provided with a steam coil. The fish is then boiled until the flesh easily separates from the bone, and the mixture of oil and water is then drained from it. The oil is separated from the aqueous extract by an elaborate system of settling tanks and the aqueous extract precipitated by chemical means.

The solid matter, consisting of flesh and bone, after expressing all the water and oil from it, is mixed with water and transferred to a heating machine, the resulting mixture being passed through sieves. The bones in this way are separated from the fibrous matter, which is afterwards retained by passing over strainers, and can then be dried and used for agricultural purposes or otherwise. The aqueous extract is mixed with that obtained in the first process, and the whole is precipitated by chemical means, the resulting precipitate being removed from the water by a series of settling tanks. This precipitate is collected and dried, and is said to constitute a valuable manure.

—J. G. W.

## XVI.—SUGAR, STARCH, GUM, Etc.

*Cause of the Birotation of Glucose (Dextrose).* B. Tollens. Ber. 1893, 26, 1799—1802.

BÉCHAMP (Bull. Soc. Chim. [3], 9—10, 401, 511) attributes the so-called bi-rotation exhibited by freshly-prepared aqueous solutions of glucose (dextrose) to the formation of a hydrate in solution, which gradually dissociates until at the end of 24 hours the solution contains the anhydrous sugar giving the normal rotation; the author has also arrived at similar conclusions. It has already been shown (this Journal, 1892, 944) that when sugars which exhibit bi-rotation in aqueous solution are examined in very dilute ammonia, the normal rotatory powers are at once observed. When anhydrous glucose is dissolved in aqueous ammonia and in water an absorption of heat (depression in temperature) is soon afterwards observed; in the case of water, however, this is followed by an evolution of heat (rise of temperature), due, apparently, to the union of the water and the sugar. No such subsequent rise of temperature is, however, observed in the case of ammonia. The conclusion therefore that the bi-rotating sugar is glucose hydrate appears to be justified.—A. R. L.

*The Soda-Baryta Process.* Vivien. Zeits. Zuckerind. Böhmen, 1893, 18, 70.

In order to render the juice neutral during diffusion, 1 kilo. of soda is added for 1,000 kilos. of roots. The juice is heated to 85° and 1 kilo. of crystallised baryta is added to 1 m. of it. The precipitate when dry amounts to 8—16 grms. per hl. and contains 20—28 per cent. of barium sulphate, 15—20 per cent. of barium as carbonate and salt of organic acids, and 30—40 per cent. of organic substances; the remaining portion consists of lime, iron, and alumina. This precipitate contains 0.3—0.4 per cent. of the organic substances contained in the juice.

The juice obtained by this process is not raised in purity after the second saturation as is that obtained by the ordinary process, and it has a lower salt quotient than the latter.—A. R. L.

*Estimation of the Quantity and Purity of Masecutes, calculated on the Beetroots, obtained in the Manufacture of Granulated Sugar.* F. Hanns. Zeits. Zuckerind. Böhmen, 1893, 18, 5—8.

THE author's notes refer to a certain Russian factory. He shows that during a single campaign there was obtained from 105,431 kilos. of beetroots, 16,170 kilos., or 15.34 per cent., of masecuite of all grades; that is first, second, third, and fourth products and claires. The mean composition of these masecutes was as follows:—93.4 Brix, 83.5 polarisation, and 89.4 coefficient of purity.—A. R. L.

*Use of Sulphurous Acid in Sugar Manufacture.* K. Abraham, Zapiski 1893, No. 4; through Zeits. Zuckerind. Böhmen, 1893, 18, 67—68.

THE author states that sulphurous acid bleaches certain colouring matters, but by no means all, and the decolorising power is often overrated, as the original colour often returns on exposure to air.

As Drenckmann has shown (Zeits. Zuckerind. Böhmen, 1884—5, 422), carbonic acid exerts a greater purifying effect on the juice during the last saturation than sulphurous acid does.

An alkalinity of 0.05—0.06 is readily reduced to 0.03, by saturation with carbonic acid; if however, it is carried beyond that point the juice darkens. An alkalinity of 0.01 cannot be reduced by sulphurous acid because the normal sulphites have an alkaline reaction, and an excess of sulphurous acid causes inversion.

Employing triple-effect apparatus with juice of an alkalinity of 0.01, sulphurous acid also causes chemical changes, and the evaporating apparatus must be cleaned with soda and hydrochloric acid every week, whereas working with carbonic acid this operation is only needful once during the whole campaign.

The author is of opinion that the disuse of animal charcoal is productive of less masecuite and more molasses, and that the same effect is produced by the use of sulphurous acid.—A. R. L.

*Use of Sulphurous Acid in Diffusion.* Mittleman. Zeits. Zuckerind. Böhmen, 1893, 18, 66 (see also this Journal, 1893, 456).

THE uses of sulphurous acid or of acid calcium sulphite in the proportion of 500 grms. of these substances to 10 q. of roots causes the immediate precipitation of albumin and other organic substances, the lixiviation is effected more readily, and the extracted slices keep better without losing their nutritive value. Sulphurous acid when added to the warm juice protects it from undergoing the lactic fermentation. Aulard found, however, that although sulphurous acid exerted a purifying influence on the juice, it caused it to froth.—A. R. L.

*Estimation of the Amount of Sugar Lost during the Boiling of the Juice.* J. Felemann and F. Herles. Zeits. Zuckerind. Böhmen, 1893, 18, 16—21.

THE loss of sugar due to decomposition during the boiling of the juice may be arrived at by systematically testing the juice and masecuite. For example, a thin juice gave on analysis:—Pol. 10.5 per cent., and sulphated soluble ash 0.31 per cent.; whence  $\frac{10.5}{0.31} = 33.87$ . After boiling to concentrated juice, it gave Pol. 46.3 per cent., sulphated soluble ash 1.38 per cent., or a ratio of  $\frac{46.3}{1.38} = 33.55$ . The masecuite gave Pol. 87.5 per cent., sulphated soluble ash 2.67 per cent., or  $\frac{87.5}{2.67} = 32.77$ . It therefore follows that by evaporating the thin juice to concentrated juice  $33.87 - 33.55 = 0.32$  part of sugar was decomposed, therefore  $(33.87 : 0.32 = 100 : x)$  0.24 per cent. Supposing the thin juice to have been derived from beetroots containing 12.3 per cent. of sugar, the loss of sugar on the roots would

amount to 0.11 per cent. In a similar way, by the conversion of the concentrated juice into massecuite,  $33.55 - 32.77 = 0.78 = 2.3$  per cent., or 0.28 per cent. on the roots. The total loss of sugar by evaporating and boiling, calculated on the roots, is therefore  $0.11 + 0.28 = 0.39$  per cent.

—A. R. L.

*Purification of the Juice in Triple-Effect Apparatus.*  
Mittlemann. Zeits. Zuckerind. Böhmen, 1893, 18, 66—67.

THE author has observed that in proportion as the evaporating apparatus becomes coated with deposits, the alkalinity of the juice sinks and its purity increases. In support of this a table is given showing the results of 15 consecutive days' work, commencing from the day on which the apparatus was cleaned. At the end of the 15th day, although the purity quotient of the juice was 94, as against 90 on the first day, the juice was darker in colour.

Bouvier remarked that the deposits in question consisted mostly of calcium salts; the harmful influence of these on the effect of the evaporating apparatus can be overcome as follows:—In the last member of the evaporating apparatus there are placed iron shavings which have been previously dipped in milk of lime and then treated with carbonic acid. The presence of these incrustated shavings induces the separation of calcium salts up to a certain point, when they are removed and replaced by fresh ones.—A. R. L.

*Defecation of Diffusion Juice with Pulverised Slaked Lime.* Mittlemann. Suer. Belge, 1893, Nos. 14 and 15; through Zeits. Zuckerind. Böhmen, 1893, 18, 64—66.

THE use of slaked lime for defecating the juice has the advantage over the employment of milk of lime, that it dilutes the juice to a less extent, thus:—To 120 litres of juice there was added 16.8 litres of milk of lime containing 16.06 litres of water, whilst by employing 4.07 kilos. of slaked lime containing 15 per cent. of water, the added water amounted only to 0.61 kilo., or 15.45 kilos. less than in the case previously cited. The juice appears to have equal purity as when milk of lime is used. Indeed, during the last campaign results have been obtained which have only seldom been reached. It is said that pulverised lime is injurious to the health of the workmen, but this objection does not apply to slaked lime containing 15 per cent. of water. Before adding the slaked lime the juice is

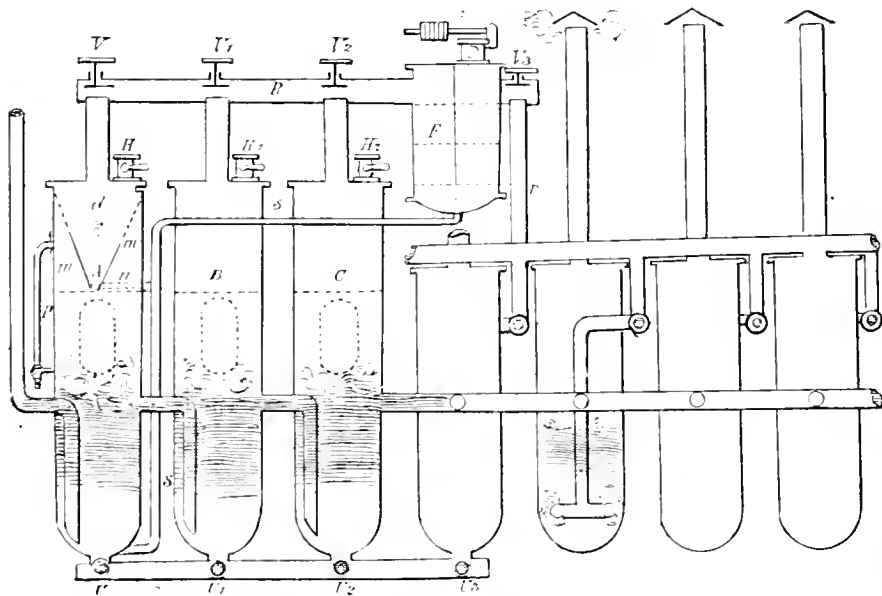
warmed to  $84^{\circ} - 85^{\circ}$  C. In the course of the discussion Vivien agreed with the author as to the advantages of employing slaked lime for defecating the juice, and as a matter of fact Dervan and Ibled had patented a similar process in 1871. Mauoury said that both caustic and slaked lime had been in use for 10 years in 40 sugar factories in Germany, Austria, and Russia. Bouvier recommended introducing the lime into the juice immediately after it had been slaked in baskets. Dissolution occurs in two minutes, and when a certain amount of fat is also added no scum is formed. No great rise of temperature accompanies the dissolution of the lime. Saturation can be commenced at the expiration of five minutes. The once saturated juice is boiled energetically with addition of lime and then saturated a second time, the following being some of the results obtained:—

Salt Quotients.					
Diffusion juice.....	24.29	24.34	23.15	19.30	22.51
After second saturation	32.85	31.60	31.53	27.38	28.24
Increase.....	8.66	7.26	8.48	7.48	5.73

—A. R. L.

*Continuous "First Saturation," Avoiding the Loss of Carbonic Acid.* T. Franc. Zeits. Zuckerind. Böhmen, 1893, 18, 1—3.

SINCE animal charcoal has fallen into disuse the most vexed question with regard to the purification of the sugar juice is the "first saturation." The saturation gas employed must be as rich as possible in carbonic acid. On this account large lime kilns furnished with ingenious heating arrangements have been erected. Endeavours have especially been made on all sides to prevent as far as possible the great losses of carbonic acid which are occasioned by the use of the existing forms of saturation apparatus. Indeed, experiments with a gas containing 26 per cent. of carbonic acid proved that 9 per cent. of the saturation gas was lost at the commencement of the operation and 19 per cent. at the end. The present invention was devised to obviate this loss, and the author points out that not only is the latter object attained, but the work is expedited.





The "first saturation" is divided into two stages:—(1.) A continuous preliminary saturation with the original gas. (2.) The employment of the gas which escapes from the first operation to finish the process.

Presuming the presence of a mixer, the juice, after mixing with lime, is led into the three high saturators A, B, C, where it rises from the first into the second, and thence into the third vessel, a continuous flow of gas being maintained meanwhile. Both the inflow and the outflow pipes are furnished with valves. From these preliminary saturators the juice enters one of the finishing saturators. The gas evolved from the preliminary saturation passes through the pipe R into the froth retainer F, in the first division of which it takes a downward direction, and in the second an upward direction, thereby destroying the froth, which is returned as juice by the tube S to the preliminary saturator, whilst the gas passes through the tube r to the finishing saturators. The tube R is furnished with valves  $V_1, V_2$ ; the tube r has likewise a valve  $V_3$ . The finishing saturator has also an independent gas and juice delivery tube, so that when desired the saturation can be conducted in the old way. When, for example, the preliminary saturator A is being cleansed the inflow valve is closed from the mixer, the juice run into the finishing saturator, and the operation conducted in the old way. The valves  $V$  and  $V_3$  are closed, whilst A is placed in connection with the empty finishing saturator No. 1, by opening the valves U and  $U_3$ , and the contents of A forced into the vessel No. 1, by means of gas. The same applies to the cleansing of the other two saturators.—A. R. L.

*Filter for Diffusion Juice.* Bouvier. Zeits. Zuckerind. Böhmen, 1893, 18, 64.

THE author makes the following remarks regarding the filtration of the diffusion juice after heating it to  $80^\circ\text{C}$ . and precipitating the albumin:—To assist filtration 1–2 per cent. of fresh saturation sludge (Jopolsky's process), animal charcoal dust or pulverised "Eskarbil" is added. The author has constructed a filter, the upper portion of which is furnished with a removable sheet-iron retainer, having a perforated or wire-gauze sieve which keeps back the pulp and fragments of root slices. When the perforations become clogged the sieve is replaced by another similar one. The under portion of the filter has a double bottom, the upper part of which is perforated. As filtering medium a layer of coarse sand, animal charcoal, wood shavings, or the like, is used. The apparatus is furnished with paddles, which prevent the deposition of the mud. The filtering medium is washed with hot water.—A. R. L.

*Characteristics of Glucose Syrups.* W. E. Stone and C. Dickson. J. Anal. and Appl. Chem. 1893, 7, 317.

See under XXIII., page 178.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*Butyl Alcoholic Fermentation, and the Organism which causes it.* M. W. Beyerinck. Rec. Trav. Chim. 12, 141–153.

THE organism supposed by Fitz (Ber. 1882, 15, 867) to be the ferment of normal butyl alcohol was in reality the butyric ferment. Both resemble one another, and belong to a new species of *Granulobacter*, so called because they contain granulose.

The true butylic organism—*Granulobacter butylicum*—was isolated by the author from certain hulled barley—*Hordeum distichon nudum* and *Hordeum vulgare hivalense*. About 20 grms. of the grains are ground, and the flour made into a paste with 100 cc. of boiling water, so that the

temperature of the paste does not rise above  $95^\circ$ , a precaution which it is necessary to observe, as the spores of the butylic organism are killed when heated for a few seconds at  $95$ – $100$ . Other organisms are, however, destroyed below  $95$ . Fermentation is allowed to proceed at  $35$ . After 48 days the starch liquefies, gas is evolved, and the odour of butyl alcohol is apparent. The spores of *Bacillus subtilis* are often present in the fermenting liquid, but these exert no ill-effect on the butylic organism, which is anaerobic and grows in the lower portion of the liquid. When, however, the butyric organism, which is also anaerobic, is present, the fermentation of *G. butylicum* is arrested.

Pure cultures are now made in sterilised fairly acid beer wort in vessels which admit of the exclusion of air; fermentation is conducted at  $35$ , and lasts about 60 hours. The yield of butyl alcohol is about 1–2 per cent. of the flour employed, and the other products are carbonic anhydride and hydrogen; volatile organic acids are quite absent, which distinguishes the organism from the butyric ferment. The organism can be precipitated from the liquid by addition of ordinary alcohol, sufficient of this being added until the mixture contains 70 per cent.; after being dried in the air it forms a brown powder, which is coloured blue by iodine. In this condition the bacteria may be kept for six years without losing their vitality. The organism contains an enzyme—*amylase*—which is secreted upon the outer surface of the bacteria, and which converts starch into maltose and dextrin like the diastase of germinated grain. The granulose mentioned above is deposited in the cell contents of the bacteria.

The spores of the organism retain their vitality in the moist condition for three months only, whilst the bacteria themselves die earlier; the amylase then penetrates the cells and saccharifies the granulose. The bacteria are therefore best preserved in the dry condition.

The author then states that he has recognised two forms of the organism, one which propagates in an atmosphere of oxygen under reduced pressure—the "oxygen form"—and one which is truly anaerobic—the "clostridium form." The former is not easily distinguishable from *Bacillus subtilis*, is very mobile, and is coloured yellowish-brown by iodine; hence it does not contain granulose. When the pressure of oxygen exceeds a certain limit the bacteria become filiform, and finally multiplication is arrested.

The butylic fermentation always commences with a period in which the "oxygen form" predominates, the composition of the evolved gases being  $\text{CO}_2 + 4\text{H}_2$ . When the last trace of oxygen has disappeared, the clostridium form makes its appearance, the development of hydrogen diminishes, and that of carbonic anhydride increases until, at the close of the fermentation, the composition of the evolved gases is  $5\text{CO}_2 + \text{H}_2$ . The principal phase of the fermentation is characterised by the abundance of the "clostridium form." The spindles have at one extremity an ellipsoidal cavity which encloses a spore of the same shape; these spores may be heated at  $100^\circ$  for some seconds without being killed. The anaerobic cultures of the ferment are best obtained at a temperature of  $10$ – $15^\circ$  with solutions of glucose or starch in water, to which 0.05 per cent. of potassium hydrogen phosphate has been added.—A. R. L.

*The Glucosides of the Alcohols.* E. Fischer. Ber. 1893 26, 2400–2412.

THE only method known at present for the artificial preparation of glucosides is that devised by Michael (Comptes rend. 89, 355), which depends on the interaction of aceto-chlorhydrase with the alkali derivatives of phenols. It is, however, restricted to the latter compounds; and, on account of its complexity and the poorness of the yield of product, it has only been employed with success in a few cases. The author finds that when hydrogen chloride is passed into a solution of glucose in methyl alcohol the methyl derivative  $\text{C}_6\text{H}_{11}\text{O}_6 \cdot \text{CH}_3$ , which he proposes to call methyl glucoside, is formed. It is best prepared by mixing a very concentrated aqueous syrup of glucose with a saturated solution of hydrogen chloride in methyl alcohol. It is a crystalline compound having a

sweet taste; it melts at  $165^{\circ}$ — $166^{\circ}$ , and is readily soluble in water. The solution is dextro-rotatory—

$$[\alpha]_{D_{20}} = +157.5.$$

but it does not reduce Fehling's solution.

The reaction above indicated seems to be available for all alcohols; in the case of glucose it has been proved with ethyl, propyl, amyl, isopropyl, allyl, and benzyl alcohols, as also with ethylene glycol and glycerol. In certain cases it is rendered difficult on account of the small solubility of the sugar in the alcohol. It may even be employed for the hydroxy acids, as was proved by the preparation of lactic glucoside. When the sugar is completely insoluble in the alcohol, which is the case with many compounds of the aromatic and terpene series, the method cannot be employed, but in such cases, in the place of glucose, acetochlorhydrate, or pentaacetylglucose, both of which are readily soluble in ether, benzene, and chloroform, may be used. The acetyl groups are here split off by the hydrochloric acid, and the products are those already mentioned. The author speculates upon the probable constitution of these compounds and of the sugars from which they are derived, but no definite conclusions are arrived at. It is interesting to observe that he regards their structure as analogous to that of the bioses—maltose, milk sugar, &c. The latter are made up of two hexose residues, whilst the new compounds are made up of a hexose residue and a simple alkyl group.

The bioses are hydrolysed by hydrochloric acid, and cannot therefore be converted into glucoside-like compounds by the method above indicated.

The new synthetic compounds resemble the natural glucosides in their properties. They are unattacked by boiling alkalis, Fehling's solution, and phenylhydrazine; if, however, they be heated with dilute acid they are readily hydrolysed into their components: they are likewise hydrolysed by the invertase of yeast. *Benzylglucoside* and *ethylrhannoside* have a bitter taste; and it would seem possible that many naturally occurring bitter principles may belong to this class of compounds.

Gautier's so-called diglucose (Bull. Soc. Chem. 1874 [2] 22, 145), which he obtained by saturating an alcoholic solution of glucose with hydrogen chloride, is in reality ethyl glucoside; this being so, the only hexabiose which has up to the present been obtained synthetically, is the author's isomaltose.—A. R. L.

#### PATENTS.

*Improvements in Preserving Yeast for Transport.* J. Heron, Battersea. Eng. Pat. 3077, February 11, 1893.

THE yeast is pressed and mixed with a solution of glucose containing only about 10 to 12 per cent. of water, or it is submitted to more than the ordinary pressure so as to make it drier than is customary, and mixed with a solution of glucose of the concentration usually employed for brewing purposes. The two are thoroughly incorporated, and the mixture put into moulds and left to set. A mixture of one part by weight of ordinary pressed yeast and four parts by weight of extra concentrated glucose is recommended.—J. G. W.

*Treating Brewers' Spent Hops, Wet and Dry, for the Production of Fodder and Litter.* J. J. Adkins, London. Eng. Pat. 17,268, September 14, 1893.

FOR use as a substitute for ensilage the wet and fresh hops are flavoured with essences of caraway, coriander, and aniseed, with the addition of fennigreek and salt. As a substitute for hay the hops are dried and flavoured with the same mixture. For use as litter the hops are simply dried.

—J. G. W.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY AND WATER PURIFICATION, DISINFECTANTS.

### (A).—CHEMISTRY OF FOODS.

*The Sterilisation of Bread and Biscuit after Withdrawal from the Oven.* Balland and Masson. Comptes rend. 1893, 117, 797.

See under XVIII. B., page 169.

*The Determination of Casein in Cows' Milk.* L. L. Van Slyke. J. Amer. Chem. Soc. 15, 635.

See under XXIII., page 181.

#### PATENTS.

*A Method of and Apparatus for Preserving Meat, Cereals, and other Animal or Organic Substances; Destroying Parasites and Vermin, Extinguishing Fires, and Disinfecting Leather, Fabrics, and the like.* J. Vigerie, Lille, France. Eng. Pat. 2694, February 7, 1893.

THESE several purposes are effected by the use of carbon dioxide under pressure, obtained from a cylinder of the liquid gas, the apparatus for preserving substances consisting simply of a receptacle for those substances, into which carbon dioxide can be forced. The apparatus for extinguishing fires is merely an arrangement for forcing carbon dioxide gas into water, and, when required, forcing out the water thus impregnated with the gas by means of the expansion taking place during the change from the liquid to the gaseous state.—J. G. W.

*An Improved Cattle Food.* G. Holland, Glasgow, Lanark, N.B. From G. Holland, Ahlfelde, Prussia. Eng. Pat. 3526, February 17, 1893.

THIS invention relates to a cattle food prepared by mixing molasses or treacle (100 parts) with maize, rice, wheat, rye, or bran, dried beer dregs, or any other feeding meal (100 parts) in a kneading machine; the dough is pressed into flat cakes and baked in an oven or drying apparatus, after which the cakes are ground and mixed in a dry condition with the ordinary food. The molasses food may also be made in a dry, loose state, the proportion of the bran or feeding meal being then 100 to 70; the mass is also worked up with the kneading machine. After standing for some time the dough is laid upon plates and baked.

—A. R. L.

*Improvements in Fluid Meats and the Process of Manufacturing Same.* W. Clark, Montreal. Eng. Pat. 4486, March 1, 1893.

LEAN beef is cut up into small pieces and cooked in a digester kettle. When finished, the liquor is separated from the beef by straining. The beef is minced and reduced to a homogeneous mass by passing through a series of perforated plates, and then mixed with the liquor, formerly separated, after the latter has been skimmed to remove all fat. To this mixture is added 10—15 per cent. of "essence of wheat" (Eng. Pat. 4487, 1893; see succeeding abstract), and the whole thoroughly amalgamated. This mixture is again mixed with an equal quantity of a gelatinous extract of beef, obtained by boiling the sinew and muscle in a digester kettle under pressure. It is claimed that the advantages of this combination of ingredients are that the nutritious constituents, which are necessarily insoluble, remain in suspension for a considerable time, while at the same time a rich natural beef flavour is secured and ready digestibility attained.—J. G. W.

*Improvements in Food Preparations and in their Manufacture.* W. Clark, Montreal. Eng. Pat. 4187, March 1, 1893.

WHEAT germs are soaked in water for two hours, and the solution, which contains the soluble albuminoids, drawn off. This is mixed with extract of meat (Eng. Pat. 4486, 1893; see preceding abstract); or it is coagulated under pressure and the resulting clot subjected to pressure in a press, and in this dry state incorporated with solid meat-extract.

—J. G. W.

*Improvements in the Treatment and Packing of Meats or like Articles of Food for Rendering them fit for Use for Army Rations or Analogous Uses.* P. Pfeleiderer, London. From C. J. H. Warden, Bengal, and W. A. Billings, Meerut. Eng. Pat. 6663, March 29, 1893.

MEAT, from which all fat, skin, and bone have been removed, is minced and pressed to express as much as possible of the juices. The cakes thus produced are dried in a current of warm, dry air, at a temperature rising from 120°—212° F. The expressed juices are concentrated in a vacuum-pan to the consistency of treacle, incorporated with the coagulated albumen, and then intimately mixed with the dried meat-fibre and formed into cakes, which are desiccated at a temperature of 160°—170° F. These cakes are coated with a layer of fat by plunging into a bath of melted meat-fat or butter, and are packed by enclosing each cake in paper impregnated with fat, and the whole in lead or tin foil, which can be soldered at the seams. The whole is enclosed in strong parchment paper, cemented at the joints by a solution of gelatin and potassium bichromate, which is afterwards exposed to the light to render it insoluble.—J. G. W.

#### (B.)—SANITARY CHEMISTRY AND WATER PURIFICATION.

*The Sterilisation of Bread and Biscuit after Withdrawal from the Oven.* Ballard and Masson. Comptes rend. 1893, 117, 797—799.

THE paste used in the manufacture of the regulation military bread has a mean acidity equal to 0.15—0.2 per cent., or on the dry substance 0.29—0.38 per cent. Calculated as sulphuric acid, the acidity of the crumb after baking will be approximately the same. The authors find that the combined effects of this acidity and the temperature of baking are generally sufficient to render the bread sterile; only certain spores which are resistant to elevated temperatures, survive, and ultimately, under favourable conditions, develop. In the case of bread manufactured with yeast the acidity is less, and consequently the sterilisation is not assured to the same degree. In all cases, however, pathogenic germs, notably the bacillus of typhoid and cholera, which offer less resistance to heat, ought to be destroyed.—A. R. L.

#### PATENTS.

*Improvements in Filters for the Removal of Bacteria and Germs from Water and Sewage.* F. P. Candy, Batham, Surrey. Eng. Pat. 1448, December 9, 1893.

THE object of the process is to provide a permanent filter bed for the retention of bacteria and germs, independent of deposition from the impure water, the accumulation of which is necessarily uncertain. Oxides, or better hydroxides, of iron, manganese, or aluminium, carbonised or calcined iron carbonate, or other suitable materials, are finely powdered and passed through a sieve having about 60 meshes to the lineal inch, and mixed with about the same bulk of wet sand. A layer of half an inch or more of the mixture is placed on the body of the filter bed.

It is preferable to remove any suspended matter from the water or sewage before running on to the above filter. Besides bacteriological purity, increased chemical purity is nearly always obtained.—N. H. J. M.

*Improvements in the Method of and Apparatus for Purifying Water.* A. T. Walker, Reading, Berks. Eng. Pat. 2807, December 9, 1893.

THE water first passes through a series of upright sieves, which may be readily removed and replaced. It then passes through pipes into a tank, from which it overflows into a second tank containing clean furnace or other coke, resting on perforated bricks, under which the water passes into a third tank, from which it overflows into a fourth chamber containing granulated "polarite." After the water has passed downwards through the "polarite," it enters a fifth chamber, whence it overflows into the sixth compartment, which is also filled with "polarite." The water is finally collected in reservoirs for distribution. The polarite beds are not less than 2.5 ft. deep, but of comparatively small superficial area.—N. H. J. M.

*Improvements in Apparatus for Filtering and Purifying Water.* W. Webster, Blackheath, Kent. Eng. Pat. 1676, December 9, 1893.

THE apparatus consists of a vessel containing black oxide of iron, connected from below by means of a long, narrow tube with a small chamber having a perforated bottom and charged with spongy platinum. The long connecting tube has a lateral branch terminating in a short tube parallel to the long tube. The upper end of the short tube is drawn out so as to leave a small opening, whilst the lower end is connected with an inverted bell-jar, which dips into a small water tank provided with a suitable electrolysis apparatus.

The water first filters through the iron oxide, passes down the long tube, where, at the point of junction with the lateral tube, it carries down bubbles of air, and oxygen, or oxygen alone, as may be required. On coming into contact with the spongy platinum it is sufficiently charged with oxygen to effect the oxidation of any organic matter which may be present.—N. H. J. M.

*An Improved Apparatus for Obtaining Distilled and Sterilised Water.* J. Nagel, Chemnitz, Germany. Eng. Pat. 20,296, December 9, 1893.

THE apparatus consists of a boiler with heating tubes on Field's system, an upcast tube through which the steam passes into an upper chamber. This chamber is provided with a sieve, through which the partly-condensed steam passes downwards through a set of tubes surrounding the upcast tube and enclosed in a cylinder kept cold by running water. At the lower end of the tubes there is a reservoir for collecting the distilled water. The water used for cooling may be used for distillation.

When water is to be merely boiled, the upcast tube is closed with a valve. The pressure of the steam forces the water through a smaller tube into the upper chamber, from which it flows downwards through the sieve and the cooling pipes into the collector.—N. H. J. M.

*Process for Treating the Effluent from Electrolytically or Chemically Purified Sewage for Neutralising its Alkalinity and for the Production of By-Products therefrom.* W. Webster, Blackheath, Kent. Eng. Pat. 1675, December 9, 1893.

THE effluent from the purified sewage is treated with Portland cement, fluxed lower-grey chalk ("soap"), or caustic lime previously suspended in water. The amount of substance required varies from 8 grains to 100 grains per gallon. In subsiding, the solid matter carries down the suspended matter. The effluent is then brought into intimate contact with the gaseous products from the burning of Portland cement, namely, sulphurous and carbonic anhydrides and carbonic oxide. This is effected by

running the water over a series of shallow trays in a closed vessel, through which the gases are made to pass in a contrary direction; or else by letting the water fall from the top of a tower, through which the gases pass upwards. In this manner the alkalinity of the water is neutralised, and any lime remaining in suspension precipitated. The precipitates are converted into cement by burning.

—N. H. J. M.

### (C).—DISINFECTANTS.

*Stability and Preservation of Dilute Solutions of Corrosive Sublimates.* L. Vignon. *Comptes rend.* 1893, **117**, 793.

See under XX., page 171.

### PATENT.

*Improvements in Disinfecting.* E. Hermite, E. J. Paterson, and C. F. Cooper, London. Eng. Pat. 18,370, December 16, 1893.

The authors have already described apparatus for electrolysing bleaching liquors containing chlorides, the manufacture of a liquor for disinfecting and bleaching purposes by electrolysis, the disinfecting of impure liquids by electrolysis in presence of chlorides, and apparatus for electrolysing sewage (compare this Journal, 1887, 126, 321, and 764).

By the method now described, an electrolysing plant, as already described (*loc. cit.*), is provided with reservoirs, where water to which chlorides have been added, or sea water without further addition, is electrolysed. The water is then conducted, as a disinfectant, to drains, closets, and other places as required. The liquid need only contain about 0.1 per cent. of chlorine, and its use is therefore not attended with any unpleasant odour.—N. H. J. M.

## XIX.—PAPER, PASTEBOARD, Etc.

*Brown Paper from Wood.* Papier Zeit. **18**, 2653.

A WOOD-PULP boiler made by G. Schumann, of Zeitz, is described and illustrated. It is constructed of wrought iron and lined with sheet copper to protect the shell from the action of the organic acids from the wood.

The most usual size is 4.5 metres long, by 1.5 metre diameter, and 8 cb.m. capacity. Steam of 6 atmospheres pressure is used, and is left on for from 8 to 18 hours. The longer the steaming, the darker the colour of the material produced. Water of condensation is removed at the bottom, and with it the oil of turpentine from the wood. The turpentine is sometimes recovered and used. In order to cool down the boiler and wash the wood, the former is filled with water and emptied two or three times before the contents are removed. The lining is 2.5 and 6 mm. thick, and is attached to the iron shell by copper-plated, or solid copper rivets; at the manholes the lining is flanged over. In order to test the tightness of the copper lining, holes are pierced through the iron shell, and water pumped in, when, if the lining be not tight, water will leak into the interior, and thus indicate the leaks.—R. B. P.

### PATENTS.

*Coating or Covering Paper with Aluminium.* W. Ibbotson, London. Eng. Pat. 23,603, December 22, 1892.

THE surface of the paper is coated with adhesive matter, and the latter covered with laminated or powdered aluminium, or powdered aluminium is mixed with adhesive matter and spread on the surface of the paper by brushes or any other manner convenient. The claim is "Paper coated with aluminium."—E. G. P. T.

*Improved Process for Treating the Black Ash Residuum of Wood Pulp Manufacture.* L. S. Langville, New York, U.S.A. Eng. Pat. 18,519, October 3, 1893.

THIS invention relates to a process described in Eng. Pat. 13,847, 1892 (this Journal, 1892, 935), for the production of a carbon product obtained from the black ash resulting as a residuum of wood pulp manufacture.

By the new process, the black ash residuum is mixed with water and caused to flow through a trough with riffles in its bottom at right angles to the trough; the carbon, being lighter than the foreign substances mixed with it, floats, while the impurities sink and are caught in the riffles. The liquor running from the tank is treated with weak hydrochloric acid, and the carbon washed by the aid of steam heat, dried, and powdered. It is then well adapted for making electric light carbons and India ink, and as an ingredient of writing ink, gunpowder, paint, &c.

—E. G. P. T.

*Improved Sensitised Paper for Photographic Purposes.* A. Hiesekiel, Berlin. Eng. Pat. 20,042, October 24, 1893.

THE improved paper is prepared in the usual way, but of sufficient substance to receive the impress, from rollers or otherwise, of a granular surface; the object being so to break up the surface of the paper as to give the printed matter or photograph the appearance of line or other engraving or etching effect.—E. G. P. T.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Formation of Ozone at High Temperatures.* O. Brunck. Ber. 1893, **26**, 1790—1794.

IT is a well-known fact that oxygen prepared by heating a mixture of potassium chloride and manganese dioxide has an intense odour resembling that of chlorine, and statements as to the amount of chlorine in oxygen so prepared are very numerous. If the intense odour were really due to the production of chlorine in the reaction, the residual mixture of salts should give on extraction with water an alkaline solution containing potassium manganate, whereas experiments have convinced the author that the aqueous extract is invariably neutral and colourless. This circumstance, together with the properties of the gas of rendering potassium iodide-starch paper blue, suggested that the gas might contain ozone, which supposition was confirmed. (Compare McLeod, *Proc. Chem. Soc.* 1894, and March number of this Journal, under XX.)

A series of experiments led to the result that ozonised oxygen can be heated for a short time not only to the decomposition point of potassium chlorate, but also at a red heat, without destroying the ozone. The gas evolved from potassium chlorate and manganese dioxide after washing it with alkali, not only liberates iodine from potassium iodide, but also oxidises alcohol to aldehyde; it loses its odour and oxidising properties when passed over a short layer of manganese dioxide at the ordinary temperature. The active oxygen thus produced appears to differ somewhat from ordinary ozone, thus:—Its odour is exactly that of chlorine; it withstands a higher temperature, and does not attack mercury nor organic substances, such as caoutchouc, so readily as ordinary ozone. Pure potassium chlorate yields no ozone, but the presence of the least trace of indifferent substances, such as silica, causes its formation. A mixture of equal parts of potassium chloride and manganese dioxide yielded a gas containing 0.3 per cent. of active oxygen; whilst when the relative amount of manganese dioxide used was 25 times that of the chlorate, the gas contained 1.55 per cent. of active oxygen. A preliminary account is given of experiments, now in progress, on the behaviour of manganese dioxide and other oxides at high temperatures.—A. R. L.

*Basic Bismuth Gallate (Dermatol).* H. Causse. *Comptes rend.* 1893, **117**, 232—234.

Basic bismuth gallate, better known as dermatol, was obtained by R. Fischer (*Pharm. Zeit.* 1891). The author has prepared it in small yellow crystals of the composition  $C_7H_5O_3Bi + 2H_2O$  by adding gallic acid to a solution containing bismuth and potassium nitrates in acetic acid. It is insoluble in water, but dissolves in mineral acids, and is very sensitive to the action of light and air. *Bismuth pyrogallol*,  $C_6H_3O_3Bi$ , is obtained in a similar manner when pyrogallol is substituted for gallic acid.—A. R. L.

*Stability and Preservation of Dilute Solutions of Corrosive Sublimate.* L. Vignon. *Comptes rend.* 1893, **117**, 793—795.

When a solution containing 1 grm. of mercuric chloride per litre is kept in an open vessel at a temperature of  $15^{\circ}$ — $20^{\circ}$ , a white precipitate forms and the titre of the solution rapidly diminishes, and consequently also the antiseptic value; the same phenomenon is observed if the solution be kept in a closed vessel, but the change is less rapid. The presence of colouring matters appears to retard the alteration of the solution, and in this respect indigo carmine is superior to fuchsin.—A. R. L.

*Some Decompositions of Quinine.* Zd. H. Skraup. *Monatsh. Chem.* **14**, 428—434.

The action of reagents on the trihydriodide of quinine,  $C_{20}H_{24}N_2O_2 \cdot 3HI$ , resulting in the elimination of iodine, gave rise to a number of mixtures of bases, some of which mixtures were considered to be new compounds (*cf.* *Monatsh. Chem.* **12**, 688; this *Journal* 1892, 263). Their separation was a matter of some difficulty, and hence certain of the statements in the preceding paper require modification. Two compounds *pseudoquinine*,  $C_{20}H_{24}N_2O_2$ , and *nichine*,  $C_{19}H_{23}N_2O_2$ , have, however, been isolated, and these bases, together with regenerated quinine, are always formed in varying quantities when the above trihydriodide is treated with various reagents. *Nichine* can be isolated by boiling the nitrate of the base,  $C_{20}H_{23}IN_2O_2$ , for some time with dilute alcohol. The formation of *nichine*,  $C_{19}H_{23}N_2O_2$ , from quinine,  $C_{20}H_{24}N_2O_2$ , appears to be accompanied by an evolution of formaldehyde. *Pseudoquinine* trihydriodide, like quinine trihydriodide, yields with alcoholic potash a mixture of *nichine*, *pseudoquinine*, and quinine, so that the two latter bases can be mutually converted into one another. *Nichine* trihydriodide, on the other hand, when similarly treated, yields principally *nichine*, together with a small quantity of a new base, *isonichine*,  $C_{19}H_{23}N_2O_2$ .

For decomposing the trihydriodide of quinine with alcoholic potash, one part of the substance is heated for rather more than an hour with 14 parts of a mixture of equal weights of alcohol and water containing 1.5 parts of caustic potash. The solution is then evaporated to half its bulk, and the bases which separate are dissolved in 7 parts of alcohol and 0.75 part of oxalic acid are added, when acid *nichine* oxalate separates; on further evaporation, acid *pseudoquinine* oxalate separates out, and the mother-liquor contains quinine. *Nichine*,  $C_{19}H_{23}N_2O_2$ , crystallises in needles with  $2H_2O$  and melts at  $102^{\circ}C$ . It becomes anhydrous at  $100^{\circ}C$ , and then melts at  $146^{\circ}C$ . In a dilute solution in presence of sulphuric, nitric, or oxalic acids, but not of hydrochloric acid, it shows a blue fluorescence, and it also gives the quinine reaction with chlorine water and ammonia. Its solution in alcohol is levo-rotatory—

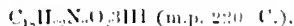
$$[\alpha]_D^{20} = -129.021^{\circ}.$$

On warming the base with 5 parts of hydriodic acid of sp. gr. 1.7, it is converted into the compound—



which is insoluble in alkalis. If this compound be decomposed with dilute alcoholic potash, two-thirds of the *nichine* can be precipitated with oxalic acid, whilst a new base, *isonichine*,  $C_{19}H_{23}N_2O_2$ , which melts at  $209^{\circ}C$ , and crystal-

lises in scales, can be isolated from the mother-liquor. By heating *nichine* with 10 parts of hydriodic acid of sp. gr. 1.7 it loses one methyl group giving—



*Pseudoquinine*,  $C_{20}H_{24}N_2O_2$ , melts at  $191^{\circ}C$  and fluoresces like quinine in solutions containing nitric, sulphuric, or oxalic acids, but not with hydrochloric acid, and it also gives the reaction of quinine with chlorine water and ammonia. One hundred parts of alcohol at  $22^{\circ}C$  dissolve 0.363—0.388 part, and  $[\alpha]_D^{20} = -161.41^{\circ}$ .

The trihydriodide is possibly identical with that of quinine. (See also this *Journal*, 1893, 707.)—T. A. L.

*The Behaviour of the Calcium Salt of Veratric Acid on Dry Distillation.* W. Heinisch. *Monatsh. Chem.* **14**, 455—461.

On briefly summarising the results of the investigation, it would appear that the calcium salt of veratric acid, when subjected to dry distillation under the conditions observed, gave the methyl ether of veratric acid and veratrol as chief products, together with a little guaiacol and veratric acid. The ether formed the largest proportion. Only small quantities of pyrocatechol could be detected in the residue. In connection herewith it may not be inopportune to call attention to the fact that phenol is found in the distillate when the calcium salt of anisic acid is distilled.—T. A. L.

*Rhodinol: the Chemical Principle contained in the Liquid Portion of Otto of Roses.* P. Barbier. *Comptes rend.* 1893, **117**, 177—178.

Eckart has shown (this *Journal*, 1892, 265) that rhodinol,  $C_{10}H_{16}O$ , is a primary alcohol containing two ethylenic bonds and giving valeric acid on oxidation. It is isomeric with linalcol and geraniol. Rhodinol is an oily liquid boiling at  $126.5^{\circ}$  under a pressure of 16 mm., and having a specific gravity at  $0^{\circ} = 0.8956$ ; its refractive index for  $\lambda$  645 is  $n = 1.4653$ , and for  $\lambda = 452.6$   $n = 1.4795$ , and its specific rotatory power is  $[\alpha]_D^{20} = -2.37^{\circ}$ . Hydrogen chloride acts on rhodinol as a dehydrating agent, producing dipentene dihydrochloride. If rhodinol be heated in a sealed tube at  $180^{\circ}$  with acetic anhydride, the acetyl derivative  $C_{10}H_{17}(OC_2H_5O)$  is formed; it is a colourless, mobile oil boiling at  $131^{\circ}$  under a pressure of 15 mm., and having a specific gravity at  $0^{\circ} = 0.9214$ ; its refractive indices are  $n = 1.4526$  for  $\lambda = 645$ , and  $n = 1.4661$  for  $\lambda = 452.6$ . When the acetyl derivative is treated with alcoholic potash, rhodinol is regenerated, so that of the two theoretically, possible stereochemical formulae, rhodinol must possess the stable form.—A. R. L.

*Mercury Gallate: A New Antisyphilitic.* Brousse and Gay. *Comptes rend.* 1893, **117**, 284—285.

The successful results obtained by numerous experimenters in the treatment of syphilis by mercury tannate, suggested the use of mercury gallate for the same purpose. The compound was prepared by titrating gallic acid (37.6 grms.) and yellow mercuric oxide (21.6 grms.) with water (25 cc.), allowing the paste remain for two days, reducing the dried mass to powder, and finally drying it over concentrated sulphuric acid. It is of a dull greenish-black colour, and contains 37 per cent. of mercury. The compound is administered in the form of pills, which are made by kneading mercury gallate (0.05 grm.) and extract of cinchona (0.1 grm.); the dose is 2–4 pills *per diem*. The absorption is rapid, and mercury may be detected in the urine 24 hours after the first dose. As a net result, mercury gallate is found to be a powerful antisyphilitic comparable with mercuric chloride or iodide, and having the advantage over these compounds in that the amount of mercury which the therapeutic dose contains, is not dangerous.—A. R. L.

*Manufacture of Morphine Sulphate.* J. Calvert. J. Anal. and Appl. Chem. 1893, 7, 331—333.

THE liquid resulting from the extraction of opium with water is precipitated with an alkali, and the crude base taken up with dilute sulphuric acid, the solution filtered through animal charcoal, and concentrated to 4.5 to 0.5° B., after which it is run into the crystallising apparatus. The latter is a leaden tank, about six inches in depth, the other dimensions being commensurate with the volume of the solution; it is hinged on a frame, and has an outlet with a plug at the lower end for drainage. "The solution is stirred gently every few minutes until crystals commence to form on the surface, and then, carefully stirring, when the solution has come to the right temperature, the morphine sulphate crystallises suddenly into a solid mass." At the end of 48 hours the plug at the lower end of the tank is removed and the mother-liquor is allowed to run off, at first slowly, and afterwards, by elevating the apparatus, the last of the mother-liquor is removed. The drainage requires several days. The crystals in the apparatus are then cut up into pieces of the shape of bricks (commencing at the upper end), which are placed on an absorbent and removed to a drying room. When the bricks are dry, the colouring matter, which always comes to the surface, is removed by slicing off the dark portions. The bricks are then cut into slices, and the latter crumbled in the hands.—A. R. L.

*The Melting Point of Cocaine Hydrochloride.* O. Hesse. Liebig's Annalen, 1893, 276, 342—344.

IN view of the medicinal importance of cocaine hydrochloride, and the methods of testing its purity, the author quotes Kinzel's observation (Pharm. Zeit. 38, 25) that pure cocaine hydrochloride melts at 200°—202°, and that a lower melting point is indicative of impurities; this value has been confirmed by other observers.

Antrick, Einhorn, and Marquardt obtained, however, the value 181.5°, whilst the author, who conducted the experiment in a Roth's apparatus, found the value 186°. A repetition of the experiment has established the accuracy of the last value when the determination is carried out in the manner specified: whereas if the melting point be determined in a sulphuric acid bath a value near to 200° is obtained. The author has also observed that when the hydrochloride is raised in temperature very slowly in a sulphuric acid bath, a thermometer being used the mercury reservoir of which is not too small, the value 186° is obtained for the melting point; when, however, the temperature is rapidly raised, and a thermometer having a very small mercury reservoir used, the apparent temperature of fusion is 200°—202°.—A. R. L.

*The Detection of "Abrastol" (Calcium  $\beta$ -naphthyl Sulphate) in Wine.* Sanglé-Ferrière. Comptes rend. 1893, 117, 796.

See under XXIIL, page 177.

*The Estimation of Hydrocyanic Acid.* G. Gregor. Zeits. Anal. Chem. 1894 [1], 30—45.

See under XXIIL, page 176.

*The Estimation and Separation of the Cacao Alkaloids.* W. E. Kunze. Zeits. Anal. Chem. 1894 [1], 1—29.

See under XXIIL, page 178.

*The Combination of Hydrocarbons with Picric Acid and other Nitro-Compounds.* W. A. Tilden and M. O. Forster. Proc. Chem. Soc. 1893 [129], 239—240.

THE authors have instituted a compound described by Lextrait in 1886, which is formed by heating picric acid with pinene. It contains the elements of picric acid and a terpene, but differs from the picates of other hydrocarbons in forming a peculiar potassium salt, in yielding picramide

and borneol when submitted to the action of alcoholic ammonia, and in forming borneol and not simply a hydrocarbon under the influence of aqueous alkalis.

The authors have also instituted experiments on the behaviour of other hydrocarbons with nitro compounds generally, from which it appears that, in order that a compound may be formed, at least two nitro groups must be present in the nitro derivative concerned. They conclude that the oxygen of the nitro groups affords the link which, in ordinary cases, binds the hydrocarbon to the nitro compound, while in the case of the pinene derivative the oxygen of the hydroxyl is probably also involved.

*Contributions to our Knowledge of the Aconite Alkaloids. VII. On Picroaconitine.* W. R. Dunstan and E. F. Harrison. Proc. Chem. Soc. 1894 [132], 6.

"Picroaconitine" can no longer be retained as the name for an alkaloid derived from *A. Napellus*. The present investigation has shown that this plant contains, besides aconitine and the non-toxic isaconitine and aconine, a very small quantity of an amorphous alkaloid yielding crystalline salts, which has been named homisaconitine, and generally a considerable quantity of a base which neither crystallises nor furnishes crystalline salts.

*Contributions to our Knowledge of the Aconite Alkaloids. IX. The Action of Heat on Aconitine.* W. R. Dunstan and F. H. Carr. Proc. Chem. Soc. 1894 [132], 7.

THE authors find that when aconitine is heated at its melting point (188°—190°) it loses about 10 per cent. of acetic acid, which distils over, leaving a new alkaloid which they propose to name pyraconitine—



Pyraconitine is obtained in the form of an amorphous varnish, sparingly soluble in water, but readily in alcohol, chloroform, and ether. It has no effect on polarised light, and is not poisonous in small doses. The alkaloid readily dissolves in acids, forming salt which can be crystallised.

The solutions of the chloride, bromide, and iodide salts have a bitter taste, but are not toxic, at all events in small doses.

Pyraconitine and its salts readily undergo hydrolysis when heated with dilute acid or with water in a closed tube. Potash and soda quickly hydrolyse the alkaloid, even in the cold, but ammonia does so only very slowly. The sole products of hydrolysis are benzoic acid and an alkaloid which has been named pyraconine—



Pyraconine is an amorphous base resembling aconine in its properties, but differing from it in several respects. It is soluble both in water and ether. The aqueous solution has a somewhat sweet taste, and is levo-rotatory—

$$[\alpha]_D = -90.99^\circ.$$

It combines with acids to form crystalline salts which are very soluble in water.

The salts of aconitine also furnish pyraconitine, losing acetic acid when heated at about 190°. Isaconitine and aconine, however, do not undergo a similar decomposition. The authors consider it probable that the production of acetic acid from aconitine may serve as the basis of a process for the estimation of this alkaloid.

*Contributions to our Knowledge of the Aconite Alkaloids. X. Further Observations on the Conversion of Aconitine into Isaconitine.* W. R. Dunstan and F. H. Carr. Proc. Chem. Soc. 1894 [132], 8.

IN a former communication, the authors showed that when certain aconitine salts are heated at 100° in slightly acid solution they are very slowly changed into the salts of isaconitine. They have since found that this conversion



may be effected with great rapidity by heating a neutral aqueous solution in a closed tube at  $120^{\circ}$ – $130^{\circ}$  during from two to three hours, when the aconitine salt disappears, often so completely that the solution produces a tingling sensation on the tongue. The isaconitine is separated from the solution, which has been rendered alkaline with ammonia by repeated extraction with ether.

It has also been proved that the production of isaconitine invariably precedes the hydrolysis of aconitine into aconine and benzoic acid, not only when the hydrolysis is effected by acid, but also when water alone is used as the hydrolytic agent. In previous experiments, the formation of isaconitine during the hydrolysis of aconitine in presence of alkali could not be proved, owing to the rapidity with which aconine is produced. If, however, a considerable excess of an aqueous solution of soda be added to a solution of an aconitine salt, and the precipitated alkaloid be allowed to stand in contact with the cold alkaline solution until some of the alkaloid is dissolved, extraction with ether separates a notable quantity of isaconitine, as well as the unchanged aconitine. It therefore appears that the non-toxic aconine is really the product of the hydrolysis of the non-toxic isaconitine into which the aconitine first changes.

The observations recorded in the foregoing abstract, which prove that aconitine salts as well as the alkaloid lose acetic acid when they are heated, led the authors to look for the production of this acid when solutions of these salts are heated. It has been found that some acetic acid is formed when aconitine salts are heated with water in the manner above described, and also when these salts or the alkaloid are hydrolysed.

#### PATENT.

*A Manufacture of Pharmaceutical Preparations for Use more particularly in Cases of Anæmia and for Weak Throats.* W. Webster and T. Moore, Blackheath, Kent. Eng. Pat. 2884, February 9, 1893.

See under XI., page 161.

### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

#### PATENTS.

*Improved Sensitised Paper for Photographic Purposes.* A. Heseckel, Berlin. Eng. Pat. 20,042, October 24, 1893.

See under XIX., page 170.

*A New or Improved Method of Treating Canvas, Shirting, Silk, Paper, and like Substances, for the Purpose of Rendering the same Sensitive to Light.* G. J. Junk, Berlin. Eng. Pat. 21,249, November 8, 1893.

A mixture of bromide and iodide of silver is precipitated at a temperature of  $28^{\circ}$  C. in the presence of a trace of gelatin, and is maintained at that temperature for an hour or so with constant agitation, so as to prevent the precipitate from coagulating. It may then be washed in a centrifugal machine to remove the alkaline nitrate, and is finally well mixed with a cold solution of arrowroot which has been boiled in water till perfectly clear. The fabric or paper to be sensitised is then coated by means of a sponge in the dark room and dried, and if the emulsion has not been washed it is soaked in water for an hour and dried again. Development takes place as for an ordinary gelatino-bromide emulsion. Prints so made lend themselves far more readily to finishing in crayons, oils, or water-colours than when the fabric has been coated with an emulsion in gelatin, as there is no fear of the whole film stripping off the canvas. Paper prints made with the arrowroot emulsion may also be finished both with chalk and the brush, which was impossible before "owing to the impenetrable nature of the gelatin film." Starch may be used instead of arrowroot, but the results are said to be less favourable.—F. H. L.

### XXII.—EXPLOSIVES, MATCHES, Etc.

*On Explosives.* F. Biltz. Ber. 26, 1378–1383.

St. Frederick Abel, in his investigations (Comptes rend. 69, 105; 78, 1227, 1301, 1362, 1432), showed that a large number of explosives cannot be exploded by merely heating, but that it will only cause them to burn quietly, or partially explode, and that to produce a sharp explosion a detonator is required, i.e., another explosive. All explosives are not suited for this purpose, even if more powerful than actually required. To explain this relation between detonator and explosive, Abel assumes that there must be a certain synchronism of the explosive waves of the two in order to effect the explosion, and proved experimentally that the mechanical blow of the detonator was not of itself sufficient to cause the explosion of the other material. V. Meyer (Ann. d. Chem. 264, 127) showed that this explanation did not hold good for dynamite, observing that, under certain conditions, only a portion of a 5-grm. charge exploded, the remainder being scattered. This result does not agree with Abel's theory, which shows that the best material for detonating any explosive is itself, because the synchronism of the explosive waves of the detonator and charge is then the most exact.

Experiments carried out by the author with nitro-glycerin, gun-cotton, picric acid, and the German military powder, which is allied to gun-cotton, showed that they all behaved like dynamite, in that they were all bad transmitters of their own explosive effect. The experiments consisted in filling glass tubes (3 mm. bore and 120 mm. long, with one end closed) for about two-thirds their length with the explosive, which was pressed in, and then heating over a Bunsen flame. A violent explosion occurred, shattering the part of the tube heated, but the other part and a considerable portion of the explosive—with gun-cotton more than half—always remained undestroyed. In other experiments the heating was done by a platinum bridge-wire fused into the end of the tube and heated electrically. In these experiments the ends of the tubes were blown off almost without noise and only that portion of the charge surrounding the wire exploded. Picric acid did not explode, but first melted and then commenced to vaporise. When a little fulminate of mercury was placed in the tube and exploded by heating, the picric acid near it also exploded, but more than half was left unaffected. The author thinks that these properties account for explosives burning away quietly when unconfined.

Experiments in which the explosives were heated over a Bunsen flame on a platinum plate are then discussed, and a description of some experiments showing the action of explosives, suitable for lecture demonstrations, given. The author then refers to Max von Förster's experiments (Max von Förster, Comprimierte Schiessbaumwolle, 1886), in which a long lead cylinder with an axial hole was filled evenly with gun-cotton, the latter being fired by a fulminate detonator placed on top. The deformation of the cylinder was less and less from the top towards the other end, which showed that the detonator had a less effect on those portions of the gun-cotton remote from it than on those near it, and consequently the effect of the explosion of the further portions was less. The author believes that the difference between the above-named explosives and others is that the latter are good transmitters of the explosive effect. The duration of the exploding of black powder is much greater than that of quick explosives, and therefore the transmission from part to part takes place more certainly. With nitrogen chlorides, the diazo bodies, and many other violent explosives, the transmitting power is for some unknown reason much greater, and there is no difference between the burning and exploding of them, as there is with the other above-named explosives.—R. B. P.

#### PATENTS.

*Improvements in Non-poisonous Matches.* V. Simonet, Vienna, Austria. Eng. Pat. 17,071, September 11, 1893.

According to this invention the striking ends of the wooden splints are impregnated with a solution of potassium chlorate, "sodium hyposulphite" (thio-sulphate), and gum arabic in



water. Thus prepared, there are no "heads" liable to fall or break off, and the matches ignite easily by friction on a surface of amorphous phosphorus. A further part of this invention consists in imparting a greater degree of inflammability to the composition used for tipping safety matches and those which can be ignited by friction on any rough surface, by adding to the composition a certain quantity of powdered zinc previously triturated with amorphous phosphorus.—W. M.

*Improvements in Methods of Securing the Chemical Stability of Nitro-Compounds.* R. S. Schüpphaus, Brooklyn, U.S.A. Eng. Pat. 22,384, November 22, 1893.

THE object of this invention is to secure the chemical stability of nitro-compounds by the addition of a suitable quantity of urea after the nitro-compounds have been freed from acid as far as possible by washing. The urea is added in the form of a solution in methyl or ethyl alcohol.—W. M.

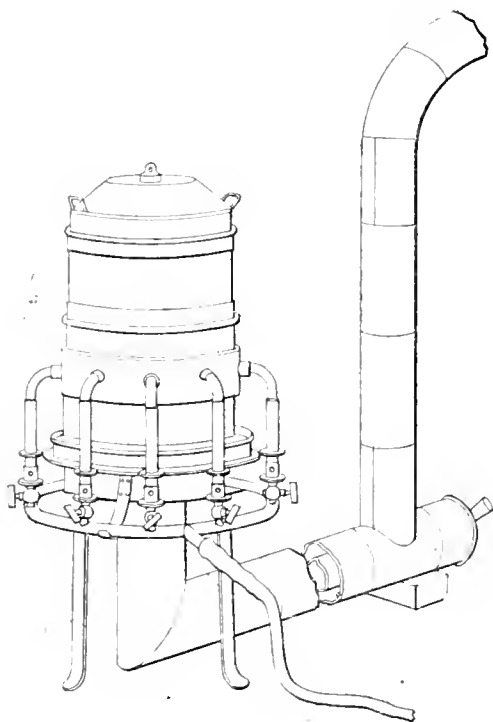
## XXIII.—ANALYTICAL CHEMISTRY.

### APPARATUS, ETC.

*A Recuperative Gas Furnace.* C. Bigot. Monit. Scient. (4) 7, 519—520.

THE gas enters the furnace laterally through a series of Bunsen burners B, each fitted with an independent ferrule. The flame passes between the casing C and the movable piece F, through which and the central tube G it descends. G is fitted with a chimney to produce a strong draught, and is surrounded by a second larger tube H, having at its outer end a controllable air inlet. The air passing along H is heated by contact with G and passes up into the furnace at the point where the flames enter, producing an intense and complete combustion. The joints of the various pieces making up the body of the furnace are made with sand lutes. The consumption of gas is small, and one can work

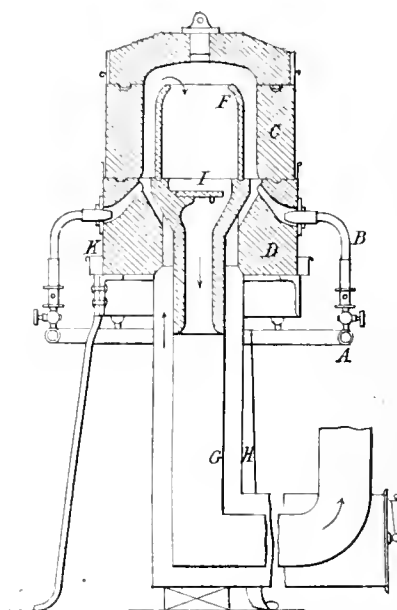
Fig. 1.



with either a reducing or an oxidising flame, as the air admitted to the furnace is under complete control. The furnace lends itself particularly to ceramic work, the fusion of metals, glass, and enamels, and to the study of limes, cements, &c. The furnace will give temperatures that have never before been attained in laboratory apparatus, save by the use of air under pressure.

The author is particularly engaged on the study of the behaviour of natural clays which, when heated to the temperature at which porcelain is burnt, give the product known as "stoneware." He has been able to prove that they contain a notable quantity of mica in the form of very

Fig. 2.

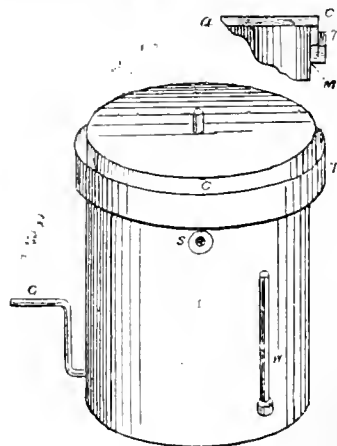


thin and small flakes intimately mixed with the clay. It appears from his experiments that this mica plays the rôle of a scouring body and permits the clay to undergo that incipient vitrification which characterises "stonewares."

—R. B. P.

*A Drying Oven for Drying in Hydrogen at the Temperature of Boiling Water.* F. W. Morse. Amer. Chem. Soc. 15, 709—710.

THE oven consists of a cylindrical copper box with double bottom and wall; the inside of the oven is seven inches in



diameter and nine inches in depth, space between walls one inch. The hydrogen enters by a brass tube G and passes through a coiled tube between bottom and wall of the oven; this tube then enters the inner oven near the top. The gas is thus heated before entering the drying chamber; W is a water-gauge and S the steam outlet. Drying is accelerated by placing a dish of sulphuric acid on the bottom of the oven, and upon this dish rests a rack to receive the watch glasses or drying dishes. It is well also to have the interior of the oven blackened to increase radiation, and the outer cover plated with nickel. Repeated trials showed differences of not more than 0.2° C. between the temperature of the water and that of the drying chamber. The hydrogen gas passes out of the oven by an outlet not shown. The cover C fits loosely in the copper trough T, and the mercury M makes a perfectly tight joint.—V. C.

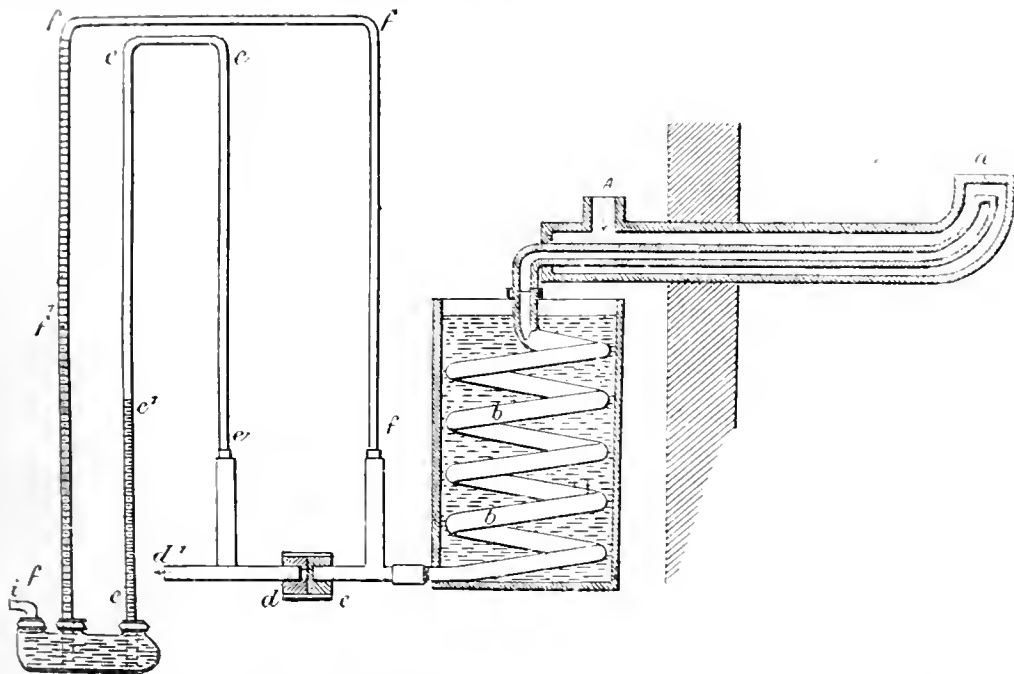
## PATENT.

*Improvements in Pyrometers.* E. A. Ueling and A. Steinbart, Birmingham, U.S.A. Eng. Pat. 15,484, August 15, 1893.

If air or gas at constant pressure be made to pass successively through two small apertures, then the pressure

between them will remain constant, provided that the temperature of the fluid passing through each of the diaphragms remains constant. If, therefore, the gas passing through the first aperture vary in temperature, whilst at the other it is kept at a certain normal temperature, then the variation of pressure between the apertures will be a measure of the temperature of the gas as it entered.

The patentees have made use of this principle in constructing their pyrometer, which is represented in the accompanying figure. The air or gas enters the pyrometer at *A* and becomes heated before passing through the first diaphragm *a*. It is then cooled to a constant temperature in passing through the worm *b b*, surrounded by ice or boiling water, and finally passes out into a constant partial vacuum at *d*<sup>1</sup> through the second diaphragm, secured between the couplings *c* and *d*. The constancy of the partial vacuum is ascertained by means of the manometer *e e*<sup>1</sup>, *i* being open to the atmosphere. The variation in pressure of the air between the apertures or diaphragms is registered by the manometer *f f*<sup>1</sup>, which is preferably graduated to read off directly the temperature of the diaphragm *a*, *i.e.*, the temperature it is desired to measure. If the pyrometer is operated by suction, some form of vacuum gauge, such as that represented in the figure, is



all that is necessary, but if it be operated by pressure, then some form of pressure gauge must be substituted for the vacuum gauge.—J. S.

reagents which decompose polysulphides (acids, alkalis, oxygen) destroy the coloration. The reaction is stated to be very delicate.—H. T. P.

*INORGANIC CHEMISTRY.—QUALITATIVE.*

*Test for Free Sulphur.* J. C. Gil. Zeits. anal. Chem.  
1894, 34--55.

A small quantity of an alkaline polysulphide, added to boiling absolute alcohol, imparts to the latter a coloration ranging from pale sky-blue to a deep greenish-blue. This reaction may be utilised for the detection of free sulphur. It is only necessary to boil the substance under examination with strong alcohol containing in solution a little potassium or sodium hydrate. In presence of sulphur the characteristic blue or green colour will be developed. The coloration disappears on cooling, but, in absence of air, is restored on again applying heat. Generally speaking, all

*INORGANIC CHEMISTRY.—*  
*QUANTITATIVE.*

*The Estimation of Phosphorus in Steel containing Silicon, and in Pig-iron.* J. Spüller and S. Kalman. *Zeits. anal. Chem.* **32**, 1893, 538-550.

THE authors find that the phosphorus in steel or iron containing silicon, may be accurately determined by the following modification of the phosphomolybdate method. The sample of iron is dissolved in nitric acid, large excess of the latter being avoided; the nitrous fumes are driven off by heat, a little dilute potassium permanganate solution added, and the solution boiled until the red tint disappears. The precipitated manganese dioxide is then reduced by

10 per cent. potassium nitrite solution, the liquid boiled and subsequently precipitated at 55–60° with molybdate solution. Under these conditions no silicon separates as silicomolybdate, as is the case if ammonium salts be present in any quantity. The phosphomolybdate is filtered through a filter which is partially filled with paper fibres, obtained by treating good filter paper with hydrochloric acid (sp. gr. 1.16), pouring the mass into water, and washing till free from acid. The authors have employed this paper sludge for many years in the filtration of barium sulphate, to prevent the precipitate from going through the filter. Phosphorus determinations made by the above method, in which the ammonium phosphomolybdate was weighed as such in the usual way, compare very favourably with those made by the ordinary magnesium pyrophosphate method.

—W. J. P.

*The Determination of Phosphoric Acid by the Titration of the Phosphomolybdate Precipitate with Standard Alkali.*  
H. Pemberton, jun. Jour. Franklin Inst. 1893, 136, 362–376.

THE solutions required in this method are to be prepared as follows:—

**Ammonium Molybdate.**—Ninety grms. are dissolved in somewhat less than a litre of water, and the clear liquor is decanted; the residue of molybdic acid is dissolved in a little ammonia and added to the bulk of the liquid. If the ammonium molybdate contains traces of  $P_2O_5$ , a few decigrams of magnesium sulphate, and ammonia to faint alkalinity, are added. The solution is now made up to one litre.

**Ammonium Nitrate.**—A saturated aqueous solution.

**Nitric Acid.**—Sp. gr. 1.4.

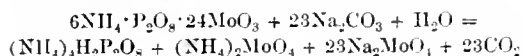
**Potash Solution.**—This is of such strength that 100 cc. of it will neutralise 32.65 cc. of normal acid. It must be freed from carbonate by barium hydrate, and its strength is best determined by a direct test upon a phosphate solution of known strength, conducted as described below.

**Standard Acid.**—This must be equivalent in strength to the potash solution.

**Phenolphthalein Solution.**—One gram. of phenolphthalein is dissolved in 100 c.c. of 60 per cent. alcohol, and at least 0.5 cc. of this solution is used for each titration.

The analysis is conducted as follows:—One gram. of a phosphate, or 2–3 grms. of a fertiliser, are dissolved in nitric acid, an excess of which can be used with impunity, and the solution made up to 250 cc. The solution need not be evaporated to dryness, nor filtered; the insoluble matter in a phosphate rock rarely exceeds 10 per cent. and its volume is only about 0.05 cc. Twenty-five cc. of the solution are measured into a 4-oz. beaker and neutralised with ammonia—until a precipitate just begins to form—and then treated with 5 cc. of the nitric acid. Ten cc. of the ammonium nitrate solution are added and the whole diluted to 50–75 cc. The solution is now heated until it boils, and 5 cc. of the ammonium molybdate solution added. This is followed by a second and third 5 cc. if necessary. The precipitate is allowed to settle, filtered at once, and washed thoroughly with water by decantation and on the filter. The filter and precipitate are transferred to the beaker, the standard alkali is run in, phenolphthalein added, and the excess of alkali titrated with the standard acid. Each cc. of the alkali is equivalent to one mgrm. of phosphorus pentoxide.

The author shows that the above ratio of alkali to  $P_2O_5$  is correct by quoting experiments which prove that 23 molecular weights of  $Na_2CO_3$  are necessary to neutralise one molecular weight of the yellow phosphomolybdate—



—A. G. B.

*Influence of Free Nitric Acid and of Aqua Regia on the Precipitation of Barium as Sulphate.* P. E. Browning. J. Anal. and Appl. Chem. 1893, 7, 325–330.

THE author's results show that the presence of an excess of nitric acid or aqua regia amounting to 10 per cent. by volume of the liquid treated, is actually beneficial in the estimation of barium as sulphate. Usually the advantage thus gained is observed in the tendency of the precipitate to fall in a coarsely crystalline state, a condition favouring ready and efficient filtration. In certain special cases in which substances such as ammonium nitrate, ammonium citrate, sodium citrate, and metaphosphoric acid, which otherwise would exert solvent action, are present, the precipitation is rendered more complete by adopting the beneficial degree of acidity above suggested. The contaminating effect of such substances when complete precipitation has been brought about, and which thus leads to excessive results, may be corrected by dissolving the precipitate first obtained in concentrated sulphuric acid, and reprecipitating by rapid evaporation, when the precipitate resulting will have assumed the coarsely crystalline condition desired, and will also be freed from the traces of contaminating substances referred to.—A. R. L.

*Quantitative Precipitation in Presence of Hydroxylamine.* P. Janonach and J. Mai. Ber. 1893, 26, 1786–1787.

THE authors find that the presence of hydroxylamine in the solution of a salt exerts an influence on the precipitation of the metallic base by ammonia, thus:—In some cases, precipitates are produced which do not result by the use of ammonia alone; in others, precipitation is completely arrested, or the precipitate itself considerably modified. The following example is cited of the precipitation of chromic hydroxide in presence of hydroxylamine:—Potassium bichromate (about 1 gram.) is dissolved in water (50 cc.) and converted into chromic salt by boiling with alcohol and hydrochloric acid; after evaporating off the alcohol, the liquid is diluted to 300 cc. and hydroxylamine hydrochloride (2 grms.) and an excess of ammonia are added to it while boiling. Precipitation is in this way complete, the excess of ammonia exerting no solvent action on the violet-red precipitate, which can be filtered and washed (without the aid of a suction pump) with much greater ease than can the ordinary chromic hydroxide precipitate. The filtrate is completely colourless, and in it the potassium may be accurately estimated.—A. R. L.

*The Estimation of Hydrocyanic Acid.* G. Gregor. Zeits. Anal. Chem. 1894, 30–45.

THE author has investigated some of the adopted methods for determining hydrocyanic acid in certain medicinal liquids, as, for instance, essence of bitter almonds, laurel-water, &c., and in conclusion recommends the following *modus operandi*.

**Gravimetric Method.**—Fifty cc. of the sample to be examined, contained in a stoppered bottle, are shaken with 5 cc. of ammonia; an excess of silver nitrate (25–30 cc. of decinormal solution) is then added, and the liquid, after shaking, slightly acidified with nitric acid. It is essential that the reagents be added in the order given, and that all the operations be conducted as quickly as possible. The precipitate of silver cyanide is finally collected and weighed in the usual manner.

**Volumetric Method.**—Similar to the above. The liquid, with precipitate, is made up to a definite volume, filtered, and the excess of silver determined in an aliquot part of the filtrate by titration with decinormal potassium thiocyanate solution, using ferrie sulphate as an indicator. The results are stated to be very accurate, and as the presence of alcohol does not influence them, the method may be directly applied to alcoholic preparations containing hydrocyanic acid. Of course, chlorine must be absent, but this is usually the case, since the liquids in question are generally prepared by distillation.—H. T. P.

## ORGANIC CHEMISTRY.—QUALITATIVE.

*A Colour Reaction of Acid Anilides.* J. Tafel. Ber. 25, 112.

THE acid anilides, like the corresponding phenylhydrazones and osazones, yield colour reactions with potassium bichromate or lead peroxide. The test is applied by dissolving a few mgrms. of the anilide in strong sulphuric acid and adding a little powdered potassium bichromate. A red to violet coloration will be developed. The reaction is common to all the simple anilides and phenylcarbamides (with one or two exceptions).—H. T. P.

*The Detection and Estimation of Formaldehyde.* A. Trillat. Bull. Soc. Chim. [5], 9, 305.

See below under Organic, quantitative.

*Detection of "Abrastol" (Calcium  $\beta$ -naphthyl Sulphate) in Wine.* Sanglé-Ferrière. Comptes rend. 1893, 117, 796—797.

THE chemical and therapeutical properties of "abrastol" have been described by Dujardin-Baumetz and Staehler (Bull. gén. de Thérap. Juli 1893). This antiseptic is calcium  $\beta$ -naphthyl sulphate,  $(C_{10}H_7OSO_2)_2Ca$ . It has the advantage over  $\beta$ -naphthol in that it is soluble in an equal quantity of water. It gives, even in a solution containing 0.2 grm. per litre, a beautiful blue coloration with ferric chloride, but this reaction cannot be utilised for its detection in wine. For the latter purpose the author proceeds as follows:—Two hundred cc. of the wine is boiled for an hour in a reflux apparatus with 8 cc. of hydrochloric acid when the abrastol is hydrolysed to  $\beta$ -naphthol, which latter may then be extracted with benzene, and the residue left on distilling the benzene solution taken up with chloroform. A fragment of caustic potash is then dropped into the chloroform solution and it is boiled for two minutes, when a blue coloration is produced, changing to green, and finally becoming yellow. With traces of  $\beta$ -naphthol the solution is slightly greenish and the fragment of potash only is coloured blue. By means of this reaction 0.0625 grm. of  $\beta$ -naphthol (corresponding to 0.1 grm. of abrastol) per litre can be detected. The presence of abrastol in no way vitiates the determination of potassium sulphate by the ordinary method, as the compound is only hydrolysed after prolonged boiling.—A. R. L.

## ORGANIC CHEMISTRY.—QUANTITATIVE.

*The Detection and Estimation of Formaldehyde.* A. Trillat. Bull. Soc. Chim. [5] 9, 305.

THE liquid to be examined for formaldehyde is mixed with  $\frac{1}{2}$  cc. of dimethylaniline and a few drops of sulphuric acid, heated for half an hour on the water-bath, then made alkaline and boiled until the odour of dimethylaniline has disappeared. The liquid is filtered, the paper moderately washed, spread out in a porcelain dish, and, after moistening with acetic acid, sprinkled over with powdered peroxide of lead. The presence of formaldehyde is indicated by the development of a blue coloration (due to the formation of tetramethyldiamidodiphenyl carbinol). Another method is based on the fact that formaldehyde yields with aniline a precipitate of anhydroformaldehydaniline  $(C_6H_5N:CH_2)$ . It is only necessary to mix dilute solutions of the two bodies; after some hours a whitish precipitate or turbidity appears. The reaction is very delicate, and allows of the detection of formaldehyde in solutions containing only 1 in 20,000. In this case, however, the turbidity only develops after several days. The reaction is also characteristic for acetaldehyde. For quantitative purposes, 1—1 cc. of the formaldehyde solution to be tested, are added drop by drop to an excess of an aqueous solution of aniline (3:1,000). After 48 hours the precipitate is collected on a tared filter-paper, dried at 40° C., and weighed.—H. T. P.

*The Alkali Test for Kerosene, Naphtha Products, Hydrocarbon Lubricants, &c.* K. Lissenko and A. Stepanow. Dingler's Polyt. J. 290, 139—143.

ABOUT ten years ago endeavours were made in the Russian petroleum districts to economise in the quantity of caustic soda used in the refining of petroleum. Kerosenes were obtained which satisfied the usual tests, but in practice were found unsuitable for lamp oils, as they did not rise freely in the wicks. Investigation proved that this was due to the presence in the kerosene of small quantities of saponaceous salts, and analysis sometimes showed as much as 0.01 grm. ash in 1,000 grms. of kerosene, this ash consisting of  $CaO$ ,  $MgO$ ,  $Fe_2O_3$ ,  $Na_2O$ , and  $SO_3$ . Further investigation proved that for a lamp oil the ash should certainly not exceed 0.01 grm. per 1,000 grms. oil, and that in the best oils, it generally varied between 0.004 and 0.007 grm. The presence of these soaps or salts is equally detrimental in lubricating oils.

As the ash determination is troublesome, and also somewhat uncertain on account of possible loss of sodium salts by volatilisation, the so-called "alkali test" was introduced. Though used in various slight modifications, this consists mainly in mixing in a flask 300 or 500 cc. kerosene with 6 or 10 cc. of a 2 per cent. solution of caustic soda, warming to 60—70°, and shaking the whole violently for five minutes. The mixture is then transferred to a separating funnel, the alkaline extract filtered through a double filter, and acidulated with HCl. The acids in the naphtha and in the saponaceous salts which had been extracted by the alkali are thus precipitated, and the degree of turbidity produced thereby indicates the amount of impurity present in the oil. The turbidity—originally simply roughly stated as satisfactory or the reverse—is now estimated by the size of print which can be read through the opalescent liquid contained in a glass cylinder of given diameter, the operator being required to specify the diameter of the cylinder, and the size of print he can just discern. The kerosenes are now divided into classes according to the results given by this "alkali test." A definitely fixed scale is being constructed, and details will be published next year (1894). Even the best kerosenes give a slight opalescence. The theory of the test is clear, viz., that the soaps and acids present in the oil are dissolved in the dilute alkali, and on acidifying the alkaline extract the acids which are insoluble in water render the extract opalescent. In testing lubricating oils, which generally contain a considerably larger quantity of ash (sometimes as much as 0.125 grm. per 1,000 grms. oil), the test is somewhat modified. In this case 5 cc. of a 1.5 per cent. caustic soda solution is mixed with 10 cc. of the oil, the whole heated to 80°, shaken violently for some time, allowed to stand at 70° for 2 to 3 hours to settle, and then examined. If the aqueous layer is then milky, and there is a more or less considerable precipitate between the aqueous and oil layers, the oil is undoubtedly insufficiently purified. In most cases the aqueous solution is clear, but a more or less marked yellowish red film is seen at the point of contact of the two layers. In such cases, if the ash is small, the oil is a satisfactory lubricating oil. It only very rarely occurs that the oil is so absolutely free from salts and acids as to give no film. Another form of this test sometimes used is to shake the oil to be tested with a small quantity of strong caustic soda solution. If the kerosene is pure the two liquids rapidly separate again with a perfectly bright contact surface. If the kerosene is impure a whitish film forms at the point of contact of the two liquids. This form of test, however, only decides between pure and impure oil, and gives no indication of the degree of impurity.

The view held in Baku and elsewhere of this alkali test was that the alkali dissolved out the soaps (possibly in the form of basic salts), and that the test was a definite proof of the presence or absence of these soaps in the oil. The authors have carefully investigated this matter, and their conclusions (confirmatory in many respects of the work of Doroschenko and of Engler) are as follows. These saponaceous salts are almost absolutely insoluble in pure neutral petroleum, but dissolve readily in the same if it contains a

trace of naphtha acids. Almost all petroleum contains more or less of these acids, which are generally mixtures of hydro-aromatic and sulphonic acids and phenolic compounds. Kerosene, when distilled in the air, or even when shaken at 70–80 with air, generally undergoes slight oxidation with formation of traces of these acid compounds, so that absolutely neutral kerosene is rarely met with. The salts of these naphtha acids undergo partial hydrolysis in the presence of water, and though excess of alkali in some degree prevents this hydrolysis, the latter is not completely stopped till the excess of caustic soda amounts to at least 10 per cent. of the water. Thus, in the purification of petroleum by weak alkali, as now often adopted, and subsequent washing with water, part of the salts first formed becomes again hydrolysed, the acid so liberated is taken up again by the petroleum, and the acid petroleum in its turn re-dissolves some of the salts. The authors therefore believe that for good purification, final treatment with strong caustic alkali should be adopted. The presence of small quantities of these naphtha acids in the oil does not seem to be detrimental as long as no salts are present. From this point of view the alkali test, as at present carried out, is unsatisfactory, as it really only determines the presence or absence of these naphtha acids without deciding whether they are present in the free state alone or also in the form of the detrimental salts. As the ash determination is difficult and not altogether satisfactory, testing for these salts is at present unsatisfactory. The authors suggest as a test which, as far as preliminary trials go, seems to be satisfactory, the shaking of the petroleum to be tested with water containing a few drops of phenolphthalein. If salts are present, hydrolysis takes place and the liberated alkali, being taken up by the water (whilst the acid remains in the oil), colours the solution violet. The aqueous extract may even be titrated. Further investigation of this test is, however, required.—L. T. T.

*Characteristics of Glucose Syrups.* W. E. Stone and C. Dickson. *J. Anal. and Appl. Chem.* 1893, **7**, 317–321.

*Specific Gravity.*—A given weight of the syrup is dissolved in water and the volume made up to 100 cc. The specific gravity of this solution is then ascertained by a Westphal hydrostatic balance. The specific gravity of the syrup may be then calculated with approximate accuracy by the formula  $D = \frac{W}{100 - (w - W)}$  in which W is the weight of syrup, and w the weight of 100 cc. of the solution.

*Water.*—The method adopted consists in mixing a weighed portion of the syrup with weak alcohol and a known weight of clean sand, heating over a water-bath with occasional stirring until dry, adding a quantity (5 cc.) of strong alcohol, evaporating over a water-bath, and finally drying in an air-bath at 100°.

*Ash.*—A beaker containing a sufficient quantity of the syrup and a small pipette are accurately weighed; the pipette is then filled with syrup, and the upper end closed with rubber tubing and a screw pinch-cock. The pipette thus charged is suspended over a weighed platinum dish, which is heated to redness, and the syrup allowed to drop from the pipette into it at such a rate that each drop is carbonised and partially incinerated before its successor falls. When sufficient syrup has been run in, the pipette is returned to the beaker and the whole reweighed. The partially burned mass is extracted with water, the residue incinerated at a high temperature, and the watery extract finally added to it; the whole is then evaporated to dryness and weighed as crude ash.

*Relative Reducing Power.*—The cupric reducing power was found to be considerably augmented after inverting the sample. This was effected by dissolving 5 grms. in 200 cc. of water, adding 10 cc. of concentrated hydrochloric acid, and heating the mixture for half an hour at 60°–70°, neutralising the acid, and diluting to 250 cc. The minimum increase in the reduction after inversion was 0.97, and the maximum 22.76; the syrup which exhibited the larger difference commanded the highest price.

*Specific Rotary Power.*—The determination of this constant in the original syrup, after inverting it, and after fermenting it, furnishes an interesting means of comparison between samples.—A. R. L.

*The Estimation and Separation of the Cacao-Alkaloids.* W. E. Kunze. *Zeits. Anal. Chem.* 1894 [1], 1–29.

In the earlier portion of his paper the author describes and criticises the principal methods employed for the above purpose, and concludes that they are all more or less untrustworthy. In most cases the presence of caffeine appears to be either entirely neglected, the mixture of alkaloids finally obtained being regarded as pure theobromine; or an imperfect means is adopted of separating them (solution of the caffeine in benzene, &c.). Details are then given of experiments made to determine the best mode of operating. The removal of the fat (from cocoa, &c.) by petroleum ether, &c., prior to the extraction of the alkaloids, is not recommended, as the caffeine is partially or wholly dissolved with the fat. Methods in which an aqueous extract of the raw material is evaporated with magnesia, lime, &c., the residue being treated with chloroform, are unsatisfactory, because the mass obstinately retains a large portion of the alkaloids. A similar objection applies when the aqueous extract (after clarification with animal charcoal or compounds of iron and aluminium) is shaken with a solvent. Much of the alkaloid appears to be absorbed or precipitated by the clarifying agent.

As the result of his experience the author has worked out the following method, which he claims to be more accurate and expeditious than any process hitherto published.

1. For the estimation of total alkaloids, the material to be examined is boiled with dilute sulphuric acid (5 per cent.), filtered, and the alkaloids precipitated from the filtrate by a large excess of a nitric acid solution of sodium phosphomolybdate. The precipitate, which settles well, is filtered off after 24 hours, washed with dilute sulphuric acid, and at once decomposed by treatment with baryta water, the excess of barium being removed by carbon dioxide. The liquid (and precipitate) is then evaporated to dryness, and the residue extracted by boiling chloroform. The chloroformic solution on evaporation leaves the alkaloids almost perfectly pure, and containing only a trace of ash.

2. Separation of the alkaloids may be effected with great accuracy by converting the theobromine into the insoluble silver salt. For this purpose the mixture of alkaloids is dissolved in ammonia, a considerable excess of silver nitrate added, and the solution boiled down to a very small bulk, and until all free ammonia is expelled. The crystalline precipitate ( $C_8H_7AgN_3O_2$ ) is collected, washed with boiling water, ignited, and the metallic silver weighed. The process may be made volumetric by titrating the excess of silver in the filtrate by Volhard's method. In the latter case the alkaloids may be readily isolated from the precipitate and filtrate (after titration) and tested as to their purity, identity, &c.—H. T. P.

*The Separation of Volatile Fatty Acids.* M. Wechsler. *Monatsh. f. Chem.* **14**, 462–469.

THE method of partial neutralisation and subsequent distillation was employed by Liebig (*Annalen*, **71**, 355) for isolating butyric acid from a mixture of butyric and isovaleric acids, and further for separating acetic acid when mixed with either of the above acids. In the former case he states that butyric acid distils off, leaving isovaleric acid behind as a salt; whilst in the latter case the acetic acid remains as a salt, and the butyric or isovaleric acid distils over. Vieil (*Annalen*, **148**, 163) finds the opposite result in the case of a mixture of butyric and isovaleric acids, and observes that when the mixture is partially saturated, isovaleric acid distils off, leaving butyric acid behind. Fitz (*Ber.* **11**, 42) and Hecht (*Annalen*, **209**, 319) both state that when a mixture of acetic, butyric, and caproic acids is distilled with steam, the acids of highest

A GENERAL COMPARATIVE TABLE FOR THE ANALYSIS OF FATTY MATTER.

No.	Fatty Substance.	1. Density at 15° C.	2. Action of Nitrous Vapours.	3. Sulphuric Acid. Rise of Temperature.		4. Indices.		5. Freezing Points of Oils, of Fatty Acids.	6. Fusing Points
				Absolute.	Relative.	Bromine.	Iodine.		
1	Olive .....	0.915 to 0.917	Solidification, Elaidin melts at 32°	35	94	500 to 544	80 to 84	+ 1 to 4	27
2	Peanut .....	Unshelled, 0.917-5 Shelled, 0.921	Liquid mass	46	127	539	7	- 1	31
3	Sesame .....	0.922	"	54	159	605	101	- 5	26
4	Cotton-seed (edible) .....	0.925	"	52	144	645	108	- 12	36 to 37.5
5	Poppy (drying) .....	0.914-5	"	89	222	835	3	- 18	20.5
6	Colza .....	0.915	"	48	133	610	99	- 6.5	17
7	Rape seed .....	0.922-5	"	56	155	632	103	- 4	13.5
8	Linseed .....	0.926	"	121	326	1,000	56	- 27.5	23
9	Walnut .....	0.926	"	49	275	737	144	- 39	Fluid
10	Cameline .....	0.921	"	51	141	817	132	- 18	"
11	Hecchnut .....	0.918-5	"	59	163	852	106	- 18	24
12	Mustard seed .....	0.917	"	39	108	763	96	- 5 to 9	16
13	Sweet almond .....	0.915	Soft consistence	47	130	644	98	- 10	14
14	Hazelnut .....	0.915	Rather firm	31	86	563	87.5	- 21	25
15	Caster .....	0.915	Formation of ricin-chalidin	10	111	539	84	- 5	12
16	Coprah .....	0.924-5	Solidification	47.5	98.5	74	12	Solid, 22.5 Fusible, 26 24.5 to 26.5	26.5
17	Cablage palm .....	0.915	"	19	52.7	49	16	25 to 35	37 to 39
18	Palm .....	0.915	"	17	47	315	51	23 to 27	46
19	Mowrah .....	0.920	"	26	71	403	72.3	11-28.3	46
20	Marcassin from cotton-seed oil .....	0.917-5	Soft consistence	47.5	136	588	84	- 28 to 39	56
21	Hipp .....	0.917	Solidification	13.5	37	152	24.7	23 to 25.5	46.5
22	Karite .....	0.917-7	"	28.5	79	316	67.2	19 to 31	37 to 39
23	Butter .....	0.920 to 0.926	"	Mean, 23	Mean, 43	100 to 216	26 to 35	23 to 25	10 to 20
24	Suet .....	Mutton, 0.917 Beef, 0.915	"	16	14	247	40	33 to 35	40 to 45
25	Lard .....	0.923 to 0.930	"	33	91	565	59	32 to 34	45
26	Fish oil .....	0.923 to 0.930	Liquid mass	50 to 100	138 to 277	763 to 872	123 to 141	Concreals at 0	"
27	Resin .....	1.08	"	"	"	718	116	"	"
28	Resin oil .....	0.90 to 0.909	Fluid mass	"	"	267	48	"	"
29	Commercial olein (olive acid) .....	0.90	Solidification, Elaidic acid	24	"	437 to 518	68.5 to 89.5	"	4 to 12
30	Miscellaneous:								
	Mineral oils:								
	Petroleum .....	0.850 to 0.890	Liquid	5	8	80	13	"	"
	Shale .....	"	"	22	61	130	21	"	"

A GENERAL COMPARATIVE TABLE FOR THE ANALYSIS OF FATTY MATTER—*cont.*

No.	Fatty Substance.	7. Solidification of Fatty Acids.	8. Saturation by No. of Number of Cubic Centim. of Liquid absorbed by 5 Grams. of Fatty Acid.	9. Solubility in Alco- hol, Ether, and Neutral Oil, dissolved by 1000 Grams. Alcohol.	10. Deviation of the Plane of Polarisation.	Characteristic Properties.
1	Oil of Olive .....	23	17.86	43	0° G. sucrose.	<i>Action of Various Reagents.</i> —Oxidized by Guibet's process, turning on 20 grams, oil, 6 grains, H <sub>2</sub> SO <sub>4</sub> and 1 gram, HNO <sub>3</sub> . The characteristic colorations are: first, with H <sub>2</sub> SO <sub>4</sub> , green; second, with H <sub>2</sub> SO <sub>4</sub> , 1 HNO <sub>3</sub> , grey-green; third, after boiling, straw-yellow; fourth, after cooling, a hard mass, color of white butter. Anhydrous acid, CaH <sub>2</sub> O <sub>4</sub> (fusing point 75°), is characteristic and allows the detection of pen- <i>Analysis of Oil.</i> —The oil is contained in all oils containing it. The red color obtained by treating the rectified fatty acid of the oil with hydrochloric acid and sugar is characteristic. The fatty acid is first collected and melted in an oven. <i>Mellin's Process.</i> —The black color obtained by the reduction of AgNO <sub>3</sub> in the presence of the saponified products of the rectified oil is characteristic. Poppy oil, when used as an adulterant of olive oil, prevents the solidification of the mass by the action of various vapours. A solution of potassium hydroxide heated with unrefined colza oil blackens with lead acetate and reddens with potassium nitroferrocyanide. Density and fusing points of the fatty acids. Density, freezing point, sulphuric saponification, and iodine index. Sulphuric saponification, iodine index, freezing points, and fluidity of the fatty acid. Iodine index, freezing point, and fluidity of the fatty acid. <i>Boile's Reaction.</i> —Two cubic centimetres, nitric acid, 0.1 gram, dried albumin, 10 cubic centimetres; when agitated the mixture takes a characteristic vermilion tint. Sulphuric saponification and iodine index. Nitrous vapours and solidification of fatty acid. Sulphuric saponification and freezing points. Density, solubility in alcohol and crystallisable acetic acid, insolubility in petroleum ether. <i>Mellin's Process.</i> —The neutralised oil is soluble at 31° in twice its volume of absolute alcohol, and becomes insoluble on the addition of seed oil. Saturation, iodine index. <i>Mellin's Process.</i> —Like the preceding, but requires four volumes of absolute alcohol. Density, sulphuric saponification, partial solubility in absolute alcohol. Density, saturation, when shaken with alcohol gives a characteristic milky tint. Iodine index, solidification of the oil: Mellin's process (4). Sulphuric saponification, iodine index, fusing point of the fatty acids. Fusing point of the fatty acids, saturation, when agitated with alcohol gives a characteristic milky tint. Volatile fatty acids, iodine index, solidification of the neutral matter, saturation, microscopic examination. Sulphuric saponification, solidification of the fatty acids, insolubility in absolute alcohol. Sulphuric saponification, iodine index. Iodine index, solubility in alcohol (milky emulsion), fusion point of cholesterol. Density, saturation, solubility in alcohol, deviation of the plane of polarisation. Density, deviation of the plane of polarisation, insolubility in crystallisable acetic acid. Density, nitrous vapours, solubility in alcohol. Density, iodine index, insolubility in alcohol.
2	Peanut .....	28	17.82	66	Variable	
3	Sesame .....	22	17.70	41	"	
4	Cotton-seed (edible) .....	35	18.17	62	"	
5	Poppy (drying) .....	16	18.13	45	- 0.7°	
6	Colza .....	16	16.49	29	Variable	
7	Rape seed .....	15	16.68	15	+ 10	
8	Linseed .....	21	17.98	70	Inactive	
9	Walnut .....	Fluid	18.26	44	"	
10	Camelline .....	"	17.90	78	- 3	
11	Bremond .....	17	18.18	45	Almost inactive	
12	Mustard seed .....	15	17.97	28	+ 3	
13	Sweet almond .....	5	18.22	39	Almost inactive	
14	Hazel nut .....	23	17.63	33	"	
15	Castor .....	4	16.77	Soluble	Mean = 63	
16	Coprah .....	22.7	21.1	Soluble at 31° C. in 2 vol. alcohol.		
17	Cabbage palm .....	24	22.5	Soluble at 31° C. in 4 vol. alcohol.		
18	Palm .....	12 to 16	18.5	60		
19	Morrah .....	12.5	15.5	160		
20	Macassar fern root- tissue oil .....	12.5	18.2	61		
21	Hyperic .....	52.5	17.7	260	Un- determined.	
22	Karite .....	52.5	14.9	80		
23	Butter .....	35 to 37	21.15	65.4		
24	Suet .....	45 to 47	18.5			
25	Lard .....	43 to 45	18.25	3.18		
26	Fish oil .....	34	18.12	43		
27	Rosin .....	"	17.71	Cod liver, 240		
28	Rosin oil .....	"	15.13	Soluble	Mean = - 15° + 30°	
29	Commercial olein (oleic acid) .....	0 to + 8	"	Almost insoluble cold.		
30	Mineral oils .....	"	17.7	Soluble		
		"	"	Insoluble cold		

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molecular weight come over first. Erlenmeyer and Hell's method (Annalen, 160, 226) consists in fractional saturation with silver carbonate, the acids of highest molecular weight separating first. Barré (Comptes rend. 68, 122) converts the acids into their ethyl ethers, and separates these by fractional distillation, whilst Luck (Zeits. f. analyt. Chem. 10) has devised a method founded on the different solubilities of the barium salts of the acids in alcohol. According to Lieben, Liebig's method is not as accurate as Liebig considered, but a satisfactory separation can be obtained by suitably modifying it, and by partially neutralising the mixture it is always the higher acids which distil off, whereas the lower remain behind as salts. The author, at Professor Lieben's suggestion, has tested the method by applying it to equivalent amounts of mixtures of the following acids:—Formic and acetic, acetic and propionic, acetic and butyric, acetic and isobutyric, propionic and butyric, butyric and caproic acids. In order to separate them the acids are mixed in an aqueous solution with 0.8 of the theoretical amount of alkali, and distilled so long as the distillate is acid. The residue is then decomposed with 0.6 of the theoretical amount of dilute sulphuric acid and again distilled, and finally, after adding more sulphuric acid, the last fifth is distilled off. The first fraction contains the higher and the last the lower acids in an almost pure state. The mixture of butyric and isovaleric acids could not be separated by this method, a result in variance with the statements both of Liebig and of Veil.—T. A. L.

*Valuation of Tallow by Determination of the Solidification Point of its Fatty Acids.* F. Wolfbauer. Mitth. k.k. Tech. Gewerbe-Museums in Wien, 1894, 57—68.

In order to obtain concordant results when different observers examine tallow as regards its "titre," or solidification point of the fatty acids obtained by saponification, &c., it is necessary that a uniform mode of working should be adopted; for this purpose the author modifies "Dalcian's process" as follows. 120 grms. of the tallow to be tested are heated in a beaker to a temperature but little above the melting point, and 45 cc. of caustic potash solution added (sp. gr. 1.509, containing 48 per cent. of KOH); the whole is well stirred until a uniform magma is produced, and then allowed to stand two hours in a chamber at 100°, the beaker being covered with a clock glass, and the whole stirred from time to time. A sample is then tested by warming with 50 per cent. spirit; if a clear solution is produced, the saponification is complete; if not, the heating at 100° is continued until a sample does show a clear solution. 165 cc. of dilute sulphuric acid of sp. gr. 1.113 are then added (or 22 cc. of concentrated acid and 150 of water), and the whole boiled (preferably in a silver dish) until the separated fatty acids are perfectly clear. After cooling (by floating the dish on cold water), the watery fluid is separated, and the fatty acids boiled up with 5 per cent. sulphuric acid solution, and then repeatedly with water to wash out mineral matter. The fatty acids are then dried at 100° for two hours, and transferred to a thin-walled test tube, fitted with and surrounded by a cork, which itself fits into an empty wide-mouthed bottle. A thermometer fitted through a cork into the test tube, is immersed in the fatty acids up to 33°, a bulb being blown on the stem between 2° and 28°, so as to shorten its length by some 26°; stirring is kept up (using the thermometer as a stirrer) until the mass becomes distinctly turbid, showing that incipient solidification has set in; at this stage the thermometer indicates a slight rise. For some minutes a stationary temperature is then registered, the disengagement of latent heat as solidification progresses balancing the loss of heat by radiation, &c. This stationary temperature is the "titre" of the tallow. Duplicate valuations should agree within 0.1° C., the thermometer scale being divided into fifths of a degree; the fatty acids should be tested with alcohol and ammonia to make sure that saponification is complete.

A number of specially made experiments as regards possible sources of error and of want of concordance, yielded the following results. As regards the saponification process, no difference in the titre finally deduced is

observable whether watery or alcoholic caustic potash solution be used, provided that the alcohol be completely washed out from the soap by 1½ hours' boiling with a litre of water; nor does the time of heating affect the result, the same value being obtained in two duplicate experiments, in which the times were respectively 1½ and 15 hours. On the other hand, incomplete removal of water during the drying of the fatty acids tends to lower the titre by some tenths of a degree, as also does the use of a narrower test tube. The size recommended is 35 mm. diameter and 180 long, holding 105 cc. of melted fatty acids when filled to about 10 mm. from the top, requiring fully 120 grms. of tallow. Comparative trials with tubes of this size and of 25 mm. diameter (the lengths being the same) gave values averaging 0.2 lower with the narrower tube; on the other hand, no measurable increment in value was observed with tubes of double width, 70 mm. When the thermometer employed is constructed as above described, so that the quicksilver thread projects but little above the cork of the test tube, the correction requisite for the expansion of the cooler outer portion of the thread is practically inappreciable; but if a thermometer of ordinary construction is employed, so that a considerably longer thread projects, the correction is not negligible, amounting to upwards of 0.1° C.

—C. R. A. W.

*The Methods of Testing Fats and Oils.* E. Millian. Jour. Franklin Inst. 1893, 136, 376—388 and 433—442.

THIS paper has already appeared in this Journal, 1893, 714. There is now added a table (see pages 179 and 180) comprising the constants and characteristics of the vegetable and animal oils and fats.—A. G. B.

*The Determination of Casein in Cows' Milk.* L. L. Slyke, J. Amer. Chem. Soc. 15, 635—645.

THE methods of Hoppe-Seyler and Ritthausen have been commonly employed for determination of casein in cows' milk. The milk is diluted, acetic acid added and precipitation is rendered complete either by warming to 40° C. or by passing carbonic acid through the liquid. The precipitated casein is washed with ether to free it from fat (a tedious operation), dried, and weighed. In the present research, Kjeldahl's method of nitrogen determination was used, a method now much employed in analysis of dairy products. The factor 6.25 is used to convert the nitrogen into casein. In fresh milk 10 grms. are diluted to 100 c.c. at about 41° C., and 1.5 cc. of a 10 per cent. solution of acetic acid is added. Stir and let stand three to five minutes. Decant on filter, wash a few times with cold water. If filtrate not clear, re-filter. The washed precipitate and filter paper are digested as in the Kjeldahl method. In old milk, which shows a marked development of lactic acid, the author finds that the results of casein determination are only approximate. Milk may, however, be preserved from the development of lactic acid, so that the casein may be determined after standing some days, if one part of finely-powdered mercuric chloride is added to 2,000 parts of milk.—V. C.

ANALYTICAL AND SCIENTIFIC NOTES.

*The Diffusibility of certain Gases through Caoutchouc Membranes.* A. Reyher. Bull. Soc. Chim. [3] 9, 404.

ACCORDING to the author, Pietet's observation that sulphur and carbon dioxides diffuse more rapidly than other gases through a caoutchouc membrane, must be referred to an occlusion phenomenon. These gases have a strong affinity for caoutchouc and are rapidly absorbed by one side of the membrane and as rapidly exhaled by the other side. The true diffusion process is said to proceed more slowly.

—H. T. P.

## New Books.

CHEMISCH-TECHNISCHES REPERTORIUM. Uebersichtlich geordnete Mittheilungen der neuesten Erfindungen, Fortschritte und Verbesserungen auf dem Gebiete der technischen und industriellen Chemie, mit Hinweis auf Maschinen, Apparate und Literatur. Herausgegeben von Dr. EMIL JACOBSEN. 1893. Erste Halbjahr. Zweite Hälfte. Berlin: R. Gaertner's Verlagsbuchhandlung. Hermann Heyfelder, Schönebergerstrasse 26, S.W. London: H. Grevel and Co., 33, King Street, Covent Garden.

THE second issue for the first half of 1893. It contains 158 pages of well-illustrated subject matter. The following are the groups of Chemical-technological subjects treated of in the volume:—Food-stuffs. Paper Photography and its Applications. Residuals and their Treatment. Refuse Matters. Manures. Disinfection and Sanitation. Soaps. Explosives. Preparation and Purification of Chemicals. Chemical Analysis. Apparatus. Machinery. Electro-technology and Thermo-technology. Appendix, treating of Adulterations of Trade Products, &c. New Books.

HANDWÖRTERBUCH DER PHARMACIE. Herausgegeben von A. BRESTOWSKI. Wien und Leipzig: Wilhelm Braumüller. K.U.K. Hof- und Universitäts Buchhändler. 1893. London: H. Grevel and Co., 33, King Street, Covent Garden.

VOLUME II. of Brestowski's Dictionary of Pharmacy commences with Parts 13 and 14 of the entire work. These numbers have now been issued. Part 13 commences under the letter L, with "Lab," a secretion in the juices of the stomachs of mammals, and ends with "Magnesium"; whilst Part 14 commences with "Magnesium Acetate" and ends with a monograph on "Mineral Waters," which is to be continued in Part 15.

## Trade Report.

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

(From the Board of Trade Journal.)

#### NEW CUSTOMS LAW OF THE ARGENTINE REPUBLIC.

The following is a statement of some of the rates of duty leviable under the new law:—

All goods imported from abroad for consumption in the Argentine Republic will pay a duty of 25 per cent., with the exception of the following, which pay as follows:—

40 per cent.—Dry gelatin plates of bromide of silver.

5 per cent.—Fire-bricks; soda-ash, specially intended for use in the industries of the country; nitrate of soda; sulphur, unpurified; and sulphate of lime.

The following will pay specific duties:—

Articles.	Rates of Duty.	
Starch.....	Kilo.	Dols. 0·025
Vegetable oils.....	"	0·12
Sugar, unrefined.....	"	0·003
" refined.....	"	0·07

Articles.	Rates of Duty.	
Candles, stearine or paraffin.....	Kilo.	Dols. 0·15
Chocolate.....	"	0·60
Copper ingots.....	"	0·20
" sheets.....	"	0·10
Drugs used for curing sheep scabs.....	"	0·0125
Tin.....	"	0·02
Pecula.....	"	0·0075
Glycerin, less than 30°.....	"	0·02
Lead.....	"	0·015
Salt, common.....	Hectolitre	0·20
" fine.....	100 kilos.	2·00
Stearine.....	Kilo.	0·14
Zinc, ingots and bars.....	10 kilos.	0·05

The following articles will be free of duty:—Pasteur filters; fulminate for dynamite; fuses for mines; hops; mercury; naphtha, petroleum, impure; pulp for paper making; gunpowder for mines.

#### SWITZERLAND.

##### Customs Decisions.

Note.—Quintal = 220·4 lb. avoirdupois. Franc = 9  $\frac{6}{10}$  d.

The following decisions affecting the classification of articles in the Swiss Customs tariff were given by the Swiss Customs authorities in the months of October, November, and December last:—

Anhydrous ammoniac, chlorine, oxygen, sulphurous acid, compressed, liquid, in receptacles of wrought iron.—Category 74, duty 2 francs per quintal.

In categories 479 to 481 and 484 the weight of 180 grms. per square metre is considered as the limit between paper of two or more layers and cardboard paper, in the sense that the products weighing up to 180 grms. are still classified as paper, while those exceeding a weight of 180 grms. pay duty as cardboard paper.

#### FRANCE.

##### Customs Decisions.

Note.—Kilo. = 2·204 lb. avoirdupois. Franc = 9  $\frac{6}{10}$  d.

The following decisions affecting the classification of articles in the French Customs tariff have recently been given by the French Customs authorities:—

An alloy of copper, zinc, and a small proportion of manganese and of iron, known under the name of "Delta metal," pays duty as copper alloyed with zinc, raw or manufactured, as the case may be.

Nitro-naphthalene.—Category 280, 2nd paragraph. Duty 15 francs per 100 kilos.

Artificial essences of rum, cognac, &c., or of fruits consisting of alcoholic solutions of ether still containing alcohol, are classified as chemical products not distinguished with an alcoholic base. (Category 282, 1st paragraph.) Under this category they pay the Customs duty of 80 frs. and the interior consumption tax (156·26 frs.) on the proportion of alcohol which they contain.

Carbolic soap is dutiable at 80 centimes or 60 centimes per kilo. net.

Soaps and other products with a basis of pine extract are prohibited from being imported. (Category 316, 2nd paragraph.)

#### UNITED STATES.

##### Customs Decisions.

The following decisions respecting the construction to be given to Acts of Congress relating to the classification of

articles in the Customs tariff, and the application of the Customs laws of the United States, have recently been given by the Customs authorities in that country.

White fusible enamel, to be used in the manufacture of watches, is dutiable as fusible enamel at 45 per cent. *ad val.* under paragraph 122.

White enamel, cylindrical in form, one-eighth of an inch in diameter, fusible, and commonly used by jewellers in the manufacture of sleeve buttons, watch dials, clock faces, and for other purposes, is dutiable at 45 per cent. *ad val.* under the provision in paragraph 122 for fusible enamel.

Certain "marine oil," a combination of rape-seed oil, cotton-seed oil, low-grade olive oil, and possibly some other expressed oils, prepared after a secret formula and imported for use in the manufacture of marine lubricating oil by blending it with a certain domestic mineral oil, the name of which is not divulged, is dutiable as rape-seed oil at 10 cents per gallon.

Crude eosine, one of the so-called resorcinol colours and a coal-tar preparation, chemical compound, and a coal-tar colour or dye, is dutiable at 35 per cent. *ad val.* under the provisions of paragraph 18 N.T.

Sodium salicylate powder, a medicinal preparation not containing alcohol, and in the preparation of which alcohol is not used, is dutiable at 25 per cent. under paragraph 75.

Ether butyric is dutiable at 2.50 dollars per pound, as a fruit ether or essence under paragraph 25 N.T.

Sodium benzoate, a medicinal preparation not containing alcohol, and in the preparation of which alcohol is not used, is dutiable at 25 per cent. under paragraph 75 N.T.

#### BOUNTIES ON THE MANUFACTURE OF SHALE OILS FOR LIGHTING PURPOSES IN FRANCE.

Lord Dufferin, Her Majesty's Ambassador at Paris, in a despatch to the Foreign Office, dated the 8th January, encloses copy of a French law granting bounties on the manufacture of shale oils for lighting purposes.

This law provides that, dating from the 12th July 1893, and for a period of six years, there will be allowed to the extractors of bituminous schist, intended for the manufacture of shale oils for lighting, bounties to an amount not exceeding 300,000 francs annually, and fixed according to the proportional quantities of raw oils manufactured, without in any case the bounty exceeding three francs per hectolitre of raw oil.

Further regulations will determine the conditions for the application of the law.

Any person proved guilty of fraud or attempted fraud in connection with the obtaining of a bounty will be debarred from benefiting by the bounty and will be liable to the penalties provided in Art. 423 of the penal code.

#### GENERAL TRADE NOTES.

##### PROPOSED CHANGES IN THE UNITED STATES PATENT LAW.

House Bill No. 5,014, now under consideration by the Committee on Patents of the House of Representatives, contains some new provisions affecting holders of patents. The important sections of the Bill are the following:—

"No person shall be debarred from receiving a patent for his invention or discovery, nor shall any patent be declared invalid by reason of its having been patented or caused to be patented in a foreign country less than two years prior to the application for a patent on the same invention in this country, but every such patent hereafter granted shall be limited in duration to the term of 17 years from the time when the earliest foreign patent commences to run.

"That section 4921 of the Revised Statutes be amended by adding the following: but hereafter, whenever a patent is alleged to be infringed, patentee or his representative shall seek remedy by bringing suit against manufacturer or vendor of the article alleged to infringe said patent, and shall in no case bring suit against any individual

who shall have purchased, in good faith, article of a regular dealer in the open market for his own use until the patent has been sustained by the court; provided, that such individual purchaser shall give to patentee or his representative, at his request, the name and residence of the party from whom said article was purchased; and where the damage so claimed is less than 50 dollars, plaintiff shall pay costs of suits of both defendant and plaintiff; and, provided also that this exemption from liability of individual purchasers shall not apply to any corporation or party as to any patented machine or process made or used by them. Actions at law or suits in equity for infringements of patent rights may be brought where the infringement occurs, whether defendant be domiciled therein or elsewhere."—*Engineering and Mining Journal*.

#### THE NEW YORK DRUG TRADE AND THE TARIFF.

The Drug Trade Section of the New York Board of Trade have unanimously decided to present to the Senate and House of Representatives, a memorial containing certain recommendations for amendment of the proposed new tariff bill in the schedules which affect the drug trade. It is recommended that specific duties in preference to *ad valorem* duties be levied, wherever practicable, upon articles pertaining to the drug and chemical trade, because, as these articles are imported in comparatively small quantities, the argument about false weighing does not apply, and the experience of many years proves that specific duties favour the importation of purer and higher grades of drugs, while *ad valorem* duties induce false invoicing by dishonest parties.—*Chemist and Druggist*.

#### THE DENATURING OF ALCOHOL IN GERMANY.

In the course of the debate in the Reichstag on the credits for the Ministry of Home Affairs, the socialist deputy Wurm, who is himself a chemist, asked that the Inland Revenue authorities should adopt a method of denaturing alcohol which would be less injurious to the health of the men engaged in the work. The director of the Board of Health admitted the fact of the noxiousness of the pyridine process, and regretted it, but pointed out that in spite of much experimenting no less physically injurious and otherwise satisfactory process had yet been discovered.—*Ibid*.

#### THE EXPORTATION OF TINCTURES.

In a general order (No. 3, 1894) dated February 10, and issued from Somerset House, it is ordered that tinctures made by licensed rectifiers and compounders may be exported on drawback, by weight or by measure, in iron drums ranging in size from 2 to 10 gallons, and that such drums need not be packed in cases. The tinctures may also be exported by weight or by measure, in earthenware jars ranging in size from 1 to 5 gallons, provided the jars be placed in cases. It is also ordered that no case of tinctures exported in bond shall contain less than 2 gallons, and that any internal package larger than half a gallon shall contain not less than 1 gallon, 2 gallons, &c., or 8 lb., 16 lb., &c. Decimal fractions are to be shown in the schedule of the notice when the exportation is according to weight. The weight per gallon and the bulk quantity should be shown in two decimal places, and the proof to three decimal places. When the weight per gallon as ascertained in the laboratory, taking the average as in the case of strengths, does not exceed the declared weight by more than  $\frac{1}{16}$  lb., the declared weight may be passed as correct. If the average excess is greater, special directions as to the basis of calculation will be issued by the board. When tinctures are exported by measure, officers are to check the contents of bottles by seeing not fewer than one in twenty emptied into a suitable and stamped imperial measure provided by the exporter, and if jars or drums be used, one in every five must be checked. If the exportation be conducted by weight, the quantity in the bottles, jars, or drums, must be checked in the same proportions by measure or by weight, at the exporter's option. Drawback will in future be allowed on toilet vinegars and waters, dentifrices, hair washes, and

brilliantines, according to the quantity of proof spirits present, whether the strength can be ascertained by the hydrometer or not.—*Ibid.*

#### PRODUCTION OF LIME JUICES AND CITRIC ACID.

*Galbraith. J. Anal. and Appl. Chem.* 1893, 7, 310—314.

The lime industry is carried on in a more systematic manner in Montserrat than in any other islands of this group; indeed, the area in Montserrat now under cultivation is about 1,200 acres, of which about 900 acres contain fruit-bearing trees. There is a considerable quantity of lime juice manufactured in Dominica, but there the orchards are smaller, and, with one or two exceptions, the same care is not bestowed in the cultivation of the trees and the manufacture of the juice. The industry is, however, growing, and if the Government succeeds in making roads through the island, it is anticipated that there will be a large increase in the cultivation of the trees. The few trees scattered over the islands of Antigua and St. Christopher suffice only to supply the inhabitants with fruit for their own use. (See this Journal, 1892, 783.)

The fruit begins to ripen in June, when the harvest commences, and reaches its height in August.

Each orchard (in full bearing) yields an annual average of 60—80 barrels per acre, which it is thought could be augmented by careful cultivation and manuring. A barrel of fruit yields from six to eight gallons of juice, containing 12 to 15 oz. of citric acid per gallon. No efficient remedy has yet been found for blight, which causes much trouble and materially affects the yield of fruit.

The exported produce from a lime plantation is: "raw" and "concentrated" lime juice, green and pickled limes, and essential oil. "Raw" lime juice is simply the juice from good, sound, ripe fruit carefully preserved in casks. For the best quality the limes are crushed between the rollers of a mill, old cane mills being usually employed. Most of the raw juice comes to the English market. In the manufacture of concentrated lime juice the fruits are not selected; on the contrary, the fruits which are unfit for manufacturing "raw" juice are made into "concentrated" juice. The process consists in boiling the juice in open pans until it is reduced to about one-tenth its volume: it is then a black viscid fluid containing 80—100 oz. of citric acid per gallon. The concentrated juice is shipped in second-hand beer casks principally to the New York market.

The small quantity which is shipped of green limes goes to the English market; the fruit is carefully selected, wrapped in paper, and packed in crates.

A small quantity of sound ripe limes are also treated with strong brine in casks, which are closed and rendered airtight, and exported: limes preserved in this manner are invariably sent to Boston.

Essential oil is of two kinds: the hand-made, which is the most valuable, and that prepared by distillation. The common mode of extracting this oil from the lime consists in the use of a shallow concave metal plate shaped like an ordinary saucer, in which are studded a number of blunt copper spikes, from the centre of which there is a tube into which the oil runs as it escapes from the rind of the fruit. The tube serves as a handle, and the lime fruit is gently and quickly rolled over the blunt copper spikes until all the oil sacs are burst and the oil escapes and runs into the tube. When full, the tube is emptied into bottles and securely corked. The bottles are allowed to remain until the water and other impurities settle, when the oil is carefully drawn off, filtered, and run into tinued copper vessels for export. Most of the oil exported from Dominica is manufactured by distillation; it is chiefly employed for scenting soaps, and in the manufacture of common essences and perfumes.

The price of the products is about the same in both England and the United States (New York). The average annual shipments from Montserrat for the last five years are as follows:—Raw lime juice, 800 puncheons (120 gallons each); concentrated juice, 200 casks (54 gallons each); green limes, 1,000 boxes; pickled limes, 300 barrels; essential oil, 2,500 pounds.—A. R. L.

#### MINERAL PRODUCTION OF THE UNITED STATES.

The annual report upon the mineral resources of the United States for the calendar year 1892 to the Chief of the Division of Mining Statistics and Technology of the Geological Survey is issued. The summary shows the total value of the mineral products of the country for that year to have been 137,075,176¢, the highest ever known. The following table gives the amount of the production of the several more important articles:—

Products.		Quantities.
Lead.....	Tons	213,262
Zinc.....	"	87,260
Quicksilver.....	Flasks	27,993
Petroleum.....	Barrels	50,509,136
Lime.....	"	65,000,000
Cement.....	"	8,758,621
Salt.....	"	11,998,890
Phosphate rock.....	Tons	681,571
Mineral waters.....	Gallons sold	21,876,604
Zinc, white.....	Tons	27,500
Potters' clay.....	"	420,000

—*Chemical Trade Journal.*

#### COAL DUST IN MINES.

A blue book has been issued, containing a report made by Mr. Henry Hall, one of Her Majesty's Inspectors of Mines, to the Royal Commission on Explosions from Coal Dust in Mines, giving the result of a series of experiments made with coal dust collected from the principal seams in various mining districts. Altogether 52 samples were received, and (with the exception of four or five) all were tested in a mine shaft placed at Mr. Hall's disposal by the proprietors of the White Moss Colliery, Skelmersdale, a wrought-iron cannon being used for the gunpowder shots. Mr. Hall says that the phenomena presented by these experiments as they progressed, and a careful examination of the detailed results, warrant the following conclusions:—

(1) That the flame from a blowing-out gunpowder shot in the presence of dry coal dust always ignites more or less of such dust, and so increases the burning and charring effects of the shot; (2) that when a large flame, such as that of a blowing-out gunpowder shot, or the flame from the ignition of a small quantity of firedamp, traverses an atmosphere containing a very moderate quantity of dry coal dust, the dusty atmosphere will explode with great violence, and the explosion will continue on and pass throughout any length of such atmosphere, its violence and force increasing as it progresses; (3) that coal dust from several seams in different districts, notably those from Glamorgan, Monmouth, Durham, Lancashire, Yorkshire, and Scotland, are almost as sensitive to explosion as gunpowder itself; (4) that coal dust is, as a rule, more sensitive to explosion in proportion to its high quality and freedom from impurities; (5) that a ready supply of oxygen, such as is supplied by a brisk ventilation, has the effect of making coal dust explosions more probable and more severe; (6) that certain "high explosives" are incapable of igniting or exploding coal dust. Of the whole of the dusts tested, that from the Allion Colliery, Glamorgan (Aberdare or Merthyr 4-ft. seam or upper 4 ft.), excelled all others in violence and sensitiveness to explosion, and this seam has the worst history of any in the kingdom, upwards of 1,600 persons having been killed in it by explosions since the year 1845. It was also evident from the experiments that the higher the quality of the coal seam the more liability there is to explosions of dust. With regard to precautionary measures to be taken in the face of these facts, Mr. Hall urges the total abolition of gunpowder from coal mines, and the substitution of

certain "high explosives." Many of the largest firms in the country have, he says, already of their own motion taken this step.—*Ibid.*

#### INTERNATIONAL STANDARDS FOR THE ANALYSIS OF IRON AND STEEL.

The Sub-Committee on Methods, the composition of which is given below, has just issued its bulletin No. 1, from which we take the following statements concerning its organisation and proposed work:—

At the World's Congress of Chemists, in Chicago, last August, following the papers of Professor J. W. Langley, "On the Work of the Committee on International Standards for the Analysis of Iron and Steel," and of Dr. C. B. Dudley, "On the Need of Standard Methods for the Analysis of Iron and Steel with some Proposed Standard Methods," was a brief discussion, which resulted in the reference by that body of the whole subject of standard methods for the analysis of iron and steel, to the Committee on International Standards for Analysis of Iron and Steel. That committee, it will be remembered, consists of seven chemists, in each of five different countries, namely, England, France, Germany, Sweden, and the United States. The American Committee was appointed jointly by the American Society of Civil Engineers and the University of Michigan, with Professor J. W. Langley, Case School of Science, Cleveland, Ohio, as chairman. The other members of that committee were W. P. Barba, Midvale Steel Works, Nicetown, Philadelphia; A. A. Blair, Philadelphia; Professor Regis Chauvenet, President, State School of Mines, Golden, Col.; Professor T. M. Drown, Massachusetts Institute of Technology, Boston; Dr. C. B. Dudley, Chemist, Pennsylvania Railroad, Altoona, Pa.; and Porter W. Shimer, Easton, Pa.

Following the reference of the subject to this committee, it was decided, after consultation, to appoint a sub-committee to take up the question of standard methods. The sub-committee is constituted as follows:—W. P. Barba, A. A. Blair, T. M. Drown, Porter W. Shimer, and C. B. Dudley, chairman. The sub-committee held an organising meeting at the office of A. A. Blair, Philadelphia, on December 13th, all the members being present. The object of the meeting was to map out the work. It was agreed as follows:—

First.—That Mr. Blair should submit a form of circular to go to the iron and steel chemists of the country, asking for a brief outline of the methods which they prefer, and the reasons for all the important points of their methods.

Second.—That the work of the committee should comprehend the recommendation of standard methods to be used as the basis of commercial transactions, and when any of these methods could not be used in steel works in daily practice, on account of time required, an alternative rapid method should be recommended, and its limitations defined.

Third.—That the members of the committee should draw up each proposed standard method in writing, with some minuteness, and give the reasons for each important point, these written drafts to be sent to the chairman, to be duplicated, and sent to every member of the committee. Later, the points agreed upon are to be edited by some one member of the committee.

Fourth.—That only one element should be embraced in a method.

Fifth.—That the first method to be taken up should be phosphorus in steel.

Sixth.—Mr. Barber offered to furnish to each member of the committee, a suitable quantity, not less than a pound or so, of borings of three different kinds of steel, namely one of from 0.01 to 0.02 phosphorus, carbon about 0.90, and silicon about 0.40; another with phosphorus not far from 0.06, carbon 0.50 to 0.60, silicon 0.25 to 0.30, and arsenic 0.15 per cent. The above two to be crucible steel. Another sample of open-hearth steel of carbon 0.90 to 1.05, phosphorus 0.02 to 0.04, manganese 0.30 to 0.40, silicon 0.26 to 0.25, sulphur 0.02 to 0.04, and copper anywhere below 0.10.

Seventh.—Dr. Dudley offered to furnish to each member of the committee, a like amount of borings from a sample

of Bessemer steel of from 0.10 to 0.12 phosphorus, carbon about 0.50, manganese 0.80 to 1.00, silicon 0.02 to 0.05, sulphur 0.07 to 0.10, and copper from 0.07 to 0.10. These samples of steel to be used in deciding various questions that may come up in regard to proposed methods.—*Engineering and Mining Journal.*

#### BOARD OF TRADE RETURNS.

##### SUMMARY OF IMPORTS.

Articles.	Month ending January 31st.	
	1893.	1894.
	£	£
Metals.....	1,550,125	1,726,970
Chemicals and dyestuffs.....	812,283	763,642
Oils.....	601,297	665,342
Raw materials for non-textile industries.	2,246,901	2,752,124
Total value of all imports....	33,126,479	38,458,613

##### SUMMARY OF EXPORTS.

Articles.	Month ending January 31st.	
	1893.	1894.
	£	£
Metals (other than machinery) ....	2,632,783	2,677,815
Chemicals and medicines .....	728,274	729,019
Miscellaneous articles.....	2,162,905	2,068,539
Total value of all exports.....	18,026,019	18,154,880

##### IMPORTS OF METALS FOR MONTH ENDING 31ST JANUARY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Copper:—			£	£
Ore..... Tons	5,389	6,474	36,821	29,175
Regulus .....	10,404	6,457	289,176	160,512
Unwrought .... "	2,811	5,793	135,767	251,633
Iron:—				
Ore..... "	394,514	343,112	277,616	249,777
Bolt, bar, &c.... "	3,395	2,683	28,410	21,064
Steel, unwrought.. "	365	1,615	4,454	17,311
Lead, pig and sheet ..	12,653	15,927	126,413	152,272
Pyrites .....	59,708	61,524	169,026	161,313
Quicksilver..... Lb.	87,350	13,800	7,757	1,662
Silver ore..... Value £	..	..	192,063	275,203
Tin..... Cwt.	30,179	78,033	139,517	293,069
Zinc..... Tons	3,674	3,308	68,929	54,149
Other articles ... Value £	..	..	134,001	126,168
Total value of metals	..	..	1,550,125	1,726,970

## IMPORTS OF OILS FOR MONTH ENDING 31ST JANUARY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Cocoa-nut..... Cwt.	4,677	17,026	£ 5,044	£ 21,622
Olive ..... Tuns	1,344	1,912	48,245	69,239
Palm ..... Cwt.	98,777	94,451	120,219	111,712
Petroleum ..... Gall.	13,685,436	14,130,526	245,944	269,151
Seed ..... Tons	2,839	3,234	62,478	70,679
Train, &c..... Tuns	680	1,343	12,330	22,946
Turpentine ..... Cwt.	23,849	13,274	28,432	14,370
Other articles .. Value £	..	..	81,605	85,623
Total value of oils...	..	..	604,297	685,342

## IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDING 31ST JANUARY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Park, Peruvian .. Cwt.	5,237	3,631	£ 11,989	£ 7,406
Bristles..... Lb.	189,964	189,875	23,689	24,220
Caoutchouc..... Cwt.	23,170	31,368	267,518	342,034
Gum:—				
Arabic..... "	5,969	4,200	17,529	9,068
Lac, &c..... "	6,998	7,083	27,602	37,475
Gutta-percha .... "	2,546	6,756	21,802	75,764
Hides, raw:—				
Dry..... Cwt.	24,276	30,962	61,650	74,848
Wet..... "	32,814	56,805	72,631	108,364
Ivory..... "	1,128	603	56,549	23,298
Manure:—				
Guano..... Tons	7	3,707	40	18,234
Bones..... "	4,270	6,280	17,962	30,662
Nitrate of soda... "	1,787	9,918	16,025	91,005
Phosphate of lime "	20,780	28,426	43,258	51,200
Paraffin..... Cwt.	47,361	89,917	53,747	91,363
Linen rags..... Tons	1,209	1,370	10,082	12,168
Esparto..... "	23,362	13,750	107,535	66,035
Pulp of wood .... "	9,781	24,715	60,590	105,084
Rosin..... Cwt.	92,800	108,864	19,424	21,135
Tallow and stearin "	139,925	160,997	186,330	220,499
Tar ..... Barrels	1,528	1,755	1,050	1,057
Wood:—				
Hewn ..... Loads	119,960	123,752	189,143	191,289
Sawn ..... "	60,498	161,619	129,620	234,824
Staves ..... "	4,734	8,002	35,833	49,151
Mahogany ..... Tons	5,191	5,416	47,290	44,646
Other articles.... Value £	..	..	768,034	821,295
Total value .....	..	..	2,246,911	2,752,124

Besides the above, drugs to the value of 75,522*l.* were imported, as against 80,151*l.* in January 1893.

## IMPORTS OF CHEMICALS AND DYE-STUFFS FOR MONTH ENDING 31ST JANUARY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Alkali..... Cwt.	2,205	6,898	£ 1,982	£ 5,682
Bark (tanners', &c.) "	9,951	19,324	3,388	6,500
Brimstone..... "	30,204	55,815	7,067	11,854
Chemicals..... Value £	..	..	98,594	137,047
Cochineal ..... Cwt.	577	804	3,416	4,729
Cutch and gambier Tons	1,479	2,322	32,211	48,216
Dyes:—				
Aniline ..... Value £	..	..	12,168	19,640
Alizarin ..... "	..	..	13,726	27,572
Other ..... "	..	..	741	882
Indigo ..... Cwt.	21,945	13,936	481,267	296,140
Nitrate of potash . "	15,027	16,281	13,358	15,458
Valonia ..... Tons	2,573	1,836	38,285	24,822
Other articles... Value £	..	..	105,542	164,200
Total value of chemicals	..	..	812,283	763,042

## EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDING 31ST JANUARY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Gunpowder..... Lb.	738,300	606,700	£ 19,269	£ 12,825
Military stores.. Value £	..	..	73,981	60,059
Candles..... Lb.	1,459,600	1,620,200	26,196	29,024
Caoutchouc ..... Value £	..	..	92,333	91,745
Cement..... Tons	22,974	27,030	41,269	44,110
Products of coal Value £	..	..	117,726	106,107
Earthenware ... "	..	..	142,298	160,322
Stoneware ..... "	..	..	11,414	10,623
Glass:—				
Plate..... Sq. Ft.	155,071	95,398	7,499	4,917
Flint..... Cwt.	6,235	6,962	15,499	15,229
Bottles..... "	57,245	47,910	26,554	22,815
Other kinds.... "	15,974	8,948	13,312	7,799
Leather:—				
Unwrought .... "	8,904	10,474	88,315	95,224
Wrought ..... Value £	..	..	20,132	21,239
Seed oil..... Tons	5,795	5,616	114,890	118,403
Floorcloth ..... Sq. Yds.	1,462,400	1,629,300	60,775	58,911
Painters' materials Val. £	..	..	113,856	106,794
Paper ..... Cwt.	69,340	69,477	116,029	114,800
Rags..... Tons	4,715	3,702	33,120	21,588
Soap..... Cwt.	45,189	53,024	46,192	53,734
Total value .....	..	..	2,162,905	2,068,539

**EXPORTS OF METALS (OTHER THAN MACHINERY) FOR  
MONTH ENDING 31ST JANUARY.**

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Brass..... Cwt.	8,400	8,354	36,253	31,831
Copper:—				
Unwrought..... "	50,606	22,117	128,761	40,503
Wrought..... "	21,344	27,416	61,908	75,547
Mixed metal..... "	24,130	33,566	58,424	71,262
Hardware..... Value £	..	..	168,272	149,436
Implements..... "	..	..	94,140	101,791
Iron and steel..... Tons	197,616	161,804	1,632,738	1,406,963
Lead..... "	4,307	3,441	50,646	41,456
Plated wares... Value £	..	..	19,893	19,338
Telegraph wires ..	..	..	271,502	18,738
Tin..... Cwt.	7,197	8,803	34,306	35,212
Zinc..... "	18,864	19,489	15,925	13,536
Other articles .. Value £	..	..	57,555	60,292
<b>Total value .....</b>	<b>..</b>	<b>..</b>	<b>2,632,783</b>	<b>2,077,815</b>

**EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING  
31ST JANUARY.**

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Alkali..... Cwt.	552,303	483,812	189,180	145,863
Bleaching materials ..	106,848	94,307	44,430	37,736
Chemical manures. Tons	25,347	28,074	187,265	208,590
Medicines..... Value £	..	..	76,720	80,112
Other articles ... "	..	..	230,661	247,918
<b>Total value .....</b>	<b>..</b>	<b>..</b>	<b>728,274</b>	<b>720,019</b>

## Monthly Patent List.

\* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### APPLICATIONS.

1135. B. G. Martin and M. J. McMullin. Improvements in filtering plant for the purification of water or other fluids. January 18.

1136. J. E. Carroll. An improved process for purifying water and other liquids, and improvements in apparatus for eliminating soluble salts therefrom. January 18.

1303. S. Trivick, C. J. A. Head, and P. A. J. Head. Improvements in reversing regenerative furnaces for metallurgical and other purposes. January 20.

1347. R. W. B. Creeke. Improvements in and relating to destructor furnaces. January 22.

1377. C. Morel and A. Heimpel. Apparatus for crushing dry or wet materials, such as cements, lime, plaster, alabaster, kaolin, quartz, phosphates, minerals, &c. Complete Specification. Filed January 22. Date applied for August 8, 1893, being date of application in France.

1703. W. Ramsbottom. Improvement in filters specially designed for treating sewage or other fouled waters. January 26.

1790. D. Stewart. See Class XVI.

1866. O. Hamilton. A new or improved drying apparatus. January 29.

2085. J. J. Meldrum and T. F. Meldrum. Condensers. January 31.

2230. T. Drost. An improved centrifugalling process and apparatus therefor. February 1.

2588. J. Nagel. Apparatus for distilling and sterilising water. Complete Specification. February 6.

2915. D. Wickham. Improved apparatus for measuring still or gaseous liquids. February 9.

2944. R. A. Grosse. Improvements in toluol thermometers. February 10.

#### COMPLETE SPECIFICATIONS ACCEPTED.\*

1893.

2547. D. H. Lyon.—From D. Hanna. An improved process for separating impurities from water introduced into a boiler, and apparatus therefor. February 7.

4421. G. W. Elliott. Improvements in centrifugal hydro-extractors. January 31.

5375. J. Wetter.—From M. Weinrich. Improvements in the manufacture or restoration of filtering media. January 24.

6243. C. K. Mills.—From A. Costadan. An improved joint for steam, water, gas, and other liquids or fluids. February 7.

6316. H. J. Worssam. Improvements in refrigerators for cooling and evaporating liquids, such as are used in breweries and distilleries, and for such like purposes. January 31.

6408. J. F. Jones. Improvements in apparatus for cooling or heating air or other gases. January 31.

13,929. A. Müller. An improved valve for gases and liquids under high pressure. January 24.



15,488. J. V. Smith. Improvements in method of and apparatus for promoting consumption of fuel. January 24.  
20,549. C. W. Davis. Improved apparatus for mixing or sifting moist or dry powders. January 31.

21,850. H. A. Naber. Improvements in voltmeters. January 24.

23,045. R. Haddan—From F. D. Cummer. Improvements in and connected with the process of and machinery for the drying or heating of gypsum clay, sand, grain, slaughter-house refuse, and other materials. January 31.

23,367. L. Wagner and J. Marr. See Class XVII.

24,165. E. Stern. A new method of closing receivers charged with compressed or liquefied gases. January 24.

## II.—FUEL, GAS, AND LIGHT.

### APPLICATIONS.

999. J. H. Lee. Improvements in method of and apparatus for supplying air richer in oxygen than atmospheric air to steam boiler and other furnaces, and for separating and collecting air richer in oxygen on the one hand and in nitrogen on the other, for the various purposes to which air rich in these qualities may be supplied. January 16.

1029. G. St. J. Kueller. Artistically-shaped fuel for use with every kind of gas fires. Complete Specification. January 17.

1188. S. C. Hitchcock. An improved mineral oil. January 19.

1190. F. McNamee. Improvements in the method of and connected with apparatus for drying peat for fuel, litter, or similar use or purposes. January 19.

1231. F. B. Hill and R. D. Brett. Improvements relating to the burning of hydrocarbons for heating and lighting purposes, and to apparatus therefor. January 19.

1238. D. Hancock, J. B. Craig, and A. H. Hancock. Improvements in or relating to the employment of air for lighting and heating purposes. January 19.

1409. S. Williams. An improvement in candles. January 23.

1485. T. H. Jones. Improvements relating to the carbonising of wood for the production of charcoal, and in the utilisation of the products given off in the process of carbonisation. January 23.

1486. W. B. Hartridge. Improvements in and relating to the manufacture of fuel and to apparatus therefor. January 23.

1490. J. Galletly and H. Armour. Improvements in the manufacture of gas from mineral oils, and in the apparatus employed therefor. January 23.

1540. J. Foss. Condenser of noxious vapours from illuminants. January 24.

1653. A. McLean. Improvements in the treatment of peat. January 25.

1659. C. C. Walker. Improvements in apparatus to be used in the purification of coal-gas or for analogous operations. January 25.

1690. B. H. Thwaite and G. Threlfall. Improvements in methods and apparatus for generating gaseous fuel from solid hydrocarbonaceous or carbonaceous material. Complete Specification. January 26.

1833. H. J. Reid. Improvements in the manufacture of briquettes and patent fuel, and in means and apparatus employed therefor. January 27.

1856. J. H. R. Dinsmore. Improvements in the manufacture of illuminating gas. January 29.

1991. A. J. Boulton—From W. A. Koneman and A. F. Hatch, United States. Process of and apparatus for utilising producer-gas as fuel for calcining limestone. Complete Specification. January 30.

2426. F. H. Bacon and B. Donkin. Improvements in and appertaining to the purification of coal or other gases, and apparatus used therein. February 3.

2488. C. J. Yarnold. Improvements in the method and apparatus employed in the production of ozone. February 5.

2554. D. Young. The production or recovery of coal dust for foundry purposes and the machinery or apparatus therefor. February 6.

2630. J. Swallow, C. West, and A. R. Armitage. Improvements in or relating to the treatments of paraffin and other oils for rendering it or them odourless, smokeless, miscible, and non-explosive. February 6.

2702. T. de Zebrowski. A new or improved manufacture of briquettes or artificial fuel blocks. February 7.

2786. B. Bone. Chalk fuel. February 8.

2914. V. B. Lewes. Improvements in and in apparatus for the manufacture or production of gas. February 9.

2937. M. Graham. Improvements in and apparatus for charging inclined gas retorts. February 10.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

2247. J. C. W. Stanley. Improvements in or relating to the manufacture or production of block fuel, gas, and other useful products from refuse materials. January 31.

3554. T. A. Dillon. An improved method of preparing peat for heating, gas generating, and other purposes. January 24.

3815. J. R. Paisley. An improvement in apparatus for the manufacture of water-gas. February 7.

5920. F. Beale and T. F. Ennis. Improvements in apparatus for charging gas retorts. January 31.

5951. J. H. Fitzgerald. Improvements in gas washers and scrubbers. January 31.

6051. G. de Verna. Improvements in the manufacture of illuminating gas, artificial fuel blocks, and coke for metallurgical purposes. January 31.

6259. G. S. Ellis and A. M. Dolton. Improvements in the manufacture of coal briquettes. January 31.

6506. W. Cross. Improvements in anti-dip valves, employed in the manufacture of gas. January 31.

14,783. F. S. Cripps. Improvements in washers for the extraction of tar, ammonia, carbonic acid, and sulphuretted hydrogen from coal-gas. February 14.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

### COMPLETE SPECIFICATION ACCEPTED.

1893.

5856. C. D. Abel—From F. von Heyden. Process for producing esters from fatty acids with creosote, guajacol, or creosol. January 31.

## IV.—COLOURING MATTERS AND DYES.

### APPLICATIONS.

881. J. B. Boyle and E. Fatta. Perfumed writing and printing ink. January 15.

973. H. E. Newton—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of anthraquinone derivatives. January 16.

974. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of colouring matters derived from anthraquinone. January 16.

975. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of trihydroxyanthraquinones. January 16.

1063. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of  $\beta_1$ -naphthol,  $\alpha_1\beta_1$ -disulpho acid, and  $\beta_1$ -naphthylamine,  $\alpha_1\beta_1$ -disulpho acid. January 17.

1227. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the manufacture of  $\alpha_1$ -,  $\alpha_1$ -dihydroxynaphthalene- $\alpha$ -monosulpho acid. January 19.

1228. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of acid compounds derived from gallic acid, its anhydrides, or analogous products. January 19.

1229. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of dyestuffs by means of trihydroxynaphthalene monosulpho acid. January 19.

1555. H. Loesner. Manufacture of azoxy compounds of the series. January 24.

1556. H. Schultze. An improved process for the pro-alexyphosphoric acid and of gallic dyeing prepared therefrom. January 24.

1557. J. W. Johnson.—From Kalle and Co., Germany. Improvements in the manufacture and production of new dyestuffs. January 25.

1558. S. Pitt.—From L. Cassella and Co., Germany. The production of perichloronaphthol disulpho acid and disazo dyestuffs therefrom. January 29.

2113. A. W. Gilbody. The manufacture of a new base and the production of dyestuffs therefrom. January 31.

2206. H. Loesner. Improvements in the manufacture of ortho-nitro-toluene, meta- and para-azoxyltoluene, meta- and para-toluidin. February 1.

2232. W. G. Thompson and I. Moore. Improvements in the manufacture of a diamido base and of tetrazo colouring matters derived therefrom. Complete Specification. February 1.

2324. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of colouring matters. February 2.

2325. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of hydroxyanthraquinone-quinolines. February 2.

2503. Read, Holliday and Sons, Lim., and K. B. Elbel. The manufacture of a bluish-red dye belonging to the rosinduline series. February 5.

2504. Read, Holliday and Sons, Lim., and K. B. Elbel. Improvements in the manufacture of azo-colouring matters. February 5.

2591. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of dyestuffs. February 6.

2592. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The production of azo-colouring matters upon fibres. February 6.

2758. J. Altschul. Manufacture and production of a new diazo-compound for use in dyeing and printing. February 8.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

4979. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture and production of sulpho-acids of the naphthalene series, and of colouring matters obtainable therefrom. January 31.

5141. S. Pitt.—From L. Cassella and Co. Improvement in the production of disazo dyestuffs from  $\gamma$  amidonaphthol sulpho acid. January 24.

5690. O. Imray.—From The Farbwerke vormals Meister, Lucius and Brünig. Production of colouring matters. January 24.

5691. O. Imray.—From The Farbwerke vormals Meister, Lucius and Brünig. Manufacture of acid colouring matters, being the sulphonic acids of alkyl derivatives of the diamidodiphenyl-metatoxy-carbinol. January 31.

5692. O. Imray.—From The Farbwerke vormals Meister, Lucius and Brünig. Manufacture of thionine colouring matters dyeing on mordants. January 24.

6249. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik. Improvements in the manufacture of auramine. January 31.

7181. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of a new blue colouring matter. February 14.

7182. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of new alizarin dyestuffs. February 14.

7185. O. Imray.—From The Farbwerke vormals Meister, Lucius and Brünig. Manufacture of colouring matters. February 14.

7187. O. Imray.—From The Farbwerke vormals Meister, Lucius and Brünig. Manufacture of a novel diamido base, and of colouring matters from the same. February 14.

7263. C. Blaneau. A liquid metallic composition for use as a writing or printing ink or as a colour for decorating surfaces. February 7.

21082. J. C. L. Durand, D. E. Huguenin, and A. J. J. d'Andiran. Manufacture of new colouring matters. January 31.

#### V.—TEXTILES, COTTON, WOOL, SILK, Etc.

##### APPLICATIONS.

861. A. Moritz. Improvements in fireproofing felt. January 15.

943. E. B. Manby. See Class XIV.

1089. J. Smith and P. W. Nicolle. Improvements in the treatment of vegetable fibrous matters to obtain fibres therefrom. January 18.

1218. H. H. Boyle. Improvements in machinery for decorticating fibrous grasses and such like substances. January 19.

1267. D. Dregborn and J. Harvey. Improvements in and relating to the treating and scouring of wool, and in the recovery of wool grease, potash, salts, or like products therefrom. January 20.

1364. A. G. Speight. Treating wood-wool to diminish its combustibility. January 22.

1890. J. Marshall, D. H. Thornton, F. H. Cliffe, and W. Cliffe. A new and improved method of and apparatus for preparing and finishing woven fabrics or textile piece goods. January 29.

1948. F. Crosland. An improved composition for rendering, paper, cloth, or other material waterproof. January 30.

2718. E. Meyer. The manufacture or production of a new yarn and of fabrics therefrom. February 7.

2817. H. Robinson. Improvements in apparatus for conditioning yarn. February 9.

#### COMPLETE SPECIFICATION ACCEPTED.

1893.

24,144. E. Samper. A new or improved machine for extracting fibrous matters from agaves, aloes, and other similar plants. February 14.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

### APPLICATIONS.

1656. J. C. Mewburn.—From La Société Leblois Piceni et Compagnie, France. Improvements in preparing for bleaching and dyeing, and in bleaching, dyeing, and washing vegetable textile material and in apparatus therefor. January 25.

2923. B. Cawthorn and J. P. Cornett. See Class XIX.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

1623. J. Clapham, J. Picard, C. Villedien, and W. W. L. Lishman. Improvements in bleaching and discoloration. January 24.

4067. E. Brook. Improvements in or connected with machines for dyeing, scouring, or drying hanks of yarn or tops. January 24.

6600. A. McMeekin. Improvements in the method of and means employed for bleaching, dyeing, and otherwise treating flax, hemp, and other fibres. February 7.

7350. W. T. Whitehead. Improvements in resists or discharges for use in printing or dyeing cotton, wool, and other fabrics with aniline black. January 31.

7351. W. T. Whitehead. Improvements in resists or discharges for use in printing or dyeing cotton, wool, and other fabrics. January 31.

21,185. L. Weldon. Improvements in dyeing machines. January 24.

## VII.—ACIDS, ALKALIS, AND SALTS.

### APPLICATIONS.

1207. E. T. Hughes.—From H. Howard, United States. Improvements in methods of and apparatus for concentrating sulphuric acid and the like. January 19.

1442. G. S. Johnson. A new compound of sodium sulphite. January 23.

1572. T. Houghton and W. Shield. Improvements in the manufacture of bleaching powder and in apparatus therefor. January 24.

1637. M. N. d'Andria. Improvements in the method of and means for the separation of magnesia from waters and aqueous solutions containing magnesium compounds. January 25.

1905. C. M. Pielsticker. An improved process and apparatus for the production of bichromate and permanganate of potassium. January 29.

2060. H. H. Lake.—From J. B. Tibbits, United States. Improvements relating to the manufacture of lead salts. Complete Specification. January 30.

2373. J. Pedder. Improved process for effecting the desulphurisation of sodic sulphate. February 3.

2486. M. N. d'Andria. Improvements in the recovery of hydrochloric acid and oxide of manganese from solutions of manganese chloride. February 5.

2618. A. Crossley. Improvements in apparatus for producing ferro-ferric and ferric oxides. February 6.

2672. H. R. Angel. Improvements in the manufacture of sulphuric acid. February 7.

2696. A. R. Davis. Improvements in the production of sulphate of manganese and oxide of iron. February 7.

2814. F. E. Matthews. An improved method of storing and utilising chlorine. February 9.

### COMPLETE SPECIFICATIONS ACCEPTED.

18,871. J. Hargreaves and T. <sup>1892.</sup>  
Improvements in the manufacture of alkali and in apparatus therefor. January 21.  
1893.

7107. F. Hurter.—From J. Omholt. Improvements in the manufacture of soda and mono calcic phosphate. February 14.

7108. F. Hurter.—From J. Omholt. Improvements in the manufacture or production of mono calcic phosphate and ammonia. February 14.

1894.

456. P. C. Choate. Improvements in the art of preparing solutions carrying salts of zinc. February 14.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

### APPLICATIONS.

878. E. Böhm. Improvements in the manufacture of enamelled glass and crystal glass letters and other devices. January 15.

955. E. F. W. Hirsch. Improvements in glass melting. January 16.

2040. P. B. W. Kershaw and B. Loftus-Tottenham. An improved method of and apparatus for the manufacture of glass balls. January 30.

2689. D. Moore. Improvements in furnaces for annealing glass. February 7.

5505. H. Watkin. Improvements in apparatus for moulding the bodies of electric switches, push-button ceiling roses, and other like articles, in china and earthenware. February 7.

6612. T. W. Twyford. Improvements in the glazing and enamelling of heavy earthenware and other goods, and in apparatus to be employed therefor. February 14.

6661. Craven, Dunnill, and Co., Lim., and F. R. Smith. Improvements in apparatus for the manufacture of tiles. February 7.

### COMPLETE SPECIFICATION ACCEPTED.

1893.

23,388. N. M. Miller. Improvements relating to the rolling of glass, and machinery therefor. January 31.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### APPLICATIONS.

1157. H. H. Lake.—From B. Gran, Denmark. Improvements relating to the manufacture of artificial marble. January 18.

1370. A. Hooydonk and H. Hooydonk. A new or improved preparation or compound for decorating the interior of buildings. January 22.

1396. H. H. Leigh.—From G. Bagge, France. Improvements in paving blocks. Complete Specification. January 22.

1427. A. McCara. Improvements in the manufacture of Portland cement. January 23.

1579. H. V. Lanchester. Improvements in the construction of fireproof and like floors. January 24.

2247. D. Belloc and E. Bénard. Improved process for transforming roasted cements into powder, and in apparatus therefor. Complete Specification. February 1.

2322. G. Briggs and M. Leighton. Improvements in the manufacture of paving flags and other articles of concrete. February 2.

2169. F. W. Gallagher and C. M. Jackson. An improved method in the formation and application of building materials, such as clay bricks, wood, stone, concrete, or blocks of other substances. February 5.

2582. C. H. Slier. Improvements in and relating to the production of asphaltic composition in the form of a powder or of a mastic. Complete Specification. February 6.

2678. W. P. Ingham. Improvements in the manufacture of fireclay, bricks, blast-furnace "bumps," and the like. February 7.

2809. H. W. Jeffery. Improvements in or connected with composite road pavements. February 8.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

1582. F. G. Montagne. Manufacture of artificial marbles. January 31.

3106. W. Webster. Improvements in the manufacture of cement. February 7.

6328. H. B. Allays. An improved process for making mosaics. February 14.

16,454. A. J. Boulton.—From L. P. Hemmer. An improved material adapted for use as a substitute for wood, metal, stone, and other substances. January 31.

17,514. W. Smith. Improved means of protecting timber structures. January 24.

### X.—METALLURGY, MINING, Etc.

#### APPLICATIONS.

998. J. H. Lee. Improved means for separating gold and other metals from other substances by amalgamation. January 16.

1186. J. Pearson and S. Law. Improvements in the method of and means employed in hardening and tempering wire. January 19.

1212. W. S. Squire and L. R. L. Squire. Improvements in the manufacture of metallic zinc. January 19.

1311. J. B. Alzugaray.—From J. E. Torres, France. Improvements in and connected with the reduction, refining, dephosphorisation, and desulphuration of ores, mattes, slags, metals, and alloys. January 20.

1472. A. H. Bell and C. Carter. An improved apparatus for reciprocating gold and silver ores from cyanide solutions. Complete Specification. January 23.

1666. A. Kainer. A new or improved process for heating masses of metal preparatorily to their being further worked. January 25.

1915. W. Klobukowski and M. M. Reicher. New and improved process for producing iron and chromium free aluminium sulphate. January 29.

2041. J. C. Fell.—From the Emmens Zinc Company, United States. Improvements in the treatment of zinc-lead-sulphide ores carrying gold or silver or gold and silver. Complete Specification. January 30.

2236. W. T. Chamberlain. Improvements in explosive metal. February 1.

2361. J. B. Alzugaray.—From J. B. Torres, France. Improvements in and connected with the reduction, refining, and fusing of aluminium, antimony, arsenic, bismuth, cadmium, gold, glucinum, lead, magnesium, silver, tin, zinc, and all other metallic elements from their ores and other compounds. February 3.

2378. G. A. Jarvis. An improvement in the manufacture of steel. February 3.

2527. E. Norton. Improvements in the art or process of manufacturing tin orterne plate. Complete Specification. February 5.

2629. W. P. Thompson.—From J. J. C. Smith and E. C. Smith, United States. Improvements in or appertaining to casting metals. Complete Specification. February 6.

2744. P. R. J. Willis.—From J. H. Hines, United States. An improved process for ornamenting metal. February 7.

2722. J. Lagache. Improved process of treating wavelite and similar minerals. February 7.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

1906. A. V. C. Fenby, J. B. Fenby, and G. Moore. Coating iron and steel with brass and other metallic alloys. January 31.

2119. A. J. Boulton.—From C. G. Richardson and A. B. English. Improvements in or relating to the treatment of metallic ores. February 7.

3583. F. E. de Fursac, J. C. Moisset, and J. P. J. Very. Processes of separating metals, covering scraps, clippings, or other pieces of metal of all kinds. January 23.

5568. T. Twynam. Improvements in the manufacture of steel and ingot iron. January 24.

7252. W. Hayward. A new or improved process for recovering zinc or spelter from waste galvanised iron and steel, and furnaces and appliances for the said process. February 7.

23,425. W. P. B. Urick. Improvements in methods of casting solid ingots of steel. February 14.

23,979. F. W. Golby.—From C. Francisci. Improvements in furnaces for distilling zinc, cadmium, and the like. January 31.

### XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

#### APPLICATIONS.

1356. E. Andreoli. Improvements in electrolysis. January 22.

1440. J. Peel. Improved electric battery. January 23.

1744. W. S. Rawson. Improvements in electro-deposition. January 26.

1903. M. Wuillot. Improvements in or relating to electrodes. January 29.

2250. O. Hund.—From G. Wehr, Germany. Improvements in galvanic batteries. February 1.

2251. O. Hund.—From G. Wehr, Germany. Improvements in galvanic dry batteries. February 1.

2487. C. J. Hall. Improvements in or relating to secondary batteries. February 5.

2806. C. Kellner. Improvements in electrolytical decomposing apparatus. February 8.

2897. C. P. Shrewsbury, F. L. Marshall, J. Cooper, and J. L. Dobell. Improvements in the manufacture of coke or carbon for electrical or other purposes. February 9.

2900. J. Rudholzner. Improvements relating to the electro-deposition of metals. Complete Specification. February 9.

2968. O. Imray.—From The Farbwerke vormals Meister, Lucius and Brüning, Germany. An improvement in electrolysing liquids and apparatus therefor. February 10.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

2267. C. T. J. Vantin. Improvements in the method of and apparatus for the electrolytical separation of the bases of salts, and of compounds of the same, from saline solutions. January 31.

3477. H. Nehmer. Improvements in constructing and connecting galvanic elements. February 14.

3573. E. Taussig. Improvements relating to the extraction of metals by means of electricity and to apparatus therefor. February 14.

5778. A. J. Boulton.—From O. Knoefler and F. Gebauer. An improved process for manufacturing bleaching solutions by electrolysis. January 24.

5844. W. P. Thompson.—From M. Hartung. Improvements in storage batteries. January 31.

19,967. W. Petschel. Improvements in secondary electric batteries. February 7.

22,956. C. A. J. H. Schroeder and H. E. R. Schroeder. New improved electro-positive galvanic battery electrodes. January 31.

23,913. R. Hadden.—From H. S. Blackmore. Improved process and apparatus for dissociating soluble salts by electrolysis. January 24.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

### APPLICATIONS.

963. F. Burton. A process for the purification of oils, fats, and the like. January 16.

1088. A. Warwick, J. Smith, and P. W. Nicolle. Improvements in the manufacture of soap and washing compounds. January 18.

1237. M. H. Tomkins and F. Woodman. An improved cleansing compound for cleaning wood, stone, and other surfaces. January 19.

1296. F. R. Donisthorpe. Improvements in or relating to the manufacture of soap. January 20.

1758. J. C. W. Stanley. Improvements in or relating to the recovery of oil from fish, fish offal, and the like. January 26.

1760. T. F. Alten. Improvements in or relating to soap. January 26.

1764. J. C. W. Stanley. Improvements in or relating to the extraction of oil or grease from other materials. January 26.

1963. A. R. Scott. Improvements in the manufacture of cleansing or soap powder for washing fibrous and other materials. January 30.

2132. E. Petit. Improvements in the acid saponification of fatty bodies and in apparatus employed therein. January 31.

2701. T. de Zebrowski. Manufacture of a new or improved soap. February 7.

2871. C. Heydemann.—From O. Dennerlein, Holland. An apparatus for melting and purifying fats. February 9.

2896. N. Iseli. Improvements in or relating to the manufacture of alcohols, oils, greases, and other matter. February 14.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

5040. The Patent Syndicate Lightning Oil Extractor Co., Ltd., and H. A. A. Dombrain. Apparatus for driving off heavy vapours of solvents. January 24.

5621. J. Peacock and W. L. Colls. Improved manufacture or production of soap. January 24.

6123. W. Brothers. Improved detergent compound or preparation for the washing or scouring of textile fabrics or other articles. February 7.

6993. R. Hutcheson. Improvements in treating wool greases and separating their constituents. February 7.

9105. C. D. Abel.—From the Fabriques de Produits Chimiques de Thann et de Mulhouse. An improved manufacture of oils, fats, wax, and other analogous substances freed from odour and flavour. February 14.

24,461. T. Cordweener and A. de Kunwald. See Class XVIII. (A.)

## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

### APPLICATIONS.

958. J. Hickisson. Improvements in marking-ink pencils. January 16.

1107. D. Forbes and C. Hoddell. Enameline. January 18.

1301. J. W. Blake and S. J. Blake. Improvements in compositions or paints for preserving ships' bottoms and other structures. Complete Specification. January 20.

1418. J. P. Lacy and A. J. Ash. Improvements in the production of zinc compounds, suitable for use as pigments and for other purposes. January 23.

2257. I. F. Clark. A substitute for veneer or enamel. February 2.

2428. D. J. Harris. A composition or solution for making waterproof, hardening, and preserving the soles of boots and shoes. February 3.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

7117. A. C. J. Charlier. Improvements in apparatus for the manufacture of metallic pigments or compounds, and in the production of lead pigments or compounds by means of such apparatus. February 14.

7571. C. D. Abel. From the Westfälisch-Anhaltischen Sprengstoff Actien Gesellschaft. Hygroscopic paint or coating material for keeping packing cases dry. February 7.

10,619. W. Ward and J. O. Wallace. A new or improved paint. February 14.

22,137. L. Paget. Improvements in pyroxyline compounds, solutions, and solvents. January 31.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

### APPLICATIONS.

943. E. B. Manby. An improved sizing, dressing, and finishing material for application to textile fibres and fabrics. January 16.

977. G. W. Ewens. A new material for use as a substitute for leather for floor coverings and for like purposes. January 16.

2079. G. E. Wiese. Improvements in the manufacture of liquid glue. Complete Specification. January 31.

2175. J. Grillo and M. Schroeder. Process for the production of glue and gelatin from bones. Complete Specification. February 1.

2517. W. H. Jeansson. Improvements in the treatment of leather. February 5.

2978. A. J. E. Hill and F. P. Hill. Improved waterproof glue. February 10.

## XV.—AGRICULTURE AND MANURES.

### APPLICATION.

2624. J. J. Seldner and J. Watson, jun. Improvement in the manufacture of fertilisers. Complete Specification. February 6.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

### APPLICATIONS.

1146. C. H. Meyer. Improvements in the manufacture from starch or starchy substances of a material suitable for use in brewing and for other purposes. January 18.

1790. D. Stewart. Improvements in centrifugal apparatus for drying and purifying sugar or other crystalline or granular matters. January 27.

1953. E. Shaw. Improved apparatus for boiling sugar and glucose, either separately or mixed. January 30.

2732. E. Shaw. Improved apparatus appertaining to the process of boiling sugar and glucose, either separately or mixed. February 8.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

7695. L. E. A. Prangey. Improvements in the continuous refining or bleaching of sugar, and in machines employed for this purpose. January 24.

1894.

136. H. H. Lake.—From P. H. V. Weyde and O. Lugo. Improvements in the manufacture of sugar. February 7.

### XVII.—BREWING, WINES, SPIRITS, ETC.

#### APPLICATIONS.

1146. C. H. Meyer. See Class XVI.

1153. F. D. Fisher. Improvements in brewing. January 18.

1717. R. R. P. Schmiedeecke and E. O. Gade. Improvements in apparatus for the preparation of malt. Complete Specification. January 26.

2889. G. G. Cave. Improvements in the treatment of yeast. February 9.

2922. W. S. Squire. Improvements in the treatment of distillers' spent wash. February 10.

2966. W. R. Reffell. An external mashing apparatus for the use of brewers and others in mashing malt with water or raw grain liquor in brewing or distilling. February 10.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

1887. F. M. Polsky. Improvements in the manufacture of yeast. January 24.

4981. F. E. V. Baines. Improvements in the treatment of rice or other grain for the production therefrom of a material suitable for use in brewing or for other purposes. January 24.

5870. H. H. Lake.—From K. Müller. Improvements relating to the clarification of beer. January 31.

23,367. L. Wagner and J. Marr. Improvements in the methods of extracting liquids from and drying grain, malt, brewers' grains, and other like substances, and in apparatus therefor. February 14.

### XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

#### APPLICATIONS.

##### A.—Chemistry of Foods.

1170. R. F. Maclosky.—From J. H. Niemann, Australia. A new or improved meat extract. Complete Specification. January 19.

1405. F. T. Bond. Improvements in the preparation of cheese. January 23.

1564. J. F. Duke. Improvements in the method of preserving eggs. January 24.

2129. W. P. Thompson.—From G. B. R. von Poschinger, Germany. An improved process and apparatus for preparing or producing a fodder material. January 31.

2445. C. Kidson. A new method of preserving eggs. February 5.

2651. T. Charlton and A. H. Cannon. Improvements in and connected with the preservation, storage, and revivification of alimentary substances. February 7.

##### B.—Sanitary Chemistry.

1086. E. C. Ives and J. Woodlatt. An improved filter for use in purifying polluted waters, sewage, and the like. January 18.

1591. W. D. Scott-Monerieff. Improvements in apparatus for the treatment of sewage. January 24.

1762. C. Salzberger. Improvements in or relating to the disinfection or purification of water. January 26.

1865. E. C. Ives. An improved system of purifying sewage or other foul waters. January 29.

##### C.—Disinfectants.

1415. I. Levinstein. Improvements in and connected with iron compounds for use in precipitating sewage, purifying waste liquors, colouring, and the like purposes. January 23.

#### COMPLETE SPECIFICATIONS ACCEPTED.

##### A.—Chemistry of Foods.

1893.

6596. E. Sonstadt. Improvements in the preparation of extracts of tea and coffee, and in utilising a by-product of the process. February 7.

10,689. M. Watson. An improved preparation of malted pulse food. January 31.

24,191. B. Haddan.—From La Compagnie du Sel Agglomere. Improvements in and relating to the treatment of salt and the formation of blocks thereof by fusion. February 14.

24,461. T. Cordewener and A. de Kunwald. Improvements in and in apparatus for the manufacture of margarine and alimentary fat. January 24.

##### B.—Sanitary Chemistry.

23,082. S. Smithson. Improvements in or relating to machinery or apparatus for treating, deodorising, drying, and utilising excreta, nightsoil, towns' refuse, and the like. February 7.

##### C.—Disinfectants.

4950. C. R. Alder Wright. Improvements in the manufacture of antiseptic and disinfectant materials for sanitary purposes. January 31.

7038. S. Pitt.—From The Chemische Fabrik auf Actien vormals E. Sehering. A new antiseptic material to be called "Formalin." February 14.

12,276. E. Soy. Improvements in vessels for containing liquefied sulphur dioxide for use as a disinfectant and vermin destroyer, and for other medical and sanitary purposes. January 31.

### XIX.—PAPER, PASTEBOARD, ETC.

#### APPLICATIONS.

1067. H. H. Lake.—From La Société J. Noé et P. Putois, France. Improvements relating to the manufacture of artificial whalebone. January 17.

1345. J. Lander. A process for expanding celluloid and celluloid tubes into any required shape. January 22.

1599. H. G. Cadywould. Damp-resisting paper. January 25.

2214. J. La-Bua. A machine for coating photographic paper tracing paper and coloured paper with one colour only. February 1.

2923. B. Cawthorn and J. P. Cornett. Improvements in or connected with the process of bleaching fibrous materials in the manufacture of paper. February 10.

#### COMPLETE SPECIFICATION ACCEPTED.

1893.

24,652. W. M. C. Callender. The manufacture of a new material designed to serve as a substitute for bone or celluloid. February 7.

### XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

#### APPLICATION.

1287. F. W. Warriek. An improved pharmaceutical preparation of ferrous salts. January 20.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

2951. J. Morris. Means for using certain chemicals, drugs, and perfumes for therapeutic, disinfecting, antiseptic, and perfuming purposes. February 14.

6596. E. Sonstadt. *See* Class XVIII. (A.)

6749. C. Kolbe. Manufacture or production of salols. February 7.

22,931. F. W. Warriek. An improved pharmaceutical preparation of ferrous salts. January 31.

### XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.

#### APPLICATIONS.

1109. T. Maloni. Improvements in and connected with lighting apparatus for taking photographs at night or in dull weather. January 18.

2036. A. Zimmermann.—From The Chemische Fabrik auf Actien vormals E. Scherl, Germany. Improvements in the treatment of gelatinous matter and the production of photographic films. January 30.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

4214. W. H. Walker. New or improved apparatus for the manufacture of films, more especially intended for the manufacture of films for photographic purposes. January 31.

5517. W. H. Prestwich. Improved silver haloid emulsions for photographic purposes. February 14.

5932. O. Magerstedt. An improvement in the manufacture of photographic plates. January 24.

6491. E. Norden. An improved method of photographic printing in bromide of silver. January 31.

6501. C. A. Allison.—From F. B. Forster and G. G. Rockwood. Improvements in processes of making transparent photographs on glass. February 14.

### XXII.—EXPLOSIVES, MATCHES, Etc.

#### APPLICATIONS.

1074. M. von Förster. Improvements in or relating to the manufacture of smokeless powder. January 17.

1665. E. Rubin. Improvements in percussion fuses. January 25.

1722. R. H. Panshon. An improved explosive compound. January 26.

1755. F. J. Smith. Improvements in detonators. January 26.

1846. G. E. Cassel and D. Kempe. An improved process of preparing pine or analogous wood for the manufacture of matches. Complete Specification. January 27.

1982. V. I. Feeny.—From The United States Smokeless Powder Co., United States. Smokeless powder. Complete Specification. January 30.

2144. F. J. Maubeuge. Improvements in combined time and percussion fuses. January 31.

6775. I. M. Meissner. An improved igniting composition for matches. February 14.

#### COMPLETE SPECIFICATION ACCEPTED.

1893.

8677. J. V. Landuyt. Improvements in apparatus for use in the manufacture of matches and for putting the same in boxes. February 7.



# THE JOURNAL OF THE Society of Chemical Industry: A MONTHLY RECORD

FOR ALL INTERESTED IN CHEMICAL MANUFACTURES.

No. 3.—Vol. X. (1.)

MARCH 31, 1894.

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## NOTICES.

Notice is hereby given in accordance with Rule 54 of the By-Laws that the Council will propose to the forthcoming Annual Meeting the amendment of the Society's By-Laws as follows:—

1. That *Rule 7* be amended by the deletion of "and" in the first line thereof, and by the addition of the words "and 47" in the same line.

2. That under *Rule 18* the form of nomination of candidates for the Council be amended by transferring the candidate's declaration to the end thereof, and that the words "and by the nominee" be added in the third line after the above-mentioned form after the word "subscriptions."

3. That the proviso at the end of *Rule 22* be deleted.

4. That in *Rule 26* the Nomination Form for candidates for Membership be amended to read as follows:—

"We, the undersigned members of the Society of Chemical Industry, hereby propose  
(*candidate's name in full*) as a fit and proper person to become a member of the Society.

*For names only of members }  
recommending the candidate. }*

"And I, the said candidate for membership, hereby declare that if elected I will do my best to support the Society and will observe its By-Laws.

(Signature) \_\_\_\_\_ (Profession) \_\_\_\_\_

(Address) \_\_\_\_\_ (Date) \_\_\_\_\_

*Extract from By-Laws (Rules 27, 29, and 30).*

5. That *Rule 27* should read as follows:—

"Each member shall pay an annual subscription of twenty-five shillings, due on the 1st January in each year; except that any member may pay a life composition fee of twenty pounds in lieu of an annual subscription. He shall also pay on receipt of notice of election an entrance fee of one guinea in addition to his first year's subscription; except that should a member who has paid an entrance fee resign and subsequently seek re-election the Council may dispense with a further entrance fee."

6. That in the second line of *Rule 28* the words after "and" read as follows:—"issued to the members once every year."

7. That *Rule 29* read as follows:—

"No person shall be entitled to the privileges of membership who has not paid his subscription for the current year. He shall not attend meetings nor vote, nor shall he receive gratuitously the publications of the Society. Should the subscription remain unpaid eight months after it is due, the Secretary shall give the member notice that unless payment be made within four months after the date of such notice, his name will be struck off the register. At the expiry of the said period, if the member should still remain in default, the Council may order his name to be removed from the register, whereupon he shall cease to have any right, privilege, or interest in the Society, and the Council may notify the same in the Journal and recover the arrears of subscription at law."

8. That *Rule 30* read as follows:—

"Any member may withdraw from the Society at the end of any year by giving previous notice to the General Secretary of his intention to withdraw; always provided that such notice to be valid must be accompanied by the payment of all subscriptions, if any, which may be due from him to the Society at the date of the notice. By such notice and payment he shall be released from all further liability as a member."

9. That the following Rule be added after *Rule 53* of the present Rules:—

"All communications between Local Sections and Public Bodies or Societies shall be conducted through or be at the discretion of the Council; and no publication shall be issued by any Section to the general public without the previous consent of the Council."

10. That *Rules 54* and *55* be placed after *Rules 34* and *38* respectively, and that the succeeding Rules be renumbered accordingly."

#### BANKERS' ORDERS.

For the convenience of Members, the Treasurer has arranged with the Bankers of the Society that they shall collect subscriptions from Bankers in town and country; and Members, who have not already done so, are invited to fill up and sign the Banker's Order enclosed with the December number of the Journal, which should then be sent to the Honorary Treasurer, Mr. E. Rider Cook, East London Soap Works, Bow, E.

#### ANNUAL GENERAL MEETING.

The Annual General Meeting will be held in Edinburgh on the 18th, 19th, and 20th July next. Full particulars will appear in a subsequent issue.

#### INTERNATIONAL CONGRESS OF APPLIED CHEMISTRY.

We are requested by the Foreign Office to state that an International Congress, under the patronage of the Belgian Government, will open in Brussels on the 4th August next, in connection with the Antwerp Exhibition, on the subject of applied Chemistry.

The work of the Congress will be divided into four sections corresponding to those of the Belgian Chemists' Association, viz.:—Sugar, Agricultural Chemistry, Food-stuffs, and Biological Chemistry.

Those who desire to attend the Congress should apply to M. Sachs, 68, Rue d'Allemagne, Brussels (Sugar and Agricultural Chemistry), or to M. H. van Laer, 15, Rue de Hollande, Brussels (Adulteration of Food-stuffs, and Biological Chemistry), who will forward full particulars on application.

Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 41 of the bye-laws, the Society has the right of priority of publication for three months of all such papers. Infringement of this bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. EYRE and SPOTTISWOODE, the Society's printers and publishers, to whom all communications respecting them should be addressed.

#### LIST OF MEMBERS ELECTED 21st MARCH 1894.

Bailey, Wm., Graedien, Alloa, N.B., earthenware manufacturer.

Barratt, W. S., 40, Heywood Street, Moss Side, Manchester, science teacher.

Beaman, H. S., 18, Mavilla Gardens, North Kensington, W., brewer and analytical chemist.

Bowman, Jas. H., Canada Chemical Manufacturing Co., London, Ont., Canada, professor of chemistry.

Bryce, Thos., Tharsis Mines, Huelva, Spain, chemist.

Cox, Robt., 34, Drumsheugh Gardens, Edinburgh, gelatine manufacturer.

Darling, Geo. A., c/o Robinson G.M. Co., P.O. Box 1024, Johannesburg, S.A.F., metallurgist.

Fairweather, Wallace, 62, St. Vincent Street, Glasgow, chartered patent agent.

Friedman, Chas. S., 1616, Montgomery Avenue, Philadelphia, Pa., U.S.A., chemist.

Fuerst, Dr. Alex. F., 30, Belsize Road, London, N.W., chemist.

Goodwin, Prof. Wm. L., The School of Mining, Kingston, Canada, professor of chemistry.

Goskirk, Alex., 28, Hillside Street, Edinburgh, electric metallurgist.

Græff, Robt. W., 29, Miacing Lane, London, E.C., chemical agent and merchant.

Heap, Isaac H., Fern Bank, Aynsley Road, Stoke-on-Trent, pharmaceutical chemist.

Henderson, Jos., 13, Denmark Street, Darlington, analyst.

Hopkins, Herb. W., 13, Harrington Gardens, South Kensington, S.W., metallurgist.

Jarvis, Talbot McL., Castle Lodge, Bedford, brewer.

Langrick, Thos. E., 3, Cambridge Avenue, Edinburgh, paint works manager.

Llewellyn, Geo. H., Pencreaig, Caerau Park, Newport, Mon., science student.

McAlley, Robt., 43, King William Street, Greenwich, S.E., paint works manager.

McDonald, John, Distillery Offices, Fort William, N.B., distiller.

McVitie, Robt., 8, Torphichen Street, Edinburgh, biscuit manufacturer.

Moffat, Alex., c/o Linlithgow Oil Company, Limited, Linlithgow, N.B., analytical chemist.

Muspratt, Max., Scaforth Hall, Scaforth, Liverpool, technical chemist.

Oliver, Jas. M., Kirkealdy, N.B., engineer.

Pearson, Wm. H., Bryn Villas, Blaina, Mon., analytical chemist.

Rose, Andrew A., 172, Leith Walk, Edinburgh, merchant.

Rose, Hugh, 172, Leith Walk, Edinburgh, paint and varnish manufacturer.

Shalleross, Arthur, 19, City Road, Openshaw, Manchester, chemical manufacturer.

Shupley, Fred., c/o W. P. Fuller and Co., 21, Front Street, San Francisco, Cal., U.S.A., paint works chemist.

Smith, J. Cruickshank, 32, Great Guildford Street, Southwark, S.E., assistant chemical manager.

Watson, A. Forbes, 11, York Place, Edinburgh, assistant in chemical department, Edinburgh University.

Watson, Chas. S., 9, Princes Street, Pollokshields, Glasgow, analytical chemist.

#### CHANGES OF ADDRESS.

Auerbach, Dr. C. G., 1/o Ludwigshafen; 1. 7.4., Mannheim, Germany.

Barracough, W. H., 1/o Forest Gate; 83, Bedford Road, Ilford, Essex.

Bernard, Jas., 1/o Braço de Prata; Casal das Rollas, Olivaes, Lisbon.

Beveridge, Jas., 1/o Northfleet; Sulphite Pulp Mills, Hjerpen, Sweden.

Bowman, Dr. F. H., 1/o Halifax; Ashleigh, Ashley Heath, Bowdon, Cheshire.

Douglas, Wm., 1/o Demerara; 35, Vartry Road, Stamford Hill, N.

Fanta, F., 1/o Gresham Street; Effingham House, Arundel Street, Strand, W.C.

Frew, Dr. Wm.; Journals to Ottostrasse 13<sup>m</sup>, München, Bavaria.

Fryer, Dr. A. C.; 1/o Cornwallis Lodge; 13, Eaton Crescent, Clifton, Bristol.

Gamble, D., jun., 1/o Prescot; Bryn Eithyn, Colwyn Bay, North Wales.

Gibbins, H. B., 1/o Bath; Inglescombe, Manor Park, Redland, Bristol.

Haller, G., 1/o 86A; Sussex House, 52, Leadenhall Street, E.C.

Harrison, C., 1/o Sheffield; Hayle, Cornwall.

Hartley, Jns., 1/o Manchester; Ballavartyn, Santon, near Douglas, Isle of Man.

Jarmain, G. S., 1/o Marsh; Minerva Works, Turnbridge, Huddersfield.

Jarvis, Talbot; Journals to 51, Pembury Road, Tonbridge, Kent.

Lange, Dr. M., 1/o Amersfoort; Westerdokstraat 25, Amsterdam, Holland.

Lindley, H. L., 1/o Camden Road; Chemical Laboratory, Royal Veterinary College, Camden Town, N.W.

Love, Dr. E. G., 1/o 54th Street; 80, East 55th Street, New York, U.S.A.

Maenair, Dr. D. S., 1/o London; 2, Grosvenor Terrace, Hillhead, Glasgow.

McVitie, Robt.; Journals to Derwent Mills, Malton, Yorks.

Morris, Dr. G. Harris; Journals to 18, Gwendwr Road, West Kensington, W.

Newton, H. C., 1/o Bronesbury; 8, Hldgrave Road, South Hampstead, N.W.

Pearce, Wm., 1/o Brentwood; The Lhus, Salway Hill, Woodford, Essex.

Pettigrew, R., 1/o Andrie; c/o Mersey and Irwell Joint Committee, 41, Mosley Street, Manchester.

Rhodes, Jos., 1/o Glasgow; Old Park Printing Works, Belfast.

Roberts, Ernest H., 1/o Wandsworth; Chemical Works, Wicklow, Ireland.

Seymoure, A., 1/o Box 1283; Box 20, Johannesburg, S.A.R.

Saltmarsh, J. G., 1/o Brooks Bar; 103, Park Street, Greenheys, Manchester.

Sandon, R., 1/o Tufnell Park; 42, Lewisham Road, Dartmouth Park, N.W.

Savin, Chas., 1/o Laurence Pountney Hill; c/o Dollman and Pritchard, 39, King Street, Cheapside, E.C.

Sharp, H., 1/o Brookenhurst; 11, Delahay Street, Westminster, S.W.

Sisson, G., 1/o Manchester; c/o Washington Chemical Co., Lim., Washington Station, R.S.O., co. Durham.

Sowler, Alf., 1/o Beckenham; 208, Bedford Hill, Balham, S.W.

Turner, Thos., 1/o Birmingham; County Offices, Stafford.

Tweddie, G. R., 1/o Margate; Puckstone, near Sturry, Kent.

Wade, W. L.; Journals to 62, Nine Elms Lane, S.W.

White, W. H., 1/o Killingworth House; The Cottage, Killingworth, Newcastle-on-Tyne.

Wilder, F. L.; Journals to St. John Del Rey Mining Co., Morro Velho, c/o P. S. Nicolson and Co., Rio de Janeiro, Brazil.

Wingham, A., 1/o London; 3, Lincombe Views, Warren Road, Torquay.

Wood, Jos. T., 1/o Hound Road; 29, Masters Road, West Bridgeford, Nottingham.

Wright, L. T., 1/o Buenos Ayres; 10, Hazlitt Road, Sinclair Road, London, W.

#### RESIGNATIONS CANCELLED.

Davis, H. W., Government Laboratory, Somerset House, W.C.

Schlichter, Dr. H., Thothmes House, Gisburn Road, Hornsey, N.

#### CHANGE OF ADDRESS REQUIRED.

Houmfray, D.; 1/o 42, South Street, Greenwich, S.E.

#### ERRATA IN LIST.

Jürgensen, Dr. Rolof (wrongly spelt "Türgensen"). Prague, Austria.

Stone, Frank (Christian name wrongly given as "William"), Melbourne, Victoria.

#### Drath.

Bicket, J. Hugh; 1/o The Limes, Stamford Hill, N.

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### SESSION 1893-94.

#### REMAINING MEETINGS.

First Monday in each Month at 8 p.m.

- April 2nd:—  
Mr. J. W. Lovibond. "Colour as a means of Quantitative Estimation."  
Mr. W. C. Young, F.I.C., F.C.S. "The Natural Diminution of the Dissolved Organic Matter in the Water of Rivers."  
Mr. H. Louis, A.R.S.M., F.I.C., F.C.S. "Note on an Improved Specific Gravity Bottle."
- April 16th:—  
Mr. H. de Moersenthal, F.I.C., F.C.S. "On the Treatment of Gold Ore at the Witwatersrand Gold Fields."
- May 7th:—  
Mr. Claude Vautin. "The Commercial Analysis of Fused Salts."  
Messrs. W. H. Stanger, M.I.C.E., F.C.S., and B. Blount, F.I.C., F.C.S. "The Testing of Hydraulic Cements."  
Mr. W. Gowland, A.R.S.M., F.I.C., F.C.S. "A By-product obtained in Liqutating Copper with Lead."
- May 21st:—  
Mr. F. Platten. "A Modification of the Ferric Chloride Distillation Process for Estimation of Arsenic in Copper."  
Mr. Oscar Guttman, M.I.C.E. "The Manufacture of Smokeless Powder."
- June 11th:—  
Mr. W. G. McMillan:  
(a.) "Experiments on the Strength of Leather."  
(b.) "Note on the Colour of Brass."  
Mr. P. Dvorkevitch. "Distillation of Peat."

Notices of the Meetings appear regularly in the Scientific Press.

Meeting held Monday, February 5th, 1894.

#### NOTE ON AN INTERESTING EXPLOSION CAUSED BY SODIUM PEROXIDE.

BY A. DUPRÉ, PH.D., F.R.S.,

Chemical Adviser to the Explosives Department of the Home Office.

On the evening of March the 9th, 1893, an explosion happened in the goods station of the Midland Railway in Whitecross Street, and on the following morning the scene was visited by Colonel Majendie, C.B., and myself. We found numerous signs that an explosion of some violence had taken place. The box which had contained the material causing the explosion had disappeared, but fragments of it were scattered about the station, some striking the roof and others the skylights, breaking some of the panes of glass. Eight tins which, it was said, were in the box, had either disappeared entirely or were scattered in fragments all over the station. Near to the scene of the explosion, the platform, the pillars supporting the roof, other ironwork, and the roof itself, were splashed over with a white substance, evidently projected by the explosion. The van on which the box had been, and the platform

against which the van stood, had suffered a little from fire, but no structural damage had been done to any part of the building.

On investigation it was found that the box which exploded had contained sodium peroxide, packed in 14-lb. tins, eight of which were said to have been in the box. Besides this box there were on the van a larger box also containing peroxide (this on subsequent examination was found to contain 19 14-lb. tins of peroxide and one empty tin), five casks of a concentrated solution of sodium bisulphite, two casks of sanitas, a box of colour, and a parcel of drapery. The van, which arrived at the station at 8 p.m., had been backed against the platform, and everything, except three casks of bisulphite solution and the box which subsequently exploded, had been removed from the van by 9.15 p.m. At 9.50 two porters, Hill and Rose, returned to the van to complete the unloading. Hill first proceeded to remove the box, which, since it was first put into the van, had rested undisturbed on the top of two of the casks of bisulphite. He had lifted the box down on to his knees, and was either letting it down to the floor of the van, or was turning it over to place on the platform, when he heard a hissing noise coming from the box, and Rose, on turning round, saw flames issuing from it. Rose at once ran away towards the gates of the station, and while so engaged (about four or five seconds), having fallen off the platform in his hurry, four loud explosions followed each other in rapid succession, filling the station with a black smoke and setting fire to the tarpaulin of the van. Hill, who had remained on the van till after the first explosion, jumped down and also ran away; he was not seriously injured. The police and firemen from stations near, having been alarmed by the noise of the explosions, speedily arrived and put out the fire.

Inquiry showed, beyond all reasonable doubt, that the box which exploded had contained peroxide of sodium packed in square 14-lb. tins. The next step, therefore, was to ascertain how this material could have caused the explosion. For this purpose numerous experiments were made by Mr. Archbutt and myself, which fully satisfied us that sodium peroxide by itself could not be exploded either by compression, friction, or heat, even that of an oxyhydrogen blowpipe. Even the explosion of a 30-grain detonator inserted into the middle of 1 lb. of peroxide packed in a double, strong tin canister, failed to induce any decomposition. These experimental results are corroborated by theoretical consideration. The formation of sodium peroxide is not an endothermic reaction, and hence the peroxide is not one of those compounds liable to spontaneous decomposition. I have been unable to find any record of the heat value of this reaction, and some experiments which I hoped to lay before you have not yielded numerical results sufficiently accurate to warrant their publication. Nevertheless the experiments clearly show that a considerable amount of heat is evolved during the conversion of  $\text{Na}_2\text{O}$  into  $\text{Na}_2\text{O}_2$ .

When water is added to sodium peroxide much heat is liberated, some peroxide of hydrogen is formed, and oxygen and steam are evolved. The decomposition, however, has no tendency to spread, but is always strictly confined to the portion wetted. Numerous experiments were made on this point, and always with the like result.

When, however, the peroxide is mixed with, or is even in contact only with, any combustible, the result is very different. In this case the combustible matter in contact with the peroxide readily takes fire when the latter is moistened, and if anything like an intimate mixture of the two has taken place a more or less violent explosion is the result. The mixtures of the peroxide with wood meal, wood shavings, hay, cotton, wool, sulphur, and even bisulphite of sodium, readily take fire or explode when simply moistened with water. Experiments illustrating the facts were shown. A slightly rough deal board can be set on fire by merely covering it with a layer of peroxide and dropping water on it. When once the combustible has taken fire a very fierce combustion results as long as any peroxide is left, the effect being greatly enhanced by the melting of the peroxide, which wherever it flows sets fire to everything combustible. Sodium peroxide, as is of course well known, is a very powerful oxidising agent, but it differs from most

other agents of the kind by the fact that its power can be called forth both by fire and by water, and it is this latter fact chiefly which renders it so dangerous. In this behaviour we have, in my opinion, an explanation of the explosion under consideration. A qualitative analysis of various portions of the white substance splashed about, as before stated, in the neighbourhood of the explosion, revealed the presence of much sulphite and sulphate of sodium, besides the presence of caustic soda, carbonate of sodium, ferrate of sodium, globules of melted iron and tin, and in some cases of a sulphide. A quantitative analysis of some of the material projected against the roof, made by Mr. Archbutt, gave the following result:—

Sodium carbonate.....	9.55
Sodium sulphate.....	6.41
Sodium sulphite.....	52.98
Black insoluble matter, much organic matter with oxide of iron and particles of metallic iron or tin.....	31.06
	<hr/> 100.00

This material had undoubtedly come from the exploded box (the casks of bisulphite solution were found full and uninjured after the explosion), and gives, I think, the key to the solution of the difficulty. There can, I believe, be little or no doubt that besides the peroxide the box contained some sulphur compound, perhaps in the form of a strong solution. By the tilting of the box this solution came into contact with some of the peroxide which during transport had escaped from one or more of the by no means very strong tins and thus led to the accident. I am fully aware that many objections may be raised against this explanation, and shall be very glad to hear any remarks from members of this Society, many of whom must be familiar with this material. The lesson to be learnt from this explosion, whether the attempted explanation be accepted or not, is, however, clear. Peroxide of sodium should always be packed in strong metallic cases not liable to injury during ordinary transport, and should be carefully kept apart from all kinds of combustible matter.

I cannot conclude without expressing my thanks to Mr. Archbutt for the valuable help he has afforded me in the investigation of the cause of this explosion.

#### DISCUSSION.

Mr. TYLER, whilst paying a tribute to the careful work of Dr. Dupré, said that to his mind no satisfactory cause had been given. He had had considerable experience with sodium peroxide, from its earliest production, when it was much less pure, to the present article supplied by the Aluminium Company, which was very pure. One point had been overlooked, and that was the presence of small pieces of metallic sodium, which occasionally occurred in less pure specimens. There had been no evidence of leakage from the tins or from any other package into the tins, so he could not consider the explanation satisfactory. He had also tried all the experiments which Dr. Dupré had made that evening, and although plenty of heat and sometimes light were produced by the mixture of sodium peroxide with other substances and water, he never had had a violent explosion. He would add that his experiments were made, not on small, but on considerable quantities of materials, thus resembling the conditions given. It was only fair to the important industry connected with peroxide production to emphatically state that sodium peroxide *per se* was absolutely harmless, and, as far as his experience went, a most difficult compound with which to produce a violent explosion.

Mr. D. HOWARD said it was a great many years since gunpowder was invented, and it was discovered that three harmless substances properly mixed up had very unpleasant properties. Unfortunately the lesson which was thus taught was not sufficiently appreciated, viz., that perfectly harmless things if mixed together had very unpleasant results. He wished that those who had charge of the carriage of goods would learn the lesson. The popular idea was to put all

dangerous things together. In his boyhood there was a very ugly fire at one of the docks, said to have arisen from the impression that jute, cubic nitre, and sulphur were dangerous things, and, therefore, should be stored in the same warehouse. The story went that the jute and sulphur were on the lower floor, and the nitre on the top, and the heat causing the nitre to melt they could imagine what happened. This might not have been an absolutely true account, but it illustrated the sort of feeling people had that all dangerous things should be put together, then which there could not be a more dangerous delusion. He feared there was an idea that peroxide of sodium was one of these horribly dangerous things which were to be put with other dangerous articles, and, therefore, it seemed to him there was great value in Dr. Dupré's careful experiments. Peroxide of sodium was apparently absolutely incapable of explosion unless mixed with something else with the very something else with which it was likely to be brought in contact on the principle he had enunciated. It was very clear that what was required was an absolutely careful package. He did not know what the package might have been in this case, but he had known nitric acid of 1.500 specific gravity packed in sawdust, and the result was not unlike Dr. Dupré's description, but happily it took place in an open yard. This was sent to him to be shipped under deck. Peroxide of sodium should be packed with mineral meal, or something of that kind perfectly incombustible, and under those circumstances it would be perfectly safe. On the other hand, if it were badly packed to begin with, and then put in dangerous company, no doubt it might become a source of serious danger. The idea that because a thing could explode it was to be condemned was a very serious evil. There were very few things which would not explode. It had been recently discovered that methylated spirit on being shaken up and held to a candle gave a considerable rush of flame, but he had known that about as long as methylated spirit had been made. In fact there was hardly anything which could be burnt which would not make an explosion if you went the right way about it. But, on the other hand, there was hardly anything, even of a decidedly dangerous character, which might not be safely dealt with and transported if proper precautions were taken.

Dr. SQUIRE said that if a tin in which there was a crack were plunged into a solution of bisulphite of sodium, that crack would develop into a hole, and then all the rest might follow.

The CHAIRMAN said that Mr. Howard had given very good advice as to the danger of relegating all so-called dangerous substances to the same class, but he feared that advice would not reach the right people, but that the railway companies would continue to put all kinds of things down in the fancy category of dangerous explosives. Their difficulties on that subject were about on a par with those of fire insurance officials, who seemed often to classify the most dangerous parts of a place at the lowest rates and those which it was almost physically impossible to set on fire at the highest.

Dr. DUPRÉ, in reply, said that peroxide of sodium when kept by itself was perfectly harmless, but what he wanted to be more fully recognised than apparently it had been, was that when it was brought into contact with combustible substances it became exceedingly dangerous in the presence of water as well as of fire. He had often had occasion to draw public attention to the fact that when a danger was recognised it could be avoided, but if not known it led to disaster. The main point was to realise that under certain conditions there was danger, and then it could be easily avoided. He was always glad to accept any suggestion, and if he had given an explanation he was always ready to give it up if anybody else could suggest a better one. But throughout Mr. Tyler's remarks he did not recognise any suggestion as to how this explosion could have happened. If it was well packed in good boxes, and there was nothing else in it, how did the explosion occur? There was no doubt the explosion did take place. He was perfectly alive to the fact that sodium peroxide might contain metallic sodium, and no doubt a little was present in this

case, but the amount was so small that he attached no importance to it. But even if sodium were in it, how could it have gone off without something else being added? Of course it might if water got into it, but that landed one in the same difficulty. It was that which Mr. Tyrer had to explain. When dynamite was first introduced it was said it could only be exploded by a detonator, and many hundreds of lives were lost by trusting to that statement. People treated it as being non-explosive, and fatal accidents followed as a matter of course. In the same way, people were constantly coming to him with explosives which would not go off anyhow except just when they were wanted to; such statements were the cause of many accidents. He trusted that nothing he had said would be taken as a disparagement of peroxide of sodium or as casting a slur on the company who made it. That was far from his object. Peroxide of sodium was a useful substance, and he trusted it would be largely used, but he hoped the company would take warning and see that it was sent out in stronger tins and packed in such a manner that it was not possible for it to come in contact with combustible matters. If that was done it could be transported or stored for any length of time without any danger whatever.

*Meeting held Monday, March 5th, 1894.*

MR. WM. THORP IN THE CHAIR.

## THE COMMERCIAL PRODUCTION OF CHLORINE BY THE AMMONIA-SODA PROCESS.

BY F. HALE.

THE subject on which I wish to address you to-night is one of deep importance to the salt districts of this country, to our chemical trade, and therefore affects our commercial prosperity.

The competition between the old Leblanc process and the ammonia-soda processes for the production of soda and bleach which for years has been going on, has ended, I believe, in the complete success and victory of the latter over the former.

For some years past the old process was able to hold its own in having the monopoly of the bleaching trade, the ammonia-soda process being unable to make strong bleach.

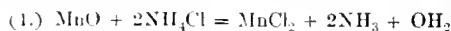
All this is now altered, and pure and strong hydrochloric acid and chlorine can be produced by the new process much more readily and cheaply than by the old process of Leblanc.

The direct recovery of the hydrochloric acid from ammonium chloride without the use of lime has been the problem which chemists interested in the ammonia-soda process have had to grapple with. So long as lime was necessary for regenerating its ammonia, the waste of chlorine as calcium chloride occurred, and there seemed no likelihood from the great stability of calcium chloride of ever obtaining hydrochloric acid or chlorine as a practical and paying process on a manufacturing scale.

Mr. Mond has from time to time attempted the volatilisation of dry ammonium chloride, thereby forming nickel oxychloride and free ammonia as a basis for an industrial method of answering this problem; and in his later patents he uses magnesia made into marbles or pills with china-clay and an alkaline chloride, and piled in a Deacon furnace. The ammonium chloride is volatilised in fused chloride of zinc, and passed in the state of vapour through the marbles thus made. The ammonia is evolved and completely driven out of the retort by a neutral gas, which is also used for raising or lowering the temperature of the retort. Hydrochloric acid is given off at first from the magnesium chloride formed, and afterwards chlorine is obtained by hot

and dry air, to the extent of from 7—10 per cent., which he passes into a Deacon bleach chamber. The recovery of chlorine, however, in conjunction with this process, for the manufacture of strong bleach is still, I believe, a problem not yet solved.

The Verein für Chemische Industrie, at Mainz, have used manganese oxide with this object in view, in the hope that the two equations—

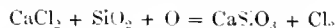


might work profitably on a large scale; but although the process has been protected, I have not heard that it has come up to the expectations of the patentees. The probable reasons for this will be explained below.

Other inventors have regarded the problem as hopeless. They have turned their attention to the recovery of the chlorine from the waste calcium chloride liquors, regarding the decomposition of ammonium chloride by means of lime as the only suitable reaction for the regeneration of the ammonia without loss.

Solvay has been most fruitful in patents in this direction, and the owners of these patents still hope, I hear, that the joint action of silica and alumina on calcium chloride at a high temperature in the presence of air will result in a commercial process for the recovery of the wasted chlorine.

The action is one which is well known, and was thought to be possible with the chlorides of the alkalis and alkaline earths by Talaude and Prudhomme many years ago. In its simplest form the change may be expressed thus:—



The substitution of magnesia for lime in decomposing the ammonium chloride liquors has also been thought a possible solution of the difficulty.

This process, which has been at work at Salindies since 1886, is now under the control of Messrs. Pechiney and Cie., and since magnesia has a tendency to form an oxychloride, which can be converted into oxide and chlorine when heated in air, the problem has been one which involved mechanical and engineering difficulties more than any novelty in the chemical reactions involved.

And lastly, Mr. Bell, of Middlesbrough, working on the lines of Schloesing, obtains his ammonia from solutions of ammonium chloride and magnesia, converts the resulting chloride of magnesium into grains by evaporation, and afterwards into anhydrous chloride of magnesium. From the first he gets hydrochloric acid gas by the downward percolation of steam, and from the second, chlorine by the downward percolation of air through the heated bed of magnesium chloride.

In the attempts which have thus been made to produce chlorine from ammonium chloride in the ammonia-soda process, none of them have been successful in producing strong and undiluted chlorine, fit for the manufacture of strong bleach, and this for the reason that air has been employed to obtain chlorine direct from the chloride formed and the air containing about 80 per cent. by volume of nitrogen.

These and other facts induced me to turn my attention to obtaining chlorine from the hydrochloric acid gas obtained from magnesium chloride through ammonium chloride. I have the honour of placing before you to-night the principles and practice by which this is accomplished, and I beg to invite your attention to the same, and your discussion thereon.

If ammonium chloride in powder is mixed with any of the monoxides in powder and gently heated, the ammonia is evolved and the chloride of the metal formed, water being at the same time produced.

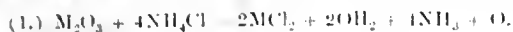


The monoxide preferred is magnesium oxide, for the readiness with which it decomposes ammonium chloride, liberating ammonia, and for the ease and low temperature with which its chloride parts with hydrochloric acid when heated, and on the passage of steam through the same, as

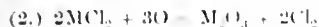
mentioned hereafter. The higher oxides cannot be used for this purpose, as they form sesquioxides on the passage of air over their chlorides.



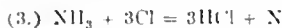
The sesquioxides thus formed cannot be used for the evolution of ammonia from the ammonium chloride without great loss of the ammonia.



The oxygen thus liberated reacts on the chloride formed, liberating chlorine, thus:—



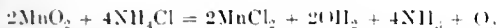
The passage of the metal from the diad state, as chloride, to the tetrad condition, as oxide, accounts for this loss, and the liberated chlorine destroys a portion of the ammonia evolved—



and this to the extent of from 30 to 33 per cent. Their use, therefore, for liberation of the ammonia from ammonium chloride is quite out of the question.

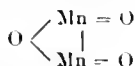
In my earlier experiments this was a source of continual trouble, and one was inclined to believe that the evolution of the ammonia was governed by no law, until it was found that the sesquioxide was the sinner whose presence was the chief cause of the mischief, and was confirmed by trying nearly every other sesquioxide, with a similar result—a loss of from 30 to 33 per cent. ammonia.

The dioxide, as ( $\text{MnO}_2$ ), does not destroy the liberated ammonia.



We thus obtain another means of distinguishing the sesquioxide from the dioxide.

As the lower oxide ( $\text{MnO}$ ), and the higher ( $\text{Mn}_2\text{O}_3$ ), does not destroy the liberated ammonia, we cannot regard, therefore, the sesquioxide ( $\text{Mn}_2\text{O}_3$ ) as a mixture of the lower and higher oxides, and this theory, I think, must be given up, as it appears to be a true compound, thus:—



These higher oxides whose chlorides form sesquioxides on the passage of air or steam over them, and whose use is so disastrous in the evolution of the ammonia, become all-important in the evolution of chlorine from hydrochloric acid gas.

The above facts have compelled us to confine the evolution of the ammonia to the action of the monoxides, while the use of the peroxides or sesquioxides is confined to the evolution of strong chlorine from gaseous hydrochloric acid.

These facts enable us to obtain the ammonia without loss beyond unavoidable working losses, and the chlorine undiluted for the manufacture of strong bleach.

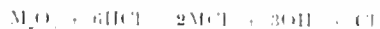
To accomplish this, it was necessary to obtain the hydrochloric acid pure, and in a fairly concentrated state. The best chloride for this purpose we found was chloride of magnesium,  $\text{MgCl}_2$ , whose readiness to evolve hydrochloric acid by heat, and superheated steam, is well known, and whose oxide gives good results for the ammonia.



The hydrochloric acid thus produced is obtained as a pure and strong continuous current, which might be used in the following manner:—

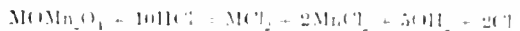
- (1.) It may be condensed in water, in the usual manner, producing the pure commercial acid.
- (2.) It may be condensed in a solution of calcic chloride, according to Wise's process, and afterwards obtained in a dry gaseous condition by heat: or, which is much preferred.
- (3.) It may be at once dried in strong sulphuric acid, descending in a tower or column, similar to the Glover tower, filled with pieces of coke, flint, &c., the gas entering the bottom, passing out dry at the top of the column.

The dry and gaseous hydrochloric acid produced by either of the above processes is heated up in a Cowper stove to about 550° C. It is then passed into a Deacon furnace containing peroxides made into balls or bricks by well known means, or used in thin layers in powder, when one third of the available chlorine is obtained strong, and practically free from hydrochloric acid.



The chlorine thus produced is freed from moisture by well-known means, and used for the production of strong bleach or mixed with dilute chlorine afterwards obtained from the chlorides formed.

Mn in the above equation might be manganese, cobalt, iron, bismuth, tin, lead, &c. Manganese oxide or mixtures of  $\text{Mn}_2\text{O}_3$ , Co, Co $_2\text{O}_3$ , Pb $_2\text{O}_3$ , give good results; the use of the first is preferred, mixed with some of the others, as the sesquioxide of manganese forms with them, manganates, and thus gives a larger yield of strong chlorine, thus:—



the remainder of the chlorine fixed in the above equation as chlorides is obtained by passing dry and heated air through the retorts, when dilute chlorine is given off, containing from 15—20 per cent. of chlorine mixed with air, and, if the drying of the marbles and the air has been carefully done, it will be free from hydrochloric acid, and be passed into the common chlorine conduit, and mixed with the strong chlorine obtained at first, and the round of operations recommenced, when this alternative process is used.

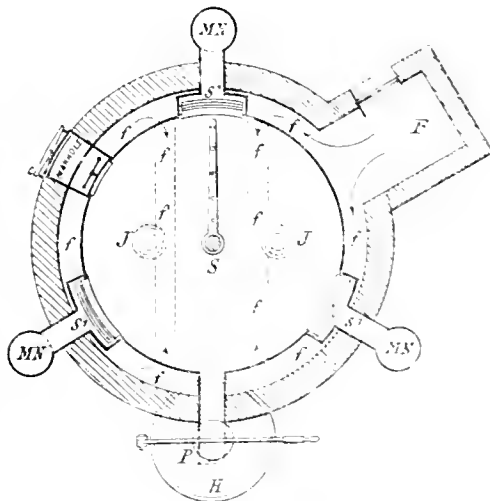
As a continuous process for the production of chlorine, I may carry on the above two reactions simultaneously, by passing a mixture of air and hydrochloric acid gas in proper proportions through the marbles in the cylinders. The gaseous mixture should first be dried in strong sulphuric acid and then passed through a Cowper stove, and heated to a temperature of about 500° C. to 550° C.; and if the drying has been carefully done, the gases on leaving the bottoms of the cylinders are, for practical purposes, free from hydrochloric acid, and after cooling and drying may be passed into a bleach chamber or otherwise used.

There are two methods of working throughout for the evolution of the ammonia and hydrochloric acid.

1. We may work the substances in powders throughout, both for the evolution of the ammonia and for the hydrochloric acid gas afterwards.

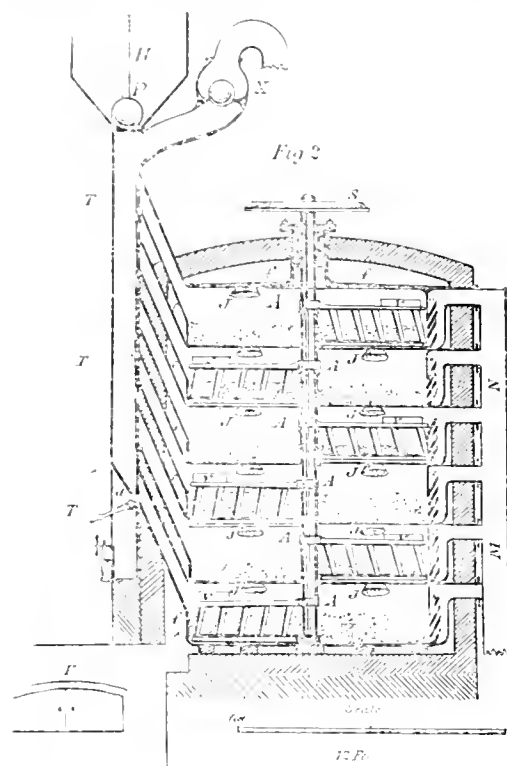
In this case, thin layers of the substances several inches deep in chambers are used, which are worked in series one above the other; Fig. 2 is a vertical section of such a chamber. The cylinder A is made of iron, and lined with

Fig. 1.





earthenware tiles or bricks. It is placed in a brick chamber which is heated by the furnace F, by flues *f f*, through which the gases from F circulate round the cylinder. By means of the stirrer S the contents can be mixed when



required, and from the hopper H the ammonium chloride is introduced at the plug P. The jets J admit superheated steam on to the mixture at the bottom of the retort.

The method of working is as follows:—

The ammonium chloride in powder is admitted from the hopper H, and passes down the tower T, till it meets the first open retort, into which it slides upon the opened door *d*, and is then caught and mixed with the oxide by the vane V, on the arm of the stirrer S, which is also kept in motion, to bring the mixture into contact with the hot sides of the retort, and to facilitate the passage of the gases formed (ammonia and hydrochloric acid) at the grated exit *s¹ s¹*, at the bottom of each of the retorts.

When the ammonia is evolved, superheated steam is admitted through the jets J J on to the mixture, and the stirring being continued, passages are thus opened for steam to penetrate into the interior of the mass, and for the hydrochloric acid gas evolved to pass away at the exit at the bottom, a current of pure and concentrated hydrochloric acid is evolved, which is dried and passed over the peroxides as described below, and the round of operations recommenced.

2. The second method is to work the mixtures in powder, during the evolution of the ammonia as above described, and then to agglomerate the resulting mixture of oxide and chloride. This is done by admitting through the jets J a spray of hot water, or one of a solution of ammonium chloride, on to the mixture, and by the stirrer moulding it into fragments or lumps, sufficient for the ready passage through it of superheated steam.

About two-thirds of the ammonium chloride is added at first as a powder, and the remainder in a saturated solution to agglomerate the mixture; most of the water used being evolved as hydrochloric acid afterwards.

The process is as follows:—

Starting at a temperature of 450° C. or 500° C., at which all the hydrochloric acid is evolved, cold air is passed

through the fragments, till they are reduced to about 200° C. The ammonium chloride in the form of powder is then passed into the retorts as above stated, through the hopper H. The mass is then well stirred with the stirrer S, the fragments and lumps of oxide readily reduced to powder, and mixed with the ammonium chloride, and the stirring is continued for the reason above stated. When the ammonia is nearly all given off, the temperature will have reached 200° C. A spray of a hot saturated solution of ammonium chloride is now allowed to enter the retorts from the jets J, and a continuous mixing causes the mixture to pass from the pasty condition to the state of grains or fragments, and the remainder of the ammonia is given off, and the retort cleared of the ammonia by a current of cold air. When this is completed, and the mass made permeable to the steam or air, the stirring is stopped. The heated air above mentioned is now passed through the fragments, and hydrochloric acid, steam, and air, are given off, which might be dried, and used for the continuous production of chlorine, as mentioned hereafter.

Superheated steam may afterwards be sent through the retort, to complete the reduction of the chloride, or a mixture of superheated steam and air.

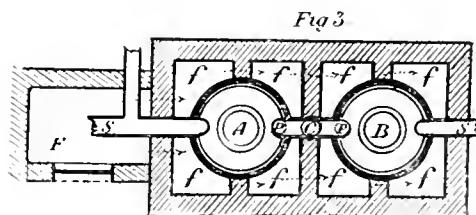
When the hydrochloric acid is completely given off, the temperature is about 500° C., and the round of operations may be recommenced.

When the solution of ammonium chloride is added, the ammonia passes away by a pipe *t*, at the top of the tower T, through the safety valve *v*, which is raised also for the gases produced in the chambers to pass this way into the ammonia and hydrochloric acid conduit M N, when from too rapid an evolution in the chambers, or from stoppage, they cannot readily pass through the bottom exits *s¹ s¹*. When the mixture has assumed the form of fragments, the top exit is closed, the bottom being opened.

The hot gaseous hydrochloric acid evolved as above described, is dried in strong sulphuric acid in a Glover tower, heated up in a Cowper stove, and passed over a peroxide made into balls, or bricks, as in the Deacon process.

By carefully regulating the rate at which the gaseous hydrochloric acid passes over the peroxide, pure and undiluted chlorine is obtained to the extent of one-third of that contained in the hydrochloric acid, while the remaining two-thirds of the chlorine exists combined with the metal of the peroxide, as a metallic chloride, from which dilute chlorine and a regenerated oxide can be obtained by treatment with hot dry air or oxygen, or from which hydrochloric acid gas can be obtained by superheated steam, from which again chlorine can be obtained as before.

Figures 3 and 4 are respectively a plan and vertical section of the apparatus used. A and B are cylinders of



iron, enamelled or lined with tiles to resist the acid, and placed in a heated chamber of brick.

Each cylinder has a outlet pipe, or false bottom, D, for chlorine to pass away to the common conduit *c c*, in which the weak and the strong chlorine becomes mixed. P and P¹ are valves worked by a screw or lever. There are three inlet pipes to each cylinder, one each for the hydrochloric acid, air, and steam.

The peroxides in balls are placed inside the two cylinders A and B, or in a series of such cylinders, which are heated by the flues *f f*, circulating the gases from the furnace F.

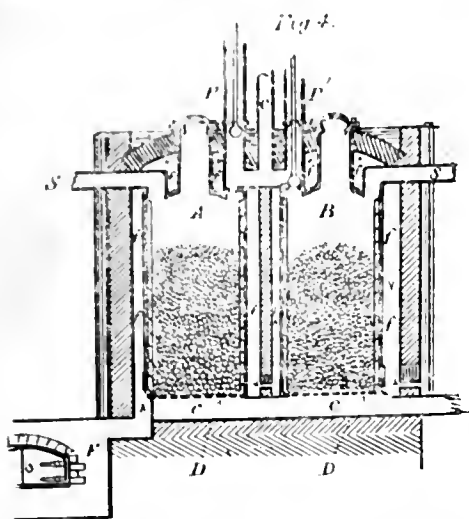


Fig. 1.

In commencing work the valve P is opened and P' shut. Hot, dry hydrochloric acid is then admitted through C. A current of strong chlorine is evolved from the cylinder A, which passes through c, to be utilised as required.

When the absorption power of the balls in cylinder A, or in a series of such cylinders, is spent, the hydrochloric acid current is diverted into another cylinder, or series of such cylinders, and the valve P' is opened.

A current of superheated steam is now passed through S into A (or its series), and the hydrochloric acid evolved, dried, and passed through a Cowper stove to be heated up again, when it is passed into B (or its series) when about one-third of its chlorine is given off strong and pure.

To convert its monoxide into peroxide a current of hot, dry air is now passed into cylinder A (or its series), and superheated steam is sent through cylinder B (or its series) by means of steam pipe S', to free the fixed hydrochloric acid, which is dried, heated up as before to about 550° C., and passing into A (or its series), it gives off strong chlorine as before. The remaining chlorine in A as chloride may be got by air through the plug P, or neglected, as about four-fifths of the original chlorine in the hydrochloric acid used have been got strong and pure, and the series of operations may be recommenced by passing hot, dry air through A, to completely convert the monoxide into peroxide, and then sending through the cylinder (or its series) hot and dry hydrochloric acid as at first.

If the continuous process of producing chlorine is followed, a mixture of hot and dry air and hydrochloric acid gas in proper proportions, is passed through C from the ammonia retorts described above, or from elsewhere into both cylinders (or series of such), both the valves P and P' being opened, and a continuous current of dilute chlorine is obtained from the bottom of the cylinders, containing about 40 per cent. or more chlorine; and the necessary precautions of drying the gases being observed, it may at once be used after cooling for the production of strong bleach.

The alternative process of using hot and dry air where superheated steam is used above may be followed with more or less advantage.

The apparatus above described, in which the mixtures in thin layers are used in powders throughout for the ammonia and hydrochloric acid, may be used with great advantage for the evolution of chlorine from hydrochloric acid gas, by using the oxide in powders of small depth but great surface, as in the apparatus in Fig. 2, of which Fig 1 is a sectional plan.

## NOTES ON LITHOGRAPHIC VARNISH.

BY F. H. LEEDS.

Lithographic varnish is the principal ingredient of the vehicle used to suspend the various pigments employed in the manufacture of painters' inks, but while its name might give rise to the idea that it is only used in lithography, this is by no means the case, as it occupies a similar position in the preparation of the better classes of ink made for printing from letterpress, and the modern photographic process block. It is a product obtained by the treatment of linseed oil alone, differing from painters' boiled oil by its containing no driers. It forms a perfectly clear, transparent substance, only slightly darker, when of the best quality, than the raw oil, but often having a faintly reddish tinge, and possessing always, when prepared by fire heat, a more or less strongly marked green fluorescence. From a curious prejudice painters generally prefer a very dark oil for their use, apparently believing that it is more likely to be genuine, or that it will work better, but printers and printing-ink makers are wiser, and, whether making lithographic varnish or other products containing driers, strive hard to keep their oils as pale as possible, which is obviously of importance, especially when light and transparent pigments are to be ground into them.

Lithographic varnish is met with in five and sometimes six different degrees of consistency, from an oil that is only slightly more viscid than raw linseed, to one which will yield between the fingers at ordinary temperatures a "string" of two or more feet in length. Such a product as the latter is known as "extra strong" lithographic varnish; then in order of decreasing "strength" come "strong," "middle," "thin," "tint," and occasionally "thin-tint." These varnishes—except in the case of the "burnt oil" described below—are valuable simply for their viscosity or "body," which enables the stiffer varieties to suspend pigments of very high specific gravity, since the drying power of the thin or tint is not appreciably greater than that of raw linseed oil, while as the boiling progresses this becomes much weaker, until the extra-strong varnish can hardly be said to dry at all.

The ordinary process of manufacture is simple, although it requires the exercise of some care to prevent a dangerous fire: the oil is heated in iron pans or boilers, which for the better qualities of varnish are enamelled inside, set over a small fire so arranged as to be capable of instant withdrawal. The pan is about two-thirds filled with raw oil, and the heat raised cautiously at first till the water is all driven off. If much effervescence take place and scum rise to the surface, the oil is too new to yield a good product, and had best be put aside for a time to settle; but a fair varnish can be obtained by carefully skimming off the froth (the so-called albuminous matter) as it rises. When all has been removed, the temperature is increased to from 500° to 550° F., and the oil allowed to boil quietly until the proper strength is reached, which is usually determined by taking a few drops out on a palette knife, dropping them on to a piece of cold metal, and testing the tackiness with the finger. This proving satisfactory, the fire is withdrawn, and the varnish allowed to rest for an hour or mere to give time for the "foots" to settle, when it is run or poured off into the storage vessels. During the process of boiling a considerable quantity of very irritating fumes are given off, so that the pot should be provided with a hood to carry them away, while in populous neighbourhoods it will probably become necessary to lead them through a small fire into the shaft of the works. The time required to reach any particular strength is not a fixed quantity, decreasing considerably as the oil gets older, beside varying inversely with the temperature employed. However, seeing that by increasing the heat, the colour of the finished varnish is darkened, it is often advisable to work more slowly at a lower temperature. The loss of weight on boiling also varies a good deal, decreasing, as would be expected, with the age of the oil, the amount being about 3 to 10 per cent.

For certain purposes, a fairly quick drying varnish, which when in bulk will skin over strongly in 24 to 48 hours at ordinary temperatures, is obtained by heating the raw oil

up to the flashing point, and allowing it to burn quietly with constant stirring until the desired strength is reached—the product being known as “burnt oil.” Linseed varnish is also prepared by steam heat, coupled with an air blast and mechanical agitation, but details of this method of manufacture cannot be given.

Another method consists essentially in treating linseed oil with oxygen for some hours at a moderate heat. In this process, the oil gains instead of losing in weight, two samples made from an inferior East Indian oil (sp. gr. 0.929) showing an increase of 3 and 5 per cent. in weight, and possessing gravities of 0.996 and 1.002 respectively. Unfortunately, these samples, which were prepared in the early days of the process, were diminished materially in value by their persistent and unpleasant odour, which would have prevented their use for any but the commonest kinds of out-of-door work. Recently the process has been improved, and Messrs. Wood and Bedford, of Leeds, the present licensees, have kindly treated some of the same oil from which the ordinary varnishes were made, preparing two qualities, one of the same strength as the middle, and the other lying between the strong and the extra-strong lithographic varnishes. They form very pale oils, no darker than the original, not quite so clear as the others, free from the fluorescence mentioned above, but still possessing a stronger and more disagreeable smell than the products obtained by fire heat alone.

From the foregoing it will be seen that age is one of the most important characteristics of linseed oil for varnish makers: in fact the difference in yield, colour, and particularly in fuel consumption, between a varnish made from a “mature” or “well-tanked” oil when first received from the crushers, and from the same oil after it has again been tanked only for a month or so, is very noticeable. Regarding the valuation of oils specially for boiling purposes—assuming their genuineness—not much information has hitherto been found obtainable by laboratory tests alone; generally speaking, the higher the specific gravity the better the oil, but samples should always be boiled in porcelain with the aid of a nitrogen thermometer, at a temperature of about 250° to 300° C., and the colour and percentage loss of the varnish noted, as with care the results approximate very closely to those obtained on the large scale.

The constants in Table I. have been obtained from varnish prepared by fire heat: the tint, thin, burnt thin, and middle being made from the Calcutta oil whose figures are given for comparison, the strong and extra strong coming from another East Indian oil, the constants of which were not noted. The specific gravities were taken on amounts of 15.0–200 grms., but are at the temperature of 15° C. The iodine absorptions are the figures obtained at six hours, the free acid is calculated, as usual, as oleic, and the “Fahrian-acids” are those insoluble in petroleum ether obtained on carrying out the process described by him, *Zeits. f. angew. Chem.* 1891, 540; this *Journal* 1891, 1015.

In Table II. are given the figures of the acids from these varnishes after removal of the unsaponifiable matter. Appliances were not at hand to prepare them in an atmosphere free from oxygen, but to eliminate as far as possible this source of error, they were all dried in flasks under uniform conditions as to temperature and time, and they were examined within 48 hours of extraction. That they were partially oxidised, however, is shown by the fact that in all cases on weighing they were found to be 94–95 per cent. of the oil saponified. The specific gravities were taken by the method of floating the acids in alcohol, and the neutralization numbers by titrating them with aqueous alkali.

Tables III. and IV. contain the corresponding figures obtained from the oxygenated oils and their acids, and also those from some of the same sample of raw linseed oil, which, to compare the effect of drying, had been exposed in a flat dish to a moderate current of air at 45° C. for about five weeks, care being taken daily to break up any skin formed, and thoroughly mix up the whole mass. In this time, the oil assumed a jelly-like condition, lumps of comparatively hard material and skin, alternating with a small amount of oil of the consistency of middle varnish. The oxidised oils are much more readily soluble in alcohol, and, especially the thinner one, possess more strongly marked drying powers than the ordinary varnishes, and they are saturated with gas which caused them to effervesce on heating, and from which they could not perfectly be freed at the temperature of the water oven, even by prolonged heating. For this reason the determination of the specific gravities was not so satisfactory as in the case of the former var-

TABLE I.

Oil.	Sp. Gr. 15° C.	Free Acid.	Saponification Equivalent.	Unsaponifiable Matter.	Fahrian's Acids.	Iodine Absorption.
		Per Cent.		Per Cent.	Per Cent.	
Raw oil.....	0.9321	0.85	288	..	0.30	169.0
Tint varnish.....	0.9584	1.46	284	..	1.50	113.2
Thin varnish.....	0.9651	1.76	285	0.62	2.50	100.0
Middle varnish.....	0.9721	1.71	284	0.85	4.20	91.6
Strong varnish.....	0.9741	2.16	294	0.79	6.50	86.7
Extra strong varnish.....	0.9780	2.51	297	0.91	7.50	83.5
Burnt thin varnish.....	0.9675	6.93	287	1.35	0.85	92.7

TABLE II.

Acids from:—	Sp. Gr. 15.5° C.	Melting Point °C.	Solidifying Point.	Combining Weight.	Neutralisation Number.	Iodine Absorption.
Raw oil.....	0.923	24–26.5	..	286.5	195.8	145.5
Tint varnish.....	0.941	20.5	15	..	..	118.3
Thin varnish.....	0.949	22	18	..	..	108.8
Middle varnish.....	0.960	24	22	272.6	205.8	97.7
Strong varnish.....	0.953	25.5	21	270.1	207.7	87.3
Extra strong varnish.....	0.955	27	23	269.8	207.9	90.8
Burnt thin varnish.....	..	23	19	..	..	99.3

TABLE III.

Oil.	Sp. Gr. 15° C.	Free Acid.	Saponifiable on Equivalent.	Unsaponifiable Matter.	Polaron's Acids.	Iodine Absorption.
		Per Cent.		Per Cent.	Per Cent.	
Oxidised oil, weak.....	1.03	18.00-28.40	254	0.89	42.82	58.8
" strong.....	1.05	18.49-28.90	251	0.97	41.49	53.5
Dried linseed oil.....	..	12.67	327	0.84	31.58	39.9

TABLE IV.

Acids from:	Sp. Gr. 15° C.	Melting Point °C.	Solidifying Point.	Combining Weight.	Neutralisation Number.	Iodine Absorption.
Oxidised oil, weak.....	..	28	26	241.4	232.1	63.2
" strong.....	..	27	25	242.5	231.5	60.6
Dried linseed oil.....	..	26	22	265.8	208.7	100.3

nishes, as the flasks had to be heated for several hours to 125° C., and from the marked discolouration of the oils, it is probable that some change in their constitution had taken place. On titrating their alcoholic solutions with aqueous alkali, a point was found where the pink colour of the phenolphthalein remained after a vigorous shaking (the first of the figures given in the third column), but disappeared on standing for a short time protected from the air, the higher amount of acid recorded being obtained by adding the alkali a few drops at a time until the colour remained constant for two or three minutes. The other constants also show that the treatment with oxygen alters the constitution of the oil far more than simple boiling, and it was noticed that although the varnishes themselves are very pale, they darken, as previously mentioned, somewhat on heating and very considerably during saponification, so that the extracted acids are much deeper in colour than those from even the extra strong lithographic varnish in Table I., while on weighing only 84—85 per cent. (*cf. ante*) of the oil was recovered.

The small amount of unsaponifiable matter in all of these varnishes shows that the "difficulty" (this Journal, 1892, 848) of estimating the amount of rosin oil present as an adulterant is not so great as might be supposed, assuming that the latter is employed in its refined state, and if the crude oil containing resin acids (which from its colour is unlikely) or rosin itself has been used, Twitchell's process is available.

It may be observed here that while Lewkowitsch (this Journal, 1893, 505) and Twitchell agree in taking 346—348 as the average combining weight of the resin acids from rosin, this is only the figure obtained by titrating the original rosin itself with its 6—7 per cent. of unsaponifiable matter, whereas the "Twitchelling" is usually applied to the mixed acids after the unsaponifiable matter has been removed; and these acids have been found in a large number of cases to have an average acid number of 183, *i.e.*, a combining weight of 306. Working with this figure, or by preference gravimetrically, repeating a second time the treatment with hydrochloric acid gas whenever the extracted resins did not appear to be perfectly brittle or had the least smell of linseed oil, and considering them on finally weighing to represent 93 per cent. of the rosin, the author has obtained, when working on known mixtures, results within 1 per cent. of the truth, and preliminary experiments on mixtures of linseed oil or varnish and resins other than rosin show that the process will in all probability be found serviceable for, such separations also.

In conclusion, I have to record my indebtedness to the directors of Messrs. John Kidd and Co., Limited, London, in whose laboratory these results have been obtained.

## AN AUTOMATIC ALARM FOR WATER OVENS AND WATER BATHS.

BY CHARLES S. S. WEBSTER, F.I.C., F.C.S.

The apparatus consists of an upright cylindrical brass tube A, surmounted by a movable cap B, the upper end of which is plugged with a circular disc of ebonite C, having a fine

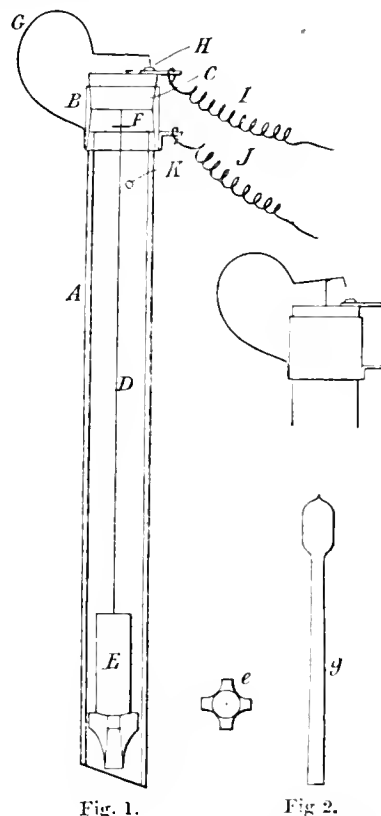


Fig. 1.

Fig. 2.

hole drilled in its centre, through which passes loosely the straight brass wire D (No. 25\*), bent at right angles above the disc, and extending below to the bottom of the tube.†

\* Numbers on standard wire gauge.

† Half-inch brass tube was used 5 ins. long.

On to the lower end of this wire is threaded the cork F, used as a float, and shaped as in the illustration (the largest part of this float is shown in section c). The wire is bent up and firmly fixed to the bottom of the float.

The reason for cutting the cork in this manner is obvious, *i.e.*, to avoid capillary attraction. A very small piece of brass foil is soldered to the wire at F, thus preventing the float from rising too high.

To the side of the cap is fixed the platinum spring G (the shape of which is seen at g). This spring is bent so that its point shall fall on the platinum button H, forming a make and break arrangement. It has been found most satisfactory to use platinum just twice the thickness of the ordinary analytical foil (*i.e.*,  $\frac{1}{1000}$  inch). The hole in the tube at K releases any steam pressure.

To fix the apparatus a large hole is cut in the top of the oven or outer ring of the water bath, and a perforated cork inserted, through which the tube is passed until it almost touches the bottom of the oven. The wires I and J are then connected with the electric bell and battery. The water in the oven will raise the float so that the spring G will be lifted, as seen in Fig. 2, and the bell will not ring.

When the water by evaporation has been reduced to the depth of about one inch the spring will be lowered so that its point will touch the platinum button, Fig. 1. The contact completes the circuit, and therefore brings into play any suitable electrical contrivance either for cutting off gas, supplying water to the oven, or ringing a bell so that the deficiency of water may be made known.

A more permanent float can be made with a modern bullet coating, which has been previously reduced in a lathe, and having three guiding wires at its lower end.

A somewhat similar apparatus may be made with a glass tube fitted with a cork at the top instead of the ebonite disc, and a circular wooden platform above to which the fittings are attached, but it is of course not so substantial as the brass pattern.

Meeting held March 7th, 1894.

MR. EUSTACE CAREY IN THE CHAIR.

## METHODS FOR THE RECTIFICATION OF OIL OF VITRIOL.

BY FRANK TATE.

RECTIFIED oil of vitriol is so widely used in the chemical and allied industries that I have ventured to think a short review of some of the methods at present employed and recently proposed for rectification may be of some interest to this Society.

As no doubt you are all aware, Prof. Lunge, in the last edition of his work on sulphuric acid and alkali, has brought us very closely up to date, and I am afraid that I have very little to add that is new. I trust, however, the few notes I have put together may be deemed worthy of a discussion which may prove of greater service to the Society than the paper itself.

Within the last ten or twelve years many suggestions and improvements have been put forward, and, as a result, the concentration of vitriol to the higher strengths is to-day a much simpler and more economical process than it was a few years since.

The only apparatus used by the majority of manufacturers in the United Kingdom up to a comparatively recent time, and indeed now very widely used, consisted of the familiar glass retorts resting on sand, in flanged cast-iron pots built into brickwork, and arranged in sets or benches in numbers as required, each retort having a separate fireplace.

Platinum was practically the only rival of glass, and, whilst glass continued to be the favourite material in this country, platinum was very generally adopted on the Continent.

I will not weary you with details of the various ideas patented in the last decade, but it will be clear to all those perusing the specifications that the chief object has been to bring about a continuous process in some form, and so obviate the great loss of time and fuel that necessarily attends the working of an intermittent process such as the old method of working glass retorts.

The Gridley, or as perhaps it is more generally termed, the Chance process, was one of the earliest steps in this direction.

Following it are proposals for continuous processes using a variety of materials, *e.g.*, various forms of platinum apparatus, retorts of iron with protection and without protection, iron lined with enamel or porcelain, brick-lined pans built into a furnace connected with brick-packed towers, &c.

At the present day, as far as I am aware, manufacturers have before them a choice of materials and systems which may be shortly classified as follows:—

1. Platinum—
  - (a) Platinum *per se*.
  - (b) Platinum gold-plated.
2. Glass and porcelain—
  - (a) Intermittent: without cooling pans, with cooling pans.
  - (b) Continuous: Chance's, Veitch's, Webb's, Levinstein's.
3. Iron—
  - (a) Iron *per se*.
  - (b) Enamelled iron (Dyson's).
4. The Kessler furnace system.

A manufacturer, in deciding on his apparatus, has to consider the following, amongst other points: 1st, the purpose for which the acid is required; 2nd, the strength of the acid available for concentration; 3rd, the space at his disposal for building; 4th, the first cost of the plant; 5th, the cost of labour, fuel, and wear and tear in working the plant.

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### SESSION 1894.

April 4th.—Mr. J. Henry Brown. "The Treatment of Cupreous Iron Pyrites as carried on at Portuguese Mines."

In considering the first point, both the purity and strength of the finished acid are important. If the acid is to be used in the textile trades for such processes as the bleaching and dyeing of cotton or woollen goods, or in the production of colours, &c., a plant composed of a material not liable to taint the acid must be aimed at, and platinum, glass, or porcelain at present claim first consideration.

The attaining of high strengths without undue wear and tear of plant is also of great importance for many purposes.

If the question is one of producing a quantity at a low cost, rather than purity of product, I think we shall find that iron has some claims.

I purpose to describe, very briefly, some of the methods at present in use for concentration, and to append some figures of costs.

**Platinum.**—Without attempting to more than refer to some of the legion of proposals as to form and arrangement of platinum plants, I think I may take it that the chief systems in use to-day are the Faure and Kessler, the Prentice, and the Delplace systems.

The Faure and Kessler system is a combination of platinum dishes with lead covers. It is very fully described by Professor Lange (Vol. I, p. 712, 725).

In the Prentice system the chief consideration is the enlargement of the heating surface by the use of a corrugated still bottom, which, it is said, increases the heating surface in the proportion of 1·57 to 1.

The system of Delplace consists of platinum stills working in series at different levels, the top still being fed with the weakest acid, the highest concentration being attained in the lowest still. This still is fed with the acid from the higher pan. The distillate from the lower pans also undergoes concentration through the series.

A form of still suggested by Desmoutis is shallow in depth, and has a bottom fitted with concentric partitions, giving a longer travel through the still.

A few details as to the cost of some of the platinum apparatus may, perhaps, prove interesting.

When working with the Faure and Kessler system, *i.e.*, platinum pans with lead covers kept cool with water (pans about 40½ ins. diameter), a system of two such pans is estimated to produce in a day of 24 hours from 11 to 12 tons of acid of 92 to 93 per cent., or about 8 tons of 95 to 96 per cent. Acid of a higher strength than 97 per cent. is not economically made by this system, as the wear and tear of the hoods is very great.

The weight of the platinum may be about 1,350 ozs. troy, which at, say, 40s. per oz., means 2,700*l.*, and to this price must be added the cost of the lead covers, the setting, and the vapour-condensing apparatus.

Using the Delplace system to produce 11 to 12 tons per day of 24 hours (of 92 to 93 per cent.) or 5 to 6 tons of 97 to 98 per cent., two platinum pans of a width of 19½ ins. and length of 58½ inches, with platinum covers and fittings weighing about 1,800 ounces troy, are needed, which, at 40s. per oz., equals 3,600*l.* A modified Delplace system has been suggested, in which one of the platinum pans has a leaden cover. This arrangement for the production of the same quantity of acid would reduce the cost of the platinum to 2,700*l.*

An arrangement of the Prentice system, using a combination of platinum pans with lead covers, and one closed platinum still, would cost rather more for the same production of acid than the modified Delplace system.

The labour on a platinum plant is of necessity small; the fuel amounts to about 3 to 4 cwt. per ton of 92–93 per cent. acid produced, but is increased to 5 to 6 cwt. if the higher strengths are aimed at.

In 1891 Mr. Heraeus, of Hanau, Germany, patented the plating of platinum with gold for use in the manufacture of vessels for the rectification of vitriol and other purposes. He proposed to heat the sheet of platinum above the melting temperature of gold, and to run over the same a layer of gold of suitable thickness. The details of this process you will find in his specification. He claims, amongst other things, that the platinum stills coated in this way will last a much longer time than ordinary stills.

Through the courtesy of Messrs. Domier, of London, I have received the following record of some results of the working of gold-lined pans from Mr. Heraeus:—

"At the beginning of February 1891 I supplied to a German works a piece of gold-platinum sheet—0·25 parts platinum, 0·005 parts gold—which was inserted in the bottom of a pan made of platinum only.

"After some time the pure platinum part of this pan became then through continual use, I took the pan out and it was found that on the gold-platinum sheet I had inserted no action of the acid whatever was visible, and so my idea was conclusively proved to be right.

"It was at first thought that it would be sufficient to coat the apparatus inside with gold only as far as the acid reached, or perhaps a little higher, and all my first orders were for pans made this way. I received orders from 11 works for plants—two on the Prentice system, two on the Faure-Kessler system, and the remainder on the Delplace system."

"These apparatus did not fulfil all expectations, especially those which were only coated with the absolutely necessary quantities of gold."

"Two others were stopped after working eight to ten months on account of the bottoms being burnt through in consequence of formation of crust."

"Five others are still working."

"Concentrating apparatus only partly gold-coated are not to be recommended, as at the place where the platinum and gold-platinum have been joined electrical action sets in and will destroy the platinum. The saving which might be effected by having apparatus only partly of gold-platinum is, therefore, more than neutralised by this loss, and I do not intend making any more partly gold-coated pans, but only such as are entirely coated with gold."

"These perfected apparatus have proved highly satisfactory in every respect, and the loss of metal in concentrating high percentage sulphuric acid is so infinitesimal that further improvements are practically out of the question."

"On the Continent and in America there are about 50 of these pans at work at present, but only from a few of these factories have I been able to get the exact loss, as these pans are, perhaps, only taken out once during the year."

"The results up till now surpass all expectation, and prove that the loss of metal in working gold-platinum pans is about one-twentieth of what is lost in working pans of platinum only. The following are some results:—

No. 1. One Prentice apparatus entirely made of gold, worked from the beginning of May 1892 to middle of June 1893 uninterruptedly, concentrating principally 66 Beaumé acid. Quantity not stated.

Total loss 39 grms., equal to about 0·08 gm. per day.

No. 2. One Delplace apparatus, only the lower part being made of platinum gold.

Production: 950 tons, 92 — 93 per cent.  $H_2SO_4$

" 491 " 96 " "

Loss per ton 0·11 gm.

No doubt a better result would have been obtained if the whole pan had been made of platinum gold.

No. 3. One Delplace apparatus, only the lower part made of platinum gold, concentrated 1,438 tons, 66 Beaumé acid. Loss per ton 0·17 gm.

No. 4. One Delplace apparatus—originally only the lower part made of platinum gold—concentrated acid of 92 to 93 per cent.  $H_2SO_4$ . Loss 0·04 gm. per ton.

This pan was returned to the works, and a new one made entirely of platinum gold supplied in its place. Since then 1,195 tons of acid have been concentrated, and it is impossible to state any loss in weight.

No. 5. One pan worked from the 14th July to 30th December, principally 97 to 98 per cent. acid. The daily concentration of about 6 tons of this high percentage gave a daily loss of 0·35 grms., equal to about 0·66 gm. per ton.

No. 6. Faure-Kessler pans coated only with gold as far as the acid reaches. In this case a better result would have been obtained if the pans had been made entirely of platinum-gold. A first result of a partly coated pan, working 93 per cent.  $H_2SO_4$  gave a loss of 0·45 grms. per ton.

The latest result with a pan made entirely of platinum-gold working 95.8 per cent.  $\text{H}_2\text{SO}_4$ , gave a loss of 0.2 gram. per ton acid.

No. 7. System same as No. 6, working acid of 93 per cent.  $\text{H}_2\text{SO}_4$ , formerly lost, using partly coated apparatus, 0.4 gram. per ton. The latest result, using apparatus entirely coated with gold—loss equal to 0.01 gram. per ton.

Quite lately one of the pans on the Delplace system was taken out and weighed.

The apparatus was entirely made of platinum-gold, and concentrated during nine months, working uninterruptedly—

1,728 tons of 95.95 per cent.  $\text{H}_2\text{SO}_4$   
135 " 96.96 " " "

The total loss on weight of the apparatus was 55 grms., equal to 0.03 gram. per ton of acid.

As a comparison of cost working with gold and without gold, taking the price of gold as 93.6s. per oz. troy and the cost of the platinum at 40s. per oz. troy, working on the Delplace system modified with gold 3,022l. 16s., without gold 2,766l. With the Faure and Kessler system with gold 2,999l. 12s., without gold 2,700l. The Prentice system with gold 3,081l. 19s., without 2,894l.

It will be seen that the first cost of platinum-gold apparatus is a serious consideration, but to firms with capital a heavy first cost simply means so much interest on the capital invested per ton of acid produced. Platinum plant of course occupies very small space, but the production of the very highest strength of sulphuric acid without undue wear and tear is perhaps a debatable point.

**Glass.**—In the working of the old form of glass plant the glass may be worked in one of two ways, 1st. working daily—the finished acid of the one day being run into cooling jars on the following morning before charging the retorts; or working on alternate days—two nights and a day being allowed for cooling before siphoning into earboys. If the acid is used in the works of the producer it can, of course be run into some form of cooler on the morning following the day of working, and the retorts be worked daily.

The first cost of a glass plant to produce about 10 tons per day of 24 hours may be roughly estimated at 800l. to 1,000l. inclusive of a condensing apparatus. As regards the fuel used there seems to be considerable diversity of opinion. I have seen statements giving the amount of fuel necessary to concentrate from 152° Tw. to 168°—170° Tw. varying in amount from 18 cwt. to 4 cwt. per ton of finished acid. The figure, using a fair class of fuel, will probably be 13 to 14 cwt. per ton of acid. The labour may be reckoned at 1s. to 4s. 6d. per ton finished acid.

**Wear and Tear.**—This varies very much according to the quality of the glasses and the workmen. For instance, Luty (this Journal, 1893, pages 153—154) tells us that in a plant directed at the Mulheim works for a daily output of three and a half tons of 169° Tw. vitriol per day, 35 glasses were broken during the first 14 months of working. This is at the rate of about two glasses per five months. My own experience is that this cost may be very much reduced, and I have known a plant of over 40 glasses run for some months without a single breakage. The building was a good one, and the glasses carefully protected from draught, and the foreman one of the most careful and conscientious men I have ever met in a chemical works.

On other parts of the plant the wear and tear is very slight, the iron pots, lasting, if properly set, for a very long time, and the brickwork requiring very occasional repairs.

**Chance Plant.**—This requires no description. For the production of 23 to 25 tons of strong vitriol per week the cost is about 300l., including about 100l. for a suitable shed.

One set of Chance glasses, four in number, will produce 10 to 12 tons of rectified vitriol per week from acid of 148°—156° Tw. Acid at 95 per cent. is readily obtained by this process, but some time is lost in getting up to 97 per cent. The cost of labour upon the plant is much less than on the old system, say, about 2s. to 3s. per ton of finished acid. The cost of fuel varies to some extent and depends upon the mode of firing—whether by means of a gas-producer or by hand-firing and ordinary fire-grate. It is of course considerably less than the intermittent system. I should put it down at about two-thirds of the

cost of the old system, and from 10 to 12 cwt. per ton of finished acid. The breakages of the glasses is not great, say, one glass to 180 to 200 tons of finished vitriol. About 1s. per ton will cover the general wear and tear. It is necessary in order to keep down breakages to work with clean acid containing as little soluble and suspended matter as possible. The plant requires a good building, but occupies little space. The labour should be intelligent and thoroughly skilled.

Mr. George Veitch of Crieff in 1889 protected an arrangement consisting of hard Bohemian or English glass retorts mounted in a line on a stepped terrace within a fire flue, all connected by a lip or spout between each of the retorts, so that the acid is made to flow continuously through the series from the retort on the highest step to that on the lowest. The specification described in this arrangement, which includes a drawing, is of considerable interest. I hoped to have had some results of working to put before you, but unfortunately they are not to hand, and I cannot say with what success the plant has worked.

In 1891, Mr. T. G. Webb of Manchester introduced a system in which beakers or cylindrical glass vessels are arranged in series in a furnace chamber. These glasses are open at the top with a lip or spout for overflow at one side. In each of these vessels is a glass tube presenting a funnel-mouth to receive the overflow from the vessels above, and extending down to the bottom of the vessel below, with a slit or other outlet at the lower end. The furnace chamber is covered by a sloping or stepped roof of iron plate in which are cast holes through which the upper part of the beakers project. Above the tops of the beakers is a roof of slabs with outlets leading to the condensing apparatus. The furnace chamber is fired by one or more fires, the fuel used being coke.

The hot gases from the fire play upon the beakers below the iron plate. The weak sulphuric acid is supplied to the highest beaker, flowing to the bottom of the beaker through the tube, gradually causing an overflow of the acid by the lip into the tube of the next beaker, and flowing from beaker to beaker, becoming more and more concentrated till at last it flows into the cooler. The cooler consists of porcelain dishes surrounded by cold vitriol, which is contained in a vessel surrounded by cold water. The plant can be arranged in one of two forms, either in the cascade arrangement, as just described, or in the horizontal arrangement, in which syphons carry the acid from one vessel to the other.

Mr. Levinstein of Manchester, in 1892, brought forward an interesting arrangement in which the acid is caused to traverse a descending series of vessels in a type of apparatus in some respects, at least, similar to the one I have just described.

Reverting to Webb's patent, the cost of plant for a run of 14 glasses, exclusive of royalty, is from 60l. to 100l., according to the elaboration of details. The plant occupies very little space, two runs of 14 glasses, or a smaller number, are generally arranged side by side, working as one set. Mr. Webb commences with glasses, but is now using porcelain as the material of which his beakers are made. From some figures that have been kindly placed before me from an independent source, a run of 14 vessels will produce a carboy of 14.9 Tw. acid, from 148° Tw. acid, per hour using less than half a cwt. of coke per hour.

One man can manage four runs easily, and in this way the labour is only about 2d. per carboy. The cost of labour, of course, depends entirely on the number of runs in work in the same building. One man can manage four runs, but it will take one man to manage one run also. Fourteen glasses will produce 5 to 6 tons per week of strong acid from 120° Tw. acid, or 11 to 12 tons per week from 148° Tw. acid. The fuel is put down as about 3 to 4 cwt. of coke per ton of rectified vitriol when working with 148° Tw. acid, or 6 to 7 cwt. when working with 120° Tw. acid.

**Wear and Tear.**—Working with glasses by this system the wear and tear, I understand, is considerable. With the porcelain vessels it seems to be very much reduced, and a gentleman who has been working some time with the process informs me that he has had no breakages since he replaced the bottom two or three glasses by porcelain vessels. Of course there is some wear and tear upon the brickwork,



but it should not be heavy. The flow of the acid is controlled by means of a thermometer inserted in one of the beakers some three or four removed from the bottom beaker.

With this plant any strength of acid may be boiled down without the use of a leaden pan for previous concentration. Mixed acids, such as we meet with in explosive works, can be readily concentrated with such an apparatus as this to very high strengths. There should be little or no loss of acid apart from the question of dissociation and loss through the condenser. Any acid spilt by overflow of the beakers or by breakages of the beakers is caught in suitably arranged gutters and returned into process again. In case of breakage only a very short time is necessary to replace the broken vessel. What has been said as regards cost of fuel and labour in reference to Mr. Webb's plant applies also to the plant which Mr. Levinstein claims.

Another form of porcelain apparatus is that of capsules arranged in cascade as proposed by M. Negrier, and described by Prof. Lunge. I have endeavoured to obtain some details of the working of this plant, but have been unsuccessful.

**Iron.**—Without entering into the history of the use of iron pans for concentrating acid, which would at least include the patents of Gridley and Menzies, it will be sufficient for me to remind you that for many years cast-iron pans have been used with more or less success for concentrating vitriol. I have received one communication with reference to the use of a wrought-iron pot  $\frac{3}{4}$  of an inch thick, about 6 ft. diameter, and 4 ft. deep, which some years ago was bought second-hand from a tar distiller. A mixture of sulphuric and nitric acids of about 159° Tw. has been concentrated in this pot for a long period, the nitric acid being recovered, and sulphuric acid of 169° to 170° Tw. being produced at a very low cost for fuel and labour. This pot is still working.

I believe cast-iron pots are in use in many places for concentrating vitriol from 148° Tw. to a high strength. The larger pots are about 9 feet diameter and 6 feet deep, 2 ins. thick, and weighing 10 to 11 tons. The pot is covered with a cast-iron lid. It is set in brickwork, and fired with a single fire. If the acid is run in at about 148° Tw. in 28 to 30 hours, it will be at about 96 per cent. Fresh acid is run in from time to time. Twelve tons of 96 per cent. acid can be finished in such a pot. If the acid is not required very pure, 12 hours is sufficient for settling and cooling, and the pot can be worked about seven times per fortnight of 14 days, producing 42 tons of acid per week. The fuel consumed amounts to about 2 cwt. per ton of finished acid. The labour can be reduced to 1s. per ton, and need not be as skilled as with other plants. Wear and tear is heavy, and amounts to from 2s. to 4s. per ton of finished acid. A pot will finish from 500 to 600 tons of acid before it is necessary to replace it. The pot has to be sludged out from time to time, and this means a loss of acid in the form of iron salts, &c., to from 4 to 5 per cent. on the finished acid. The cost of the pot is about 60*l.*, of the cover 10*l.*, brickwork setting about 30*l.* One cover outlasts three or four pots, and, of course, the old pot can be sold for scrap, and will be worth from 10*l.* to 15*l.* The plant requires no costly building, and occupies little space. The highest strength acid can be produced, and the most impure acids can be dealt with. Acids containing matters in solution and suspension which would cause considerable trouble in other plants can be readily treated in the iron pot.

**Enamelled Iron.**—In 1883, William West of Denver, through Mr. W. P. Thompson, brought forward an apparatus for concentrating sulphuric acid in an enamelled or porcelain lined retort. The process was to be a continuous one. I am not in a position to speak about the success of this, as I have no facts before me. Mr. Dyson of Manchester has recently brought out another form of apparatus having enamelled iron as its material. The material employed is of special manufacture. The plant consists of a series of enamelled iron vessels through which sulphuric acid passes, being rendered more and more concentrated until it reaches the end of the series, where it passes into a suitable cooler. According to particulars supplied to me by Mr. Dyson, for a plant producing 21 tons per week of

seven days concentrating from 52° B. to 66° B., the ground space required for a single run of 18 retorts is 20 sq. ft., and for the cooler 12 $\frac{1}{2}$  sq. ft. The elevation from the floor line is 8 ft. 6 ins. The cost of the plant, for the production of 42 tons a week, is about 500*l.* The fuel used is common coke, with a consumption of 6 cwt. per ton of 112° Tw. acid. The cost of labour is the wages of a man working day and night. I can say nothing of the cost of wear and tear on this plant. I believe it has been a work for some time, but for how long I cannot say. I can say what results, as regards wear and tear, I am not in a position to speak. As in the case of the Webb plant there need be no loss of acid, as suitable gutters are provided to catch any spilt, every part of the plant is readily accessible, each vessel is separate and distinct, and can be renewed very easily and at a small cost.

**The Kessler Furnace System.**—In 1884 M. Kessler of Clermont-Ferrand, France, patented an invention for the concentration of sulphuric acid by means of heated gas acting on large or greatly developed surfaces of sulphuric acid, so as to readily concentrate the acid to its maximum density at a low temperature. The troughs or compartments in which the concentration was to be effected were to be made of any cheap acid-resisting material able to withstand the necessary temperature.

It is impossible in this paper to do justice to this very interesting suggestion. I can only shortly describe the apparatus, which consists of two chief parts:—the one spoken of as the saturator, which consists of a large rectangular trough made of material that cannot be attacked by the hot concentrated acid (such as pumice or sandstone), the joints being made of a cement also of acid-resisting material. The trough is divided by a series of parallel partitions into compartments, some of which receive the gases which arrive through cast-iron pipes, which gases then pass into other passages raising or grazing the layer of acid which occupies the bottom of the saturator and rising into the upper portion of the apparatus. The upper portion of this arrangement is placed either directly or laterally above the saturator, from which it receives its gas and to which it sends its acid. It consists of a series of horizontal compartments placed one above another pierced with openings which are covered with caps, which force the acid to blow strongly upon the liquid or to furrow it across. The acid proceeds horizontally in a shallow layer from one wall to the other, where it meets with the return pipes, which carry it down to the lower compartment by an arrangement somewhat similar to ordinary compartment stills. The upper portion of the apparatus may be constructed entirely of lead, but preferable, if pure acid is necessary, of free-stone or earthenware. The hot gases are exhausted through the apparatus by some arrangement producing a vacuum. The fuel used is small coke from gasworks, which is too fine for use for other purposes. The consumption, it is said, does not exceed one and three-quarter cwt. per ton of acid coming direct from the chambers at 52° B. This consumption is a little increased if any fuel is used for producing a vacuum. The labour with a special stove, in which the coke furnace receives the fuel necessary for 24 hours' work, is hardly anything. The apparatus has been in operation for two years at Clermont-Ferrand, and a further apparatus, it is said, is about to be erected. For these particulars as regards this very interesting system of Kessler's, I am indebted to a paper in the "Moniteur Scientifique" by M. Gerdier. Before closing these remarks I think I should do well to state that attention has been called in the Inspector's report on alkali works to what appears to be an increased escape of gas from the condensers when working with continuous processes other than platinum. I wish in conclusion to acknowledge the kindness of Messrs. Webb, Levinstein, and Dyson in affording me opportunities of viewing their plants.

#### DISCUSSION.

Mr. M. L. DAVIES said that the best method of rectification depended upon the purpose for which the acid was required; that determined the percentage and colour. He was speaking without experience of the Kessler oven, but

imagined that great care should be exercised, or the acid would become discoloured. For some purposes that would be a great detriment.

In this country a large amount of rectified vitriol used was only 92-97 per cent.; in America it was almost exclusively that percentage. With regard to wear and tear, he did not think there was much difficulty with any process in securing 95 per cent. of monohydrate, but the moment one attempted to get beyond that, difficulties arose either in the construction of the plant or in the consumption of fuel and further, there was a considerable reduction of the output. In some experiments he found, when trying to make 97 per cent. acid, the output had been reduced by one-third.

In regard to the form or system employed, he was acquainted with most of the platinum stills, but would favour the Faure and Kessler system because of its ready access for repair and also because of the small amount of acid it held. The secret of concentration to his mind was to have a small depth of acid in any still. That he thought was defeated by some of the other processes.

As to comparison between glass and platinum, he could hardly think to which to give the preference for ordinary strengths, but for very high strengths glass had the advantage. He had had some experience with glass, and with regard to Chance's had a fair experiment made and found that he got results agreeing with Messrs. Chance's figures in output and consumption of coal gas; but in consequence of the high price of gas in Liverpool, the cost of fuel was much heavier than with slack in the platinum installation.

As to the cost of material, he thought that his experience would hardly agree with the lecturer's statement that 1s. per ton would cover the cost of glass.

Mr. TATE replied that from recent experience 1s. covered wear and tear of the plant other than of glass; he thought that he had mentioned that the glass amounted to one glass per 180 to 200 tons of finished acid; one retort cost 25s.

Mr. DAVIES, continuing, said he had found from figures that the loss of material over 12 months in platinum was 3s. per ton finished vitriol, and by glass 5s. 6d., which latter was accounted for by the heavy breakages. He did not wish to infer that this was due to the poorness of the material. They had it very well cased in, but each time a stoppage was made by the breaking of a retort, the others frequently cracked at the boiling surface owing to the admission of cold air, and it became a question whether it would not be better to renew the whole of the retorts at once.

In regard to the loss of platinum, he was looking over an old book recently and found that in 1875 when platinum pans were brought into Liverpool for the first time, several pans showed a loss of about only 0.3 grms. per ton; following it down to the present time he found a gradual increase and it appeared to him that the quality of the metal had depreciated. The quicker destruction of pans may be due to the presence of iridium and other members of the platinum group, which would render the metal more brittle and its elasticity would in consequence be reduced, which is a very important matter, especially when using pans of 40 ins. diameter and upwards. Occasionally good results were yet obtained, for instance, he found in 1890 for a matter of something like 500 tons the loss was only 0.34 grms. per ton.

He had tried several experiments to get 97 per cent. monohydrate, but with a tremendous loss of something like 20 grms. of platinum per ton. It was not only the finishing pan that suffered, but the first also, which usually scarcely suffers at all. Owing to the heavy firing and a much reduced feed, the vitriol in each pan became of the same high specific gravity and that accounted for the parallel loss.

Referring to the consumption of fuel, he did not think Mr. Tate's information was correct, that was to say, he did not find that one got reliable information from men working comparative experiments. They seemed to have an idea that the latest introduction should be the best, and unless one was very careful to check the men, the figures obtained were apt to be misleading. From his

own experience the average consumption of fuel per ton finished rectified 95 per cent. acid, which included concentration from 100° to 168° Tw.,—was about 5 cwt. of good Lancashire slack. It will be well to remember the difference between Lancashire, Staffordshire and Scotch varieties. Some of the coke figures he thought were too high.

Mr. A. E. FLETCHER asked Mr. Tate if he had examined the glass which was commonly used. It struck him as strange that the manufacturers should be satisfied with the ordinary soft lead glass. In every case he had examined the glass was of that description. There was no great difficulty in getting a harder glass. Still, as far as he knew, most glasses that were used were of the soft glass type. He had often expressed surprise that there was not more effort to get the hard flint glass, which would be less easily attacked by acid, more able to resist mechanical strain, and less liable to crack with change of temperature. Did Mr. Tate know any reason why that glass was not used?

Mr. R. F. CARPENTER said in the latter part of Mr. Tate's paper, mention had been made of the work of Inspectors under the Alkali Acts, in estimating the loss of acid during rectifying. He had had many of these continuous as well as intermittent processes under his purview, but had not made any definite quantitative test of the acid lost, so as to compare the one type of process with the other in this respect; such a comparison required that the speed of the exit flues be determined by the anemometer, and in the last report he guarded himself from straining the results of "total acidity as  $\text{SO}_3$ " tests further than they would bear. He would mention one danger that some of the continuous processes described by Mr. Tate appeared to him to be under, viz., those in which a comparatively small amount of liquor was in process at one time, surrounded by heated brick flues, which retained a large reserve of heat. Firing in such processes was controlled by thermometric indications, and the run of acid and the firing were regulated thereby. With slight alterations of run or of heat supply, he had found conditions established which led to a very considerable loss of acid by volatilisation and dissociation. The thermometers in use required themselves to be carefully tested from time to time, else the same losses might be going on when the man in charge of the plant might deem that all was going on right. In the modified Delplace platinum plant the hydrometer in the distillate from the strong pan or retort gave immediate indication of too heavy firing and the remedy then for over-heating was equally immediate in its action.

In the description of the Kessler process, p. 36 of the January number of the Journal, it was stated that rectification to 66° B. could be attained at a temperature not exceeding 170° C. None of the other processes mentioned by Mr. Tate could claim that the process finished at that low temperature; and as regards dissociation loss the superposed "recuperator" seemed specially adapted for arresting this anhydride if volatilisation took place, as it most probably would in the saturator under the influence of fuel gases entering and bubbling through the acid at 500° C. Mr. Levinstein had shown how readily the anhydrous acid was absorbed in strong acid near the point of monohydrate such as would be found in the lower compartment of the recuperator. But whether this system could be largely adopted must depend, as Mr. Davies stated, on the uses to which the acid was to be subsequently applied; some customers requiring the acid to be perfectly bright and colourless. He thought it would not do to claim too much for the method of rectifying in cast iron as regards the highest strengths, however useful it might be for works requiring acid of 95 to 95½ per cent. It had been found that in attempting by continued firing to get a higher strength that the loss by volatilisation and dissociation was greater than the concentration, so that the acid in the pan actually became weaker, as he had stated in the last report; this did not seem to be the case with platinum, though the action on the platinum certainly increased then very rapidly indeed.

Mr. E. K. MURRAY said he thought there was a good deal of misunderstanding as to the breakage of glass retorts. It depended very much upon the quality of glass in the retorts. They had had glass retorts that had broken almost daily and others had lasted nearly a year. Their experience had always been that there was great variation in the quality of glass and platinum that one worked with. In the case Mr. Davies spoke of, there was at that time great breakage and cost of material. He was trying to make extra strong concentrated sulphuric acid—a strength about 98 per cent. He thought that would account for the heavy cost of breakages at that time. His own experience was that on the whole for concentrated acid, about 93 per cent. or 94 per cent., the Faure and Kessler apparatus was the most economical in regard to fuel and wear and tear. The wear and tear which one did not work to concentrate beyond this strength was under  $\frac{1}{2}$  gram of platinum per ton of rectified acid. The fuel was extremely low, but when one spoke about the quantity of fuel, one must know the strength of the acid delivered to the apparatus, and the strength to which it was concentrated. Unless they had those two factors before them it was absolutely ridiculous to make any comparison as to quantity of fuel used. Every inventor always tried to give the best results, and not the whole truth. The Faure and Kessler new method was extremely interesting, but there were some objections to it, otherwise they might have tried it. What surprised him was the number of these new inventions, while the saving was so small. It showed there had been enormous competition in the sale of sulphuric acid, and everybody had been trying to get strong sulphuric acid at the lowest possible cost. A large amount of money had been spent on experiments, and now after 20 or 30 years' experimenting with platinum and glass, there was not much to choose between the various methods and the choice depended upon local conditions.

Mr. NICHOLSON said that in regard to the discoloration of the acid produced in the Kessler process he had received a sample of the acid produced from Mr. Kessler and it was as clear as spring water.

Mr. TATE, in reply, said he quite agreed with Mr. Davies that one of the chief features to be considered was the purpose for which the acid was to be used. If the pure acid was required, one must have a suitable plant that would not taint the acid. If, on the other hand, they had to work up an impure acid containing matters in solution and suspension, then, he thought, they would find glass practically out of the question, as also platinum, owing to the formation of deposits and crusts, and consequent wear and tear.

As to the discoloration of acid produced by the Kessler furnace, Mr. Nicholson had already referred to that.

Mr. Davies had said the strength of acid in this country was usually 92 per cent. to 93 per cent. That might be so, but he knew that in Lancashire a great deal of stronger acid was sold. He thought that Mr. Davies' experience of breakages with the Chinese plant was rather unique. It must have been an unfortunate season. The figures he had been able to obtain were certainly very much lower than Mr. Davies' figures. As regards the loss of platinum, the figures he had mentioned referred to plants in which gold-plated platinum had been used.

As to how far the quality of glass affected the wear and tear of glass plants, he regretted he was unable to give any information. He had used glass made in various works and found that some makers could supply a glass which would stand excellently. He had known times when a plant had run for months without breakage, but that he thought was due to careful annealing of the glasses and the watchfulness of the workman.

## ESTIMATION OF CADMIUM.

BY MAX MURRAY.

The following examination of the usual methods for the gravimetric determination of cadmium was carried out under the supervision of Prof. F. C. Clark, at the analytical laboratory of the Polytechnic School, Zurich. The time at my disposal was too limited to allow an exhaustive comparison of the various methods, and I do not claim to have made such, but I found a few facts not hitherto recorded, which greatly influence the accuracy of the method of determination of cadmium in the form of oxide and which may prove useful to others.

Of the three methods usually recommended for the gravimetric estimation of cadmium, a few words will suffice to show my way of applying them.

(1) *Determination of cadmium in form of oxide by previous precipitation with sodium carbonate*.—This is the method to which I have paid most attention. Its apparent simplicity led me to adopt it as a standard method with which to compare the other methods, until I discovered its errors. The cadmium is precipitated from its solution with a solution of sodium carbonate. The precipitate of cadmium carbonate is washed, dried, and separated as completely as possible from the filter paper, then ignited in a porcelain crucible, the filter paper being burnt in a platinum coil, and added to the contents of the crucible.

(2) *Determination of cadmium in the form of sulphide*. (Ros's method).—The cadmium is precipitated from its acid solution by sulphuretted hydrogen and the precipitate ignited in a current of hydrogen gas.

(3) *Electrolytic methods*. The several electrolytic methods differ chiefly in the solvent employed. I used (1) solution of KCN, (2) dilute H<sub>2</sub>SO<sub>4</sub>, (3) solution of ammonium oxalate. In either case a large platinum dish served as the anode, the anode being formed of a platinum spiral. The current employed was measured by an ordinary voltmeter, and was of such strength as to yield from 0.2 to 0.5 cc. of electrolytic gas per minute. The deposited metal was washed with alcohol and ether, dried, and weighed.

For comparing these various methods I employed a solution No. 1 of cadmium sulphate containing about 7.4 grms. of crystallised cadmium sulphate in 2 litres. For each determination a few cc. of this solution were employed. I give the results of two series of determinations made by the first method, i.e., by precipitation with sodium carbonate.

### I. CARBONATE METHOD.

50 cc. gave—

(a.) 0.0850 grms. Cd.	= 0.071 grms. Cd.
(b.) 0.0851 " "	= 0.0717 " "
(c.) 0.0854 " "	= 0.0719 " "

### II. CARBONATE METHOD.

50 cc. gave—

(a.) 0.0801 grms. Cd.	= 0.070 grms. Cd.
(b.) 0.0807 " "	= 0.0703 " "
(c.) 0.0802 " "	= 0.0702 " "

whereas the electrolytic method yielded for the same solution:—

50 cc. gave:—

(a.) 0.0794 grms. Cd.
(b.) 0.0799 " "

It will be seen that the results of each series are practically identical, but that the results of the two series, which ought to have been alike, differ from each other, and the results of the carbonate method are all of them much lower than those obtained by the electrolytic method.

In consequence of these conflicting results the electrolytic methods were more fully investigated, and were found to give identical results whatever modification was made in the solvent.

A solution (No. 2) containing 7 grms. of crystallised sulphate of cadmium in two litres of water was employed for these and the following experiments:—

## ELECTROLYTIC METHOD.

Solvent—	In 50 cc. found.	Grms. Cd.	Mean.
KCN.....	(1.) 0.0750 (2.) 0.0764	0.0753	0.0753
H <sub>2</sub> SO <sub>4</sub> .....	(1.) 0.0763 (2.) 0.0766		
Ammonium oxalate.....	1. 0.0771 2. 0.0765	0.0768	

The same solution yielded by Rose's method in 50 cc. 0.0782 grms. Cd., corresponding to 0.0764 grms. Cd.

By the carbonate method very inconsistent results were obtained. 50 cc. of the solution yielded:—

1.	0.0755 grms. CdO.
	0.0828 " "
	0.0893 " "
	0.0771 " "

The absolute consistency of the results obtained by the electrolytic method justifies their adoption as normal results.

I next endeavoured to find the causes of error of the carbonate method. The most obvious source of error might be expected to be found in the comparative ease with which cadmium oxide is reduced to the metallic state, and then volatilised, particularly that part of the oxide which, still adhering to the filter-paper, is ignited in contact with it in the platinum spiral. An addition of ammonia nitrate to the filter paper failed to improve the results.

I then tried the following modification of the carbonate process. The first precipitate of cadmium carbonate was dissolved in pure nitric acid, and the solution evaporated to dryness on a water-bath in a large porcelain crucible (previously tared). The cadmium nitrate, which formed a white cake at the bottom of the crucible, was gently heated and thus converted into oxide.

The oxide obtained by this ignition of the nitrate formed a perfectly compact black film, adhering to the crucible, from which it could only be removed by solution in acids, whilst the oxide obtained by ignition of the carbonate was a red-brown earthy powder.

The following results were obtained by this method:—

In 50 cc. of the solution used for the previous experiments (No. 2 solution) were found:

1.	0.0870 grms. CdO = 0.0761 grms. Cd.
(2.)	0.0897 " " = 0.0759 " "

These results agree fairly with those obtained by electro-deposition, but the agreement is more apparent than real, for the cadmium oxide was found to contain a small but quite appreciable quantity of sulphate. The reagents employed were quite pure, so that the sulphate must be due to the precipitation of a small quantity of basic sulphate with the original carbonate.

This error might be obviated or diminished by repeated solution and precipitation, but the method loses much of its simplicity. Whatever other faults the original method with sodium carbonate may have, it is a curious fact that the oxide obtained by the ignition of the carbonate does not contain any cadmium sulphate. Since the precipitated cadmium carbonate must have contained sulphate, this can only be explained by dissociation of the sulphate during the ignition of the carbonate, which does not take place during the ignition of the nitrate. Considering the great difference in the physical properties of the two oxides, such a difference in their chemical behaviour is quite conceivable.

The following modification of the process was next tried:—

The cadmium was precipitated as carbonate washed and dried. As much as possible of this precipitate was scraped into a weighed crucible and converted into oxide by gentle ignition. The oxide was free from sulphate. This was weighed by itself (a).

The filter paper was treated with pure nitric acid and the resulting solution and risings brought into a large porcelain crucible and evaporated to dryness. The dry nitrate was gently heated and the oxide weighed (b). The oxide

obtained in this way probably contained some sulphate, but the amount of oxide is so small a portion of that obtained under (a) that the error cannot be appreciable.

The following results were thus obtained:—

50 cc. of solution No. 2 yielded—

	Precipitate (a).	Filter (b).	Sum a + b =	Cadmium.
(1.)	0.0629	0.0209	0.0838 =	0.0753 grms.
(2.)	0.0485	0.0346	0.0831 =	0.0727 "
(3.)	0.0605	0.0231	0.0836 =	0.0731 "

It will be seen that the results are still too low. If, however, the precipitate (a) is considered to be Cd<sub>2</sub>O instead of CdO, and the results be calculated on this assumption, they will be found to be correct and to agree with the results obtained by electrolysis. This fact led to the final modification, consisting in heating the oxide (a) in a stream of oxygen until no further increase in weight takes place (maximum weight (c)).

The above precipitates (a) heated in a current of oxygen to constant weight finally furnished the following results:—

	G.	(b)	G + b.	Cd.
(1.)	0.0595	0.0209	0.0805 =	0.0765 grms.
(2.)	0.0725	0.0346	0.0871 =	0.0762 "
(3.)	0.0637	0.0231	0.0868 =	0.0760 "

These results are identical with those obtained above by the electrolytic methods and by Rose's method.

The method to be recommended is the following:—

Precipitate the cadmium as carbonate with sodium carbonate, filter, wash and dry. Detach as much as possible of the precipitate from the filter, and heat it in a porcelain crucible in a stream of oxygen. Heat gently at first, gradually raise the temperature, but never use a blast. Dissolve the small quantity of precipitate remaining on the filter in nitric acid, evaporate to dryness and convert into oxide by gently heating. Weigh this oxide by itself.

It is probable that a current of air might replace the oxygen, but I have not made the experiment.

With respect to the other methods I will add but few words.

Rose's method gave good results, but I found it too tedious for general use. It is important to remember the two allotropic forms of cadmium sulphide as regards ease of manipulation. If the precipitation with H<sub>2</sub>S takes place in dilute acid solution the precipitate is yellow, very fine and consequently difficult to wash. If the cadmium is precipitated from a concentrated acid solution the sulphide is reddish brown, very dense, and easy to work with.

I also made an attempt to adapt *Vollhard's method* for the estimation of zinc to the estimation of cadmium. Vollhard's method consists in evaporating the solution of zinc chloride to dryness in a platinum dish, mixing it with pure mercuric oxide and a little water, evaporating again and then gently heating. Mercuric chloride and the excess of mercuric oxide volatilise and zinc oxide remains behind. Whilst the method is satisfactory for zinc it has not proved useful for cadmium. Cadmium chloride is so volatile that it escapes with the mercuric chloride, and the results are much too low. When cadmium nitrate was used the presence of sulphate seriously interfered with the accuracy and the method was more complicated owing to the introduction of extra reagents and manipulations.

Of all the methods examined, preference must undoubtedly be given to the electrolytic methods for their simplicity, cleanliness, and accuracy. What I have said above sufficiently describes the method. Cadmium does not oxidise, and with a moderate amount of care very satisfactory results can be obtained. The chief precautions are to use dilute solutions and weak currents. Of the three

solvents I have enumerated I prefer the use of dilute sulphuric acid, because it yields accurate results, evolves no poisonous fumes and does not require the regulation of temperature.

Quite apart from the interest which the observations on the oxide of cadmium possess with respect to the quantitative estimation of the metal, they raise the question as to the composition of the oxide obtained by the ignition of the carbonate.

The existence of an oxide of the composition  $Cd_2O$  has often been stated as doubtful. The facts I have communicated above seem greatly to strengthen the case for its existence.

The oxide obtained by ignition of the carbonate of cadmium is a dark brown powder. By oxidation in a current of oxygen as described it still retains its colour and its form. Prolonged heating with an ordinary flame, even after discontinuing the stream of oxygen, does not cause any change. When heated with the blast a gradual loss of weight takes place, accompanied by a gradual change from the brown modification to the black. The loss in weight thus incurred bears, however, no definite relation to the original weight of the oxide, and cannot be compensated for by again heating in a current of oxygen. Indeed, the loss subsequently is so great that it cannot be otherwise accounted for than by volatilisation of cadmium.

The comparative ease with which the original oxide absorbs oxygen speaks more for its being an oxide  $Cd_2O$  than a mere mixture of the normal oxide  $CdO$  with metallic cadmium, since metallic cadmium is very difficult to oxidise.

The loss incurred on heating the normal oxide over the blast appears to be due not to a reduction to  $Cd_2O$ , but to the direct volatilisation of the oxide as such.

## SUGAR BEETROOT GROWING EXPERIMENTS IN ENGLAND, SCOTLAND, AND IRELAND IN 1893.

BY G. SCHACK SOMMER, F.R.S.

(Abstract.)

The author has continued his experiments on the growth of sugar beetroot for the fifth consecutive year. (This Journal, 1890, 153; 1891, 318; and 1893, 233.) The results, of which the following is a summary, are detailed in "Sugar," 1894, VI., pp. 43-47. They again give most satisfactory proofs of the possibility of successfully raising a larger crop of good rich quality roots in this country, and corroborate the fact that both our soil and climate are favourable to the growth of sugar beetroots. The results of 1893 show that, despite the large size of the roots obtained in England, the purity coefficient is wonderfully good, and that the percentage of sugar per acre is larger than that recorded by Mr. Licht for Germany. The Scotch roots also show a high purity coefficient.

### SUMMARY RESULTS OF THE EXPERIMENTAL CULTIVATION OF SUGAR BEETROOTS IN ENGLAND, SCOTLAND, AND IRELAND IN THE YEAR 1893.

	England.				Scotland.				Ireland.			
Number of experiments.....	22				22				2			
Average weight of entire plant in grms.....	1,147				1,191				673			
"    "    roots in grms.....	927				658				556			
Quantity of sugar in 100 parts of the juice .....	13.61				14.00				11.83			
Quantity of roots without leaves grown per acre in tons.....	Ton.	Cwt.	Qr.	Lb.	Ton.	Cwt.	Qr.	Lb.	Ton.	Cwt.	Qr.	Lb.
	19	9	1	0	26	8	3	0	14	15	0	0
Quantity of sugar per acre .....	2	13	0	10	3	14	0	3	2	3	3	0

### ERRATUM.

#### THE MANUFACTURE OF STRAW CELLULOSE.

BY JAMES BEVERIDGE.

The following table was accidentally omitted (this Journal, 1891, 101-108).

#### YIELD, ETC. OF AIR-DRY PULP FROM STRAW BY SODA-PROCESS.—FROM MANUFACTURING PRACTICE.

W. Roth. Papier Zeitung, No. 75, 1890.

Situation of Works.	1,000 Kilos. of Straw required			1,000 Kilos. of Straw Yielded	100 Parts of Air-dry Pulp required		
	Soda Ash.	Lime.	Bleach.		Soda Ash.	Lime.	Bleach.
South Germany.....	Kilos. 225	Kilos. 160	Kilos. 105	Kilos. 459	Per Cent. 50.0	Per Cent. 35.5	Per Cent. 23.3
Austria.....	225	160	72	400	56.25	40.0	18.0
Saxony.....	240	150	85	435	55.1	34.4	19.5
Bohemia.....	200	160	175	500	40.0	32.0	35.0

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*Hon. Local Secretary:*

J. Carter Bell.

Bank House, The Cliff, Higher Broughton, Manchester.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

SESSION 1893-94.

*Meeting held Friday, 2nd March 1894.*

DR. E. SCHUNCK, F.R.S., IN THE CHAIR.

### THE SAMPLING OF MATERIALS.

BY T. CLARKSON, A.R.S.M., ETC.

EVERYONE who is connected with the chemical industries appreciates the importance of sampling. The selection of a small but truly representative portion may be regarded as the first step in determining the composition of a large quantity of material.

To obtain a sample by hand necessitates much time, labour, and care. Consequently it is not surprising that the portion obtained is frequently inaccurate and non-representative. It is to be regretted that the word sample is often used in a loose manner as a general term for a small portion of material. It would avoid confusion if its use was restricted to such small portions as are truly representative of the bulk.

It is generally acknowledged by chemists that making every allowance for different methods of analysis, the differences in analyses are more often due to defective sampling than to subsequent mistakes in manipulation.

A complete investigation of the theory and practice of sampling is beyond the scope of this paper, but we may briefly examine the principles affecting the selection of a small true average portion from a large bulk of material. It is scarcely necessary to state that all solid materials which have to be sampled must be crushed or otherwise obtained in a more or less granular form. The degree of fineness of the material before taking the first sample is important. If the bulk is very large, say a cargo of phosphates or pyrites, the pieces may be proportionately large, provided that the first sample selected is of fair size, say from 2 to 10 per cent. of the whole according to the material, but before further reducing the weight of the sample the size of the pieces must be reduced upon the basis that there must be a certain minimum number of particles in a sample, below which it is unsafe to go if the representative character of the sample is not to be endangered. What this minimum number is, must be determined experimentally for each class of materials. As an illustration of this principle, assume that there are 50 bricks of a material designated by A, 25 of another material B, 15 of C, and 10 of D. Dividing these numbers by their greatest common measure, namely, 5, there would be 10 of A, 5 of B, 3 of C, 2 of D. These bricks, if taken together, would be a representative sample of the original lot, and would be one-fifth of its weight. To attempt to reduce the bulk

of the sample below this point without sub-dividing the pieces would clearly result in destroying the sample. This also illustrates the difficulty of properly sampling a large bulk of waste material containing only a few particles of value. As, for example, in the case of some gold ores, consisting of a large amount of barren quartz containing a few small nuggets or specks of metal. In such a case it is imperative to crush it all very fine and sift out any flattened nuggets that will not crush. The sifted material may then be sampled and the coarse residue reported separately.

The thoroughness of the mixing of the bulk is very important. If the mixing is perfect, then any small portion taken at random would be representative; in fact, this would be the test of the perfection of the mixing. If materials were well mixed sampling would be greatly simplified, but this is seldom the case. Generally the composition of materials is far from uniform throughout the bulk, and in some cases, owing to the differences in the size and specific gravity of the various constituents, there is a great tendency for them to separate, the fine and heavy material going to the bottom.

To obtain a fair sample of a heap of material it is necessary to take some of the richest, some of the poorest, and some of all the intermediate values of parts, and these, too, in the same proportion as that in which they occur in the bulk. Thus, if there are twice as many rich pieces as poor ones in the bulk, there must be twice as many in the sample, and so for the other different values of pieces. The difference in the size of the pieces is also an important factor, as the fine material is almost invariably different in value to the coarse; sometimes one is richer, and sometimes the other; hence it is also necessary in selecting a sample to have the fine and the coarse in the same proportion as in the bulk.

From this it will be now clear, even to those previously unfamiliar with the rigid requirements of exact sampling, that the work, if done by hand, demands great care and conscientious judgment, besides considerable time and labour.

So much for the theory of sampling.

Assuming that the material, whatever it may be, has been crushed, and that it has been decided what percentage of it to take as a sample, the next question is how best to take the percentage so as to ensure getting a fair proportion of all the various sizes and qualities of the particles making up the bulk?

This brings us to the practice of sampling.

It would be well first to consider the methods of hand sampling, then the question of machine sampling (which will be illustrated by a practical demonstration), and finally will be discussed some general considerations relative to the most efficient and economical manner of dealing with large bulks, referring particularly to chemical manures and fertilisers.

In hand-sampling a cargo or large heap it is customary to take portions from all over the exposed parts. Sometimes the sampler in dealing with the cargo will wait until it is half discharged before making a selection in order to have a larger portion of the heart of the material exposed.

The sampler must exercise care and judgment to take the coarse and fine parts in the correct proportions, and it is doubtless in the exercise of this discretion that mistakes will occur.

Sometimes a sample car load is made up by taking, say one bag or one bucketful in fifty, but however the first sample may be selected the subsequent treatment of it to obtain the final sample is much the same. If the original material is coarse the first sample must be crushed finer and then carefully mixed and quartered on a clean and dry floor. The best method of hand-mixing appears to be to build up a cone by carefully placing each spadeful of material exactly upon the centre of the first spadeful, thus causing the constituents of each spade to flow down all sides of the cone. When the cone is completed it is carefully levelled down into a flat, circular layer a few inches thick, and across this two lines are drawn at right angles to divide up the circle into four equal segments. Two opposite segments are then shovelled out and built up into a smaller cone, which is treated in precisely the same

manner as before. The process of coning and quartering with any necessary further crushing is repeated until the sample is sufficiently small. This method gives very good results if proper care is taken and sufficient time and labour allowed.

Another hand method of reducing the size of the first sample consists in taking lumps from all over the material, which is spread out in a layer a few inches thick, but care must be exercised that in taking the bits the scoop goes right through the layer, otherwise a fair proportion of the finer parts, which generally contrive to get to the bottom, will not be obtained.

Another method often employed in sampling ores, matte, &c., consists in building the material into a rectangular flat heap, which for a quantity of about 100 tons would be from 2 to 3 feet thick. The pile is then opened by shovelling out two channels at right angles across the centre of it, the material shovelled out being thrown upon the rest. From each of the eight faces or sections of the pile thus obtained, is removed a portion by "rising" the shovel from bottom to top, the total quantity thus removed being about 10 per cent. of the pile.

After crushing this sample finer it is formed into a smaller rectangular pile, which may be turned over two or three times, and then channelled and sampled as before.

It is also very important to notice the general regulations for taking samples of fertilisers and food-stuffs which have been prepared by the Board of Agriculture. Clause 13 of these regulations specifies a certain minimum number of bags or packages which are to be taken as a first sample for quantities not exceeding ten hundredweight up to quantities exceeding five tons. In the former case it is two, and in the latter ten.

The size or weight of the bags or packages is not stated, but if they were unusually small the sampler would probably take a larger number of them. It would perhaps be safer to specify for certain percentages of the weight of the total quantity to be taken as a first sample. In clause 15, which refers to the sampling of bulks, no figures are given as a guide to the size of the first sample, nor is any particular mode of selection specified; the whole bulk may or may not be turned over. If it is not turned over and the first sample is made up of outside portions only, the result can scarcely be regarded as satisfactory. To avoid great discrepancies between the sampling of various persons it might be well to specify a certain minimum percentage of the bulk to be taken for the first sample, and also that the bulk be turned over and portions taken before and after turning over; or that the method as described above for ores be adopted.

The method of reducing the first sample to convenient size, as specified in clause 14, is a very old method and on first reading these regulations the author was somewhat surprised that such an old method had not been superseded. It is certainly simple, but, like all methods of hand-sampling, something is left to the personal equation, and this is exactly what it is desirable to avoid if the results obtained by different persons are to agree.

Another drawback to this method is that the material is exposed for a much longer time—see clause 22—than is necessary. There is thus a risk of alteration in the composition of the material, and the representative character of the sample may be lost.

The disadvantages of hand-sampling have given birth to several mechanical contrivances which have been designed to save labour and time and to ensure a greater degree of accuracy than the average of hand work.

Mechanical sampling is based upon the idea of intercepting or cutting out a portion of a stream of material. This may be done either by taking part of the stream continuously or the whole stream intermittently.

Before describing the various systems of machine or automatic sampling which are illustrated in the diagram, it is well to consider what are the essential conditions to be fulfilled by an efficient sampling appliance.

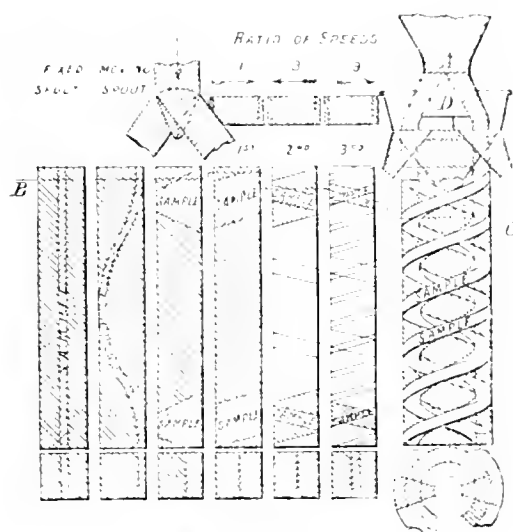
Firstly, it must select with certainty and precision a truly representative portion from the material that is passed through it, independently of the differences of the size and

specific gravity of the individual pieces of material; and secondly, it must not require any skill or care in the operator. It must also be of great simplicity and the possibility of the lodgement of material must be avoided. The machine must be adjustable so that large or small samples may be obtained as required. The machine must be capable of taking independent duplicate samples, that are exactly alike in all respects, and the accuracy of the sampling must not be affected, however imperfectly the material may be mixed.

Refer now to the diagram which represents graphically the methods of cutting adopted in five different systems of mechanical sampling. A short portion of a stream of material is shown in each case with shaded parts representing the portions of the stream removed for the sample, and the portions which are discarded. Figs. 1 and 2 illustrate the action of the fixed and oscillating spouts, which cut out a narrow

# A COMPARISON OF FIVE SYSTEMS OF MECHANICAL SAMPLING.

Fig. 1. Fig. 2. Fig. 3. Fig. 4. Fig. 5.



Section along B-C.

groove of material continuously from a solid stream, but, owing to the irregular composition of the stream and the segregation of fine and coarse material in different parts, the sampling proved unsatisfactory. This resulted some people to conclude that the only way to secure accuracy was by intercepting the whole stream for a part of the time. Several machines were designed to this end, but only in one case apparently was the result secured. Fig. 3 explains the action of several machines, the essential part of which consists in a reversible deflecting plate for diverting the stream intermittently. It is seen that as the time occupied in reversing the deflecting plate is appreciable, more of one side of the stream is intercepted than of the other, and as the two sides of the stream are unlikely to be of equal quality the selection is therefore partial. In the device whose action is illustrated in Fig. 4 the last-named defect is eliminated, for the cutting is not made by a reversible plate, but by a revolving box or compartment which passes periodically through the stream. The portions of material thus selected fall immediately, and consequently in a pulsating stream, on to a second apportioner similar to the former, but revolving in the opposite direction and at three times the speed. If this second apportioner operated upon a continuous stream it would make three actual cuts for each one of the former; but as the stream is pulsating, or intermittent, this result is not secured, and some of the cuts can only be regarded as theoretical and never actually take place. The same



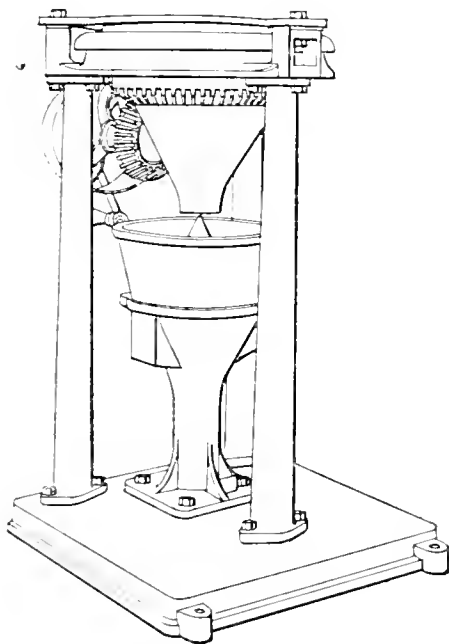
observations apply to the third apportioner, which revolves in the original direction, but at nine times the speed.

The net result is that the ultimate sample is made up of small portions of the original stream, which occur widely apart, and between which a large portion of material of unknown composition is discarded. Another drawback to this machine is the lodgement of material in difficultly accessible places, for the removal of which a strong jet of air or water is necessary. Thus, there is a danger of contamination between different parcels of material. It is also found that the machine does not work with such precision as will enable a definitely required percentage to be cut out with accuracy, the variations above or below the correct weight amounting frequently to 60 or 70 per cent.

All the machines described above are of American origin, but the next one is English.

Fig. 5 represents the essential features and *modus operandi* of a sampling machine used by the author. The upper part of the figure represents the nozzle of a rotating hopper which receives the material. This delivers a solid rotating stream of material at A, which is immediately split upon the point of the cone D, thus becoming a hollow rotating cylinder of falling material. From this annular rotating stream two or more samples are cut continuously by means of segmentally-shaped spouts, which pass completely through the stream. The effect of the rotation of the stream of material is to make each spout cut out a helical path such as shown, and if two spouts are used, as is commonly the case, a double helix results. Each of the cuts is clearly quite independent of the other.

Fig. 6.



ELEVATION.

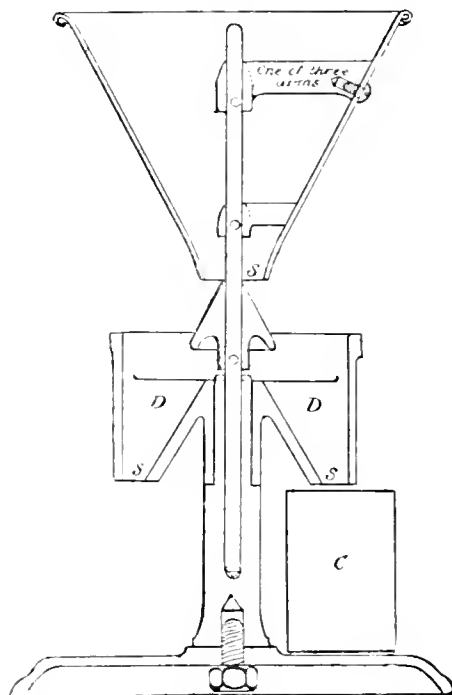
THE LABORATORY DIVIDER.

supported a feeding platform or supplementary storage hopper. In the centre will be seen the cone upon which the stream of material splits. The segmental cutting spouts are hidden from view, but the delivery ends of the spouts appear as projections upon each side of the centre column. The bulk of the material, after the samples have been taken, passes down through the column on to a lower floor or into a wagon. In each machine there are two sets of spouts, one set

The edges of the segments are made truly radial, as shown in the plan, which represents a section on the line B C. It will be clear that the percentage of material removed by each spout depends simply upon the angle between the spout edges: that is, if 10 per cent. is required the angle must be  $36^\circ$ , or 10 per cent. of the circle; and for 5 per cent.  $18^\circ$ , and so on. It is found by actual experiments that if the spout edges are made to these angles, the weights of each of the samples cut out agrees with the calculated percentage with remarkable accuracy. There is no appreciable difference indicated by an ordinary pair of scales between either of the samples and the theoretical percentage, although the variation in the sizes and weights of the pieces of material in the bulk may be great. As a further test of the precision of the apparatus, if the differently sized constituents of each of the duplicate samples are separated by sieves, it is found that there is the same percentage of the various sizes in each sample as in the bulk. Thus, for example, if the original material contains 20 per cent. of sand, 50 per cent. of wheat, and 30 per cent. of peas, then each of the samples will contain 20 per cent. of sand, 50 per cent. of wheat, and 30 per cent. of peas. It would be practically impossible to sample such a material by hand, owing to the unmixability of the constituents, but this creates no difficulty in machine-sampling.

Fig. 6 represents an external view of the machine, with the sample boxes removed. The rotating hopper runs upon a ball bearing, which is supported on three stout columns. The turning of the hopper may be effected by either hand or power, acting through the bevel wheels shown. Above the hopper is a stationary guard ring upon which may be

Fig. 7.



VERTICAL SECTION.

taking duplicates of 10 per cent. each, and the other duplicates of 5 per cent. each. Only one set is in operation at once, but it is contrived that the change from one set to the other can be made almost instantaneously by moving a lever. When 5 per cent. or 10 per cent. is too large a quantity for a sample, it is again passed through the machine, or it may pass through a smaller machine placed lower down. In some cases, where the material is fine and fairly uniform, a

sample of only 1 per cent. can be safely taken at one cut, and if this sample is again passed through, a reduction in volume of 10,000 to 1 is accomplished by the two operations. This can be easily done with the tailings coming direct from gold-stamp mills.

After a sample has been obtained it usually happens that several persons wish to have a portion of it for separate analysis; thus the sample may be divided into three parts, one being for the buyer, another for the seller, and a third for the district analyst. Sometimes six or more distinct samples are wanted, and, in order that the results of the separate tests and analyses may agree, it is clearly essential that all the samples should be identical—that each should have a fair share of the fine and the coarse, the rich and the poor, the heavy and the light particles.

To accomplish this, the divider shown in section in Fig. 7 has been prepared. It is identical in principle with the sampler; that is, the material is formed into a rotating annular stream by means of a rotating hopper and cone, but instead of only taking two samples and discarding the rest of the material, the whole of the stream is divided into six samples by means of the dividing cup D, which is made with six equal segments, delivering by the spouts S into the cases C, one of which only is shown. The *modus operandi* of this apparatus is as follows:—The hopper is set spinning by a touch of the hand, then the sample is thrown in and it is divided into six equal and exactly similar portions in less than the same number of seconds. The economy in time over hand-dividing is at least 98 per cent., and there is the further advantage of precision and cleanliness.

The question of the most convenient way of applying mechanical sampling to various cases will, of course, depend upon the particular conditions of each.

In manufactories, wharves, and mills the machine may be placed at any point where it will intercept a stream of material. In every case it is most desirable to pass the whole bulk through the machine, but where this is not practicable, the first sample (which should be a good size) can be taken by hand and then quartered down in the machine.

Such fertilisers as are made in a semi-liquid form could be, in the author's opinion, conveniently sampled just as it leaves the mixer and falls into the den, and the interposition of a sampler on the annular rotating stream principle would be found to make the contents of the den more uniform. If dry, mixed fertilisers were always prepared upon an upper floor, and then delivered on to a lower one, it would be a great advantage, for the reasons that it could be perfectly sampled while descending on to the lower floor, and at the same time the mixing would be very greatly improved. Against this would have to be put the small cost of elevating the material.

There are many other special cases which time does not permit now to be considered, such as the sampling of grain and foodstuffs, chemicals, ores, and bullion, but these the author would be glad to speak of if necessary.

In conclusion, the author begs to state that he has endeavoured to deal with the subject purely from a practical scientific standpoint, and he ventures to hope that ere long the very important operation of sampling will be lifted out of the rule of thumb into the realm of precision and scientific accuracy.

#### DISCUSSION.

MR. J. CARTER BELL thought this machine would be of great use, now that the Fertilisers and Foods Stuffs Act had come into force. The Board of Agriculture had given directions how these samples had to be taken by dividing them into three parts, and he could not see any better arrangement for sampling manures and seed cakes. He wished that the machine would be able to sample such a material as wood pulp, as it was most difficult to get an uniform sample.

MR. GEORGE E. DAVIS said that in a discussion which took place in this Society some time ago upon discrepancies in sampling and analyses, the feeling was generally expressed that most of the differences between analyses arose from the

varying composition of the samples submitted. If they were so, the introduction of a machine like that before them would be of great service, not only to the sampler, but also to the chemist, who was often blamed without sufficient cause. He had always held that the discrepancies between various analysts would disappear the moment a perfect sample or series of samples could be procured for independent examination. At the same time the sampling machine, to be a perfect one, must be capable of sampling all sorts of materials, large and small, and of every degree of physical condition. It would not do if it could only be used for granular substances, and not for materials of a pasty nature. It would be easy enough to take a sample of, say small pyrites or purple ore, but not so easy to sample by the machine before them a mixture of manure made in a continuous mixer. Then again, such substances as feeding cakes would certainly require some preliminary treatment independent of the sampling machine. Perhaps the inventor would be able to inform the meeting whether these objections were real or not, and if they were real, whether anything had been done to overcome them.

MR. CLARKSON, in reply, said, with regard to pasty materials, the machine would sample any material that could be made to flow. The cone upon which the stream divides was mounted upon a screw. The cone could be very readily raised, and there was only a very thin stream all round, and by this means they could sample oils, &c. When the pasty substances could not be made to flow, they could be forced by means of a ram. Jewellers' sweepings were ground up to the consistency of thick slime, and this was very difficult to sample, inasmuch as it contained little shots of metal which could not be screened out; but by the fixing of two steel blades, which came down to the rotating copper, satisfactory results were obtained. It would not be difficult to sample wood pulp in a granular form, but in breaking it up the percentage of moisture would be rather altered. It all depended a great deal upon the form of the material. He did not see how anything better could be done than the method adopted by Mr. Carter Bell, inasmuch as if it could be made to flow, it could be readily tested. The method adopted in Antwerp for sampling silver ore bars, was to saw each bar across the middle with a circular saw, and from 25 tons you would probably get 180 lb. of sawings of various sizes and weights. It was the custom formerly to cut them down in the usual way, and this took four or five men three-quarters of an hour to do properly. They tried it in one of these machines and found that the sawings of 25 tons could easily be cut down in 2½ minutes.

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F. Clowes.	H. J. Staples.
J. B. Coleman.	C. Taylor.
C. H. Field.	G. J. Ward.
H. Fortin.	

Treasurer: J. T. Wood.

Hon. Local Secretary:

R. L. Whiteley, University College, Nottingham.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

SESSION 1893-94.

Meeting held February 11th, 1894.

MR. J. M. C. PATON IN THE CHAIR.

## FERMENTATION IN THE LEATHER INDUSTRY.

BY J. L. WOOD.

THE leather industry can fairly lay claim to be one of the oldest of all the old arts or crafts. It can truly be said that its beginnings are "lost in the mists of antiquity."

Perhaps one of the earliest references to tanning or dressing of leather is to be found in the *Iliad* of Homer (xvii. 382—393).

He is describing the struggle of the Greeks and Trojans over the body of Patroclus; how they tug—

\* As when a man  
A huge oxhide drunken with slippery lard  
Gives to be stretched, his servants all around  
Disposed, just intervals between, the task  
Ply strenuous, and while many straining hard  
Extend it equal on all sides, it sweats  
The moisture out and drinks the unction in.\*

The process is very similar to that used by savage tribes at the present day, and the description explains almost exactly the principle by which oil or chamois leather is dressed by modern methods.

There are many references in the *Iliad* to leather, or perhaps, to speak more strictly, rudely dressed hide, used in the making of shields. These were of seven thicknesses of tough bull's hide, and made by a clever worker in leather, whose name Homer has handed down to us as Τυχίος σκυτοδόμος δὲ ἄριστος.† Aristotle (Knights, 868—893) makes Demos complain of the abominable smell which emanated from the tanyard of Clon, the sole leather tanner. Other ancient references to leather will be found in Herodotus (v. 58) and in the Bible.‡ Thus more than 3,000 years ago the tanner's art was practised, doubtless in a very similar manner to that which prevailed up to comparatively modern times.

Tanning has, like all ancient industries, become perfect in the course of ages by pure empiricism. The practical man, warned by failures in one direction or another, learned by sheer practice that method which yielded the best results, without troubling himself about the why and the wherefore of it. But the "rule of thumb" processes of tanning have shown themselves in the light of scientific investigation to be of a more complex nature than almost any other industry, hence the difficulty of successfully applying scientific methods to improve on the well-worn processes slowly and painfully evolved during centuries of practice.

To most people the word fermentation is so intimately associated with the manufacture of beers and wines, that it may seem strange it should have anything to do with such a dissimilar industry as tanning. We shall see, however, that fermentation plays a very important part in that industry. I shall not attempt to do more than indicate the present state of our knowledge of the different fermentations which occur in the manufacture of leather, briefly touching on the principal points, and giving references to the latest researches.

Looking up work already done in any subject, especially under modern conditions of a multiplicity of scientific journals and specialisation of subjects, occupies a considerable amount of time. The work, however, must be done, or frequently after months of experiment one finds that some other worker has been over the ground perhaps years before, and that the time we have spent is practically wasted. It is mainly with the object of helping in some degree in this way that I have ventured to collect these

few references bearing on fermentation in the leather industry.

Fermentation, as it affects the tanner, may be divided into two classes:—

(1.) Fermentations which are injurious, and which it is desirable to prevent.

(2.) Fermentations which are essential or helpful, and which it is desirable should go on in a regular and certain manner.

Everyone is aware that after a skin leaves the animal's back it begins to decompose (unless precautions are taken) in a time long or short, according to the temperature.

We may call this the first fermentative action met with in the leather industry, for it is very difficult to draw any hard-and-fast line between the processes of putrefaction and fermentation as usually understood. Putrefaction is always accompanied by the presence of micro-organisms, but fermentation may be due to the presence of unorganised ferments or enzymes.

Either\* considers that a specific putrefactive ferment attacks each constituent of the hide, all the putrefactive organisms utilising the easily soluble albuminoids of the skin as nutrient material. Blood and lymph are the constituents of the fresh hide most liable to putrefaction, and as the germs of putrefactive bacteria are always present in the air, they begin to develop in these materials. Although the putrefaction commences from both surfaces of the skin it is a curious fact that it begins usually on the "grain" side, notwithstanding that this is protected by the hair. The bacteria obtain access by the sweat pores and begin to liquefy the layer of cells between the epidermis and the grain; on the flesh side they enter by the blood vessels and lymphatics, and along these the putrefaction is quickly communicated to the interior of the hide. The intercellular substance or corium and the hide fibres are more stable bodies, but finally the putrefaction spreads from the vessels to the corium, causing it to liquefy; and last of all the hide fibres themselves are attacked and the skin becomes an amorphous semi-fluid mass.

The subject of putrefaction is a large one, the number of bodies formed being very numerous, while the several species of bacteria which cause the decompositions have been little investigated. The drawings, Fig. 1, show bacteria and organisms observed by the author in putrefying skin in 1886. The most constant products† which make their appearance in putrefaction are *leucin*, and probably some of its homologues, tyrosin, the volatile fatty acids of the series  $C_nH_{2n}O_2$  (formic, acetic, propionic, butyric, valeric, caproic, &c.), ammonia, some of the compound ammonias (ethylamine, propylamine, amylamine, trimethylamine)  $CO_2$ ,  $H_2S$ ,  $H$ , and  $N$ .‡

Of course the object of the tanner is to preserve the skin from the action of these destructive ferments. If it is not to be tanned immediately, the simplest methods of preservation are either drying, or the application of salt. In both these cases the bacteria are not destroyed, their action is merely arrested. In hot countries the hide must be preserved from putrefaction while drying, and for this purpose various antiseptics are used, for example, sodium arsenate, a solution of which is applied to the skin before it is stretched out to dry. These skins form the "arsenic kips" of commerce.

In the commercial application of antiseptics, the chemist has proved a valuable ally to the tanner, and of late years, numerous compounds have been introduced for this purpose.§

\* Der Gerber, xv. 3, 37.

† Vide Precher, Text Book of Tanning, 1885, 49.

‡ Liebig, Annual Chemistry (1831), 109. Pasteur Compt. Rend., June 1863. Schützenberger, Fermentation, 1876, 222. Salomonsen, Studien über die Faulnis des Blutes, 1877. Tyndall, Floating matter in the air, 1881. Hauser, Faulnis bacterien, Leipzig, 1885. Eimer, Der Gerber, xv. 337. This Journal, 1890, 401; 1893, 616. Baerlein, Ding. Polyd. J., 288; 211, &c. Von Schroeder and Passler, Ding. Polyd. J., 269, 137, &c., also this Journal, 1894, 49. Zerkow-Gerber, Archiv. F. Hygiene, xvi. 1. Centrallblatt, für Bacteriol. xiv., 111.

§ Some of the antiseptics used in tanning are sodium arsenate, crocin, mercuric iodide, mercuric chloride, carbolic acid, salicylic acid, oxynaphthoic acid, cresotic acid, trioxiphenyl sulphonic acid, pyrocatechol sulphonic acid, carbon bisulphide, and calcium Glabner's salts (sodium pyrosulphate).

\* Ὡς δ' ὅτ' ὄντην ταύροις βοὺς μέγαλον βοεῖην  
λαοῖσιν δάη παν εἶν, μεθύουσιν ἀλοιφῇ.  
Δεξάμενοι δ' ἄρα τοῖ γε διαπαντες τανύσσιν  
Κυκλῶσ', ἄραρ δέ τε ἰκμάς ἔβη δυνεῖ δέ τ' ἀλοιφῇ  
Πολύων ἑλκόντων, τάνυται δέ τε πάσα διαπρό.

† Il. vi. 219.

‡ Acts, ix. 43; x. 6.

The value of many of these different antiseptics has been admirably discussed by Eitner (*Antiseptik in der Gerberei*, *Der Gerber*, xv.), to whose work I must refer you.

The "soaks" are naturally rich in putrefactive ferments, indeed in the summer time it frequently happens that dried skins cannot be thoroughly softened before they begin to putrefy and lose weight.

Here certainly the careful use of some antiseptic may be made the means of controlling their working and of saving a large amount of loss to the tanner. Creolin (a coal tar product distilling over between 170° and 280°) is a material easily and cheaply applied to check the action of bacteria which may be present naturally in the water used by the tanner, or whose presence is due to contamination from drainage.

Fig. 1.

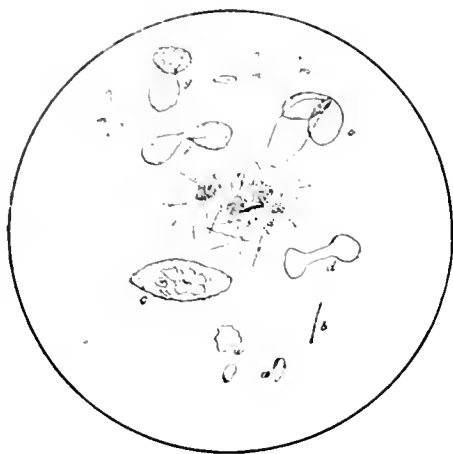


Fig. 1.—Shows organisms observed by the author in putrefying skin in 1886.

a. Swift, moving, darting animalcule, some with irregular outline.

b. A vibrio quite straight and moving very slowly,  $\times 500$ .

c. A large infusorian.

d. A colourless piece of jelly shaped like a dumb-bell. All these appeared in three days in skin placed in a little water, temperature about 20° C.

Five days.—Most striking feature was the increase of vibrios, besides many large infusoria.

e. Large infusorian, apparently double, slow rolling motion.

f. and g. The same in different positions.

Seven days.—Still more vibrios, liquid crowded.

Ten days.—Vibrios clustered round a small particle of matter, whole mass of skin disappeared.

m. Very transparent trembling animalcule, with short darting motion, in large numbers.

From the "soaks" the skin passes to the "limes." It would naturally be thought that here at least it would be safe from the action of destructive bacteria. This, however, is not always the case, for although freshly-made lime liquor or milk of lime is an antiseptic,\* yet it is capable of dissolving the corium of the hide, and as soon as it has taken up a sufficient quantity, which combines with it to form ammonia compounds, scarcely a better medium could be found for the nutriment of certain organised ferments.

It is not, however, until the lime gets really old or putrid that it has a destructive action on the skin; on the contrary the bacteria in limes have a beneficial effect up to a certain point. It is well known that a newly-made lime

liquor is too harsh and sharp in its action, especially on "green" skins, i.e., skins with little or no lime in them.

After a time it becomes what is known as "mellow," in which condition the fibres exert a more favourable reaction on the fibres of the corium, and at its whole substance wears a new firmness, and the surface of the skin appearing to close the pores and thus prevent the rapid effect. This "mellowness" has also a great influence on the action of the bacteria, and it is of great importance to know how far this mellowing process of the skin may be carried. I have been several times investigating their behaviour and properties, in a work yet to be published.

In the next process, that of "bating," the bacteria play a more important part, and may be said to form a second category, viz., helpful to the tanner, though from the very nature of the material used, it forms a nidus for various putrefactive ferments, and is therefore dangerous. The materials usually employed are boiling and dog-bone; the former of these is sharp and piercing in its action, while the latter has a more softening action on the skin. In both bates, however, a rapid solution of the gelatinous and albuminous hide substance (corium) takes place. The hide fibres, however, are not attacked until all the nutrient material in the liquid has been consumed by the bacteria.

Eitner (*Der Gerber*, xv., 158) states that a sterile bate is without action on the skins, and appears in that article to attribute the entire working of the bate to the action of micro-organisms. He seems to have based his conclusions on experiments with an old bate sterilised with creolin, which he found had no action on the skins. If, however, a fresh bate be made and boiled for half-an-hour, then allowed to cool to 90—95° F., it will be found to have a considerable action on the skins, though not so rapid a one as the unboiled bate. The boiling kills all organisms, and fresh ones have not time to develop from spores still remaining before the experiment is at an end.

Lately, the action of unorganised digestive ferments in the dung has been found to be considerable. This bating is an exceedingly complex process. The action appears to be threefold:—

(i.) A purely chemical action of the soluble salts present in the bate on the lime in the skin.\*

(ii.) An action due to organised ferments.

(iii.) An action due to unorganised ferments.

The precise amount of influence each of these actions has on the skin is difficult to assign. The chemical action of the ammonia compounds dissolves the lime remaining in the skin, but the simple removal of the lime is not sufficient, as may be shown by removing it completely with dilute hydro-chloric acid or other similar means, and washing perfectly free from acid in distilled water. When tanned such a skin is hard and brittle.

The organised ferments or bacteria, of which there are many species in the bate, probably act on the skin by secreting soluble ferments which have the power of dissolving hide fibre. I have isolated in plate cultivations several species which liquefy the gelatine.

If skins are allowed to lie in the bate, zoogloae of these bacteria collect in the folds and attack the fine "grain," so that the leather is covered with lines and markings or "flaked." Thus the operation is a very critical one, requiring careful watching; under certain atmospheric conditions and at a temperature of 35—40° C. the skin, if only left a short time too long, will completely melt away. The more skin substance is dissolved in the liquid, the more rapid and pronounced the bacterial action becomes.

\* *R. Eitner's* *Antiseptik*.

Practitioner, Text-book of Tanning, 1887, 1891.

*Der Gerber*, x, 1884, 1897, 1900, 1901.

xv., 188, 1267.

J. Anal. and Appl. Chem., 1893, 7, 579.

This Journal, x, 539, 1900, 1901, 1902, 1903.

Science, W. J. Tech. Quarterly, 1892, 5, 81, 82.

This Journal, xii, 77, 1902.

\* One kilo. of lime (prepared from marble and slaked) in 20 litres of water has been found to destroy the bacteria of swine fever, hen cholera, and glanders.

So far as is at present known the unorganised ferments in the dung, besides those which are secreted by the bacteria, are mostly digestive ferments\*—pepsine, pancreatine, and trypsin, certain quantities of which pass out of the animal's body in an unchanged condition. Of these *pepsine* only acts in an acid solution, and although fresh bate liquor is faintly acid to litmus it quickly becomes neutralised by the lime in the skins, so that the action of this ferment can only be limited in extent. *Pancreatine*

will act in a neutral solution, and has, therefore a considerable effect on the skin.

I find by experiments with the purified ferments that their action is very slow compared with the bate itself. Two portions of the same skin were taken, one of them was treated with a 1 per cent. solution of pepsine acidified with 0.2 per cent. of hydrochloric acid; the other in a bate liquor of dog's dung, both at a temperature of 40° C. (104° F.). At

## SOME DUNG ANALYSES.

Hen Dung.		Dog Dung.		Guano.	
	Per Cent.		Per Cent.		Per Cent.
Moisture .....	60.88	Ca .....	43.049	Urea .....	5.00
Organic Matter† .....	19.22	Mg .....	0.087	K <sub>2</sub> SO <sub>4</sub> .....	7.90
Phosphates .....	4.47	K .....	0.392	NaCl .....	5.00
CaCO <sub>3</sub> and CaSO <sub>4</sub> .....	7.85	N .....	0.138	(NH <sub>4</sub> ) <sub>3</sub> PO <sub>4</sub> .....	5.50
Alkaline Salts .....	1.09	Si .....	0.004	(NH <sub>4</sub> ) <sub>2</sub> O .....	0.60
Silica and Sand .....	6.69	PO <sub>4</sub> .....	3.146	SiO <sub>2</sub> .....	2.25
	100.00	CO <sub>2</sub> .....	7.464	Ca <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub> .....	9.00
		Cl .....	0.037	MgNH <sub>4</sub> PO <sub>4</sub> .....	15.25
		Fe and loss .....	0.008	Ammon. Urate .....	15.25
		Organic matter .....	11.152	Organic Matter (17 per cent. N) .....	41.73
		H <sub>2</sub> O .....	31.013	Moisture .....	6.52
			100.000		100.00

\* Containing N = ammonia 0.74.

W. I. Macland, This Jour., 1888, 86.

Viellot Diction d'Analyses Chim.

The dog dung actually used in a tannery is from animals fed on a more vegetable diet, the one given having an extremely high percentage of lime, no doubt owing to the animal having eaten bones. A dung as brought from the kennels was found to contain:—

	Per Cent.
Mineral Matter .....	4.679
Organic .....	9.731
Water .....	85.590
	100.000

the end of one hour the skin in the pepsine solution was considerably "fallen," but that in the manure solution was bated nearly away, i.e., the greater part of it was dissolved.

A 1 per cent. solution of pancreatine (Merck's) was found to act far more rapidly than pepsine. At 10° C. in a neutral solution the skin fell rapidly, and the action continued even in the cold. In this experiment it was found that in 15 hours the liquid was swarming with minute bacteria. In order to guard against the influence of bacteria a similar experiment, at the suggestion of Mr. H. R. Procter, of the Yorkshire College, Leeds, was tried with the addition to the pancreatine solution of 1.5 per cent. of chloroform; this prevents the development of bacteria, while it does not interfere with the action of the pancreatine. The skin was reduced as before, but in neither case had it the peculiar touch of a "pured" skin, nor were the characteristics of the leather the same.

There is a wide field of research open in the chemistry and bacteriology of bating, but from the present state of our knowledge it is quite evident that it is well nigh impossible to produce commercially an artificial bate which shall possess similar properties to the ordinary dung bate.† It

will be found that most of the substitutes proposed have fulfilled only one of the conditions, and none of them have combined properties which are evidently essential.

The next fermentive process, viz. "Drenching," has been described and investigated by myself (this Jour. IX., 27), and more recently in conjunction with Mr. W. H. Willecox, B.Sc. (this Jour. XII. 422), and the work is still being continued. I shall, therefore, pass on to the next process the skin goes through, viz., the tanning process proper.

From the drench (which is, however, only used for certain classes of leather) the skin usually passes directly into the tan liquor, and carries with it vast numbers of fermentive organisms, so that it might naturally be supposed some kind of fermentation would result. This is so, for during tanning infusions of nearly all vegetable tanning materials undergo fermentation, with formation of a little alcohol, gallic, butyric and lactic acids accompanied by evolution of carbon dioxide. In the early days of scientific investigation in the tannery, Pelouze\* attributed these changes to the oxygen of the air, and even so late as 1892 this theory received support. F. G. Reissinger (*Deutsche Gerber Zeitung*, xxxii. 93) endeavoured to show that moist pine bark absorbed oxygen with the production of volatile acids. Eitner and Jahoda (*Der Gerber*, xviii. 245) showed that the absorption of oxygen was a mechanical one, due to the resinous matter of the bark, and that the volatile acids were produced by the action of bacteria.

It was naturally supposed that the bacteria decomposed the tannin itself, forming acids and gases. Tannins were

\* Harris & Gow., *Jour. of Physiol.*, xiii., 429. G. Tanninm., *Zeitschr. f. Physiol. Chem.*, xvi., 271. O. Loew., *J. Prakt. Chem.*, 37, 101. This Jour., 1888, 224.

† A few substitutes for the bate which have been tried are: the glucose bate, *vide* Procter's "Text Book of Tanning," ed. 1885, 187; American patent bate (*Der Gerber*, 1887, 257); cresotinic acid (oxytoluic acid); Nesbit's process (described *Der Gerber*, 1887, 113); Borol; Pique Gras (*Der Gerber*, 1889, 87, 90). For list of American bate substitutes, *see* Davis, "Manufacture of Leather," Philadelphia, 1885, pp. 335-351.

\* Ann. Chim. Phys. 1834.

known to be glucosides, and as such easily capable of fermentation. But in 1886 B. Kohnstein and Simand (Der Gerber, xii., 253) showed that these acids are not produced from the tannin, but are the result of a fermentation of the extractive matter\* which accompanies the various tannins. In order to prove this they absorbed the tannic and gallic acids and colouring matter of a tan liquor by means of freshly ignited magnesium oxide. They sterilised the liquids by boiling, and inoculated the clear filtrate, which contained the extractive matter but no tannin, with a lactic acid ferment, at the same time inoculating the original liquor with the same ferment. The liquids were kept on the incubator for 14 days. At the end of this time they found the amount of tannin in the original liquor practically unchanged, while both liquids contained a considerable quantity of acid. More acid, indeed, was produced from the liquid free from tannin than in the one containing it. They also noted that, although moulds developed freely in an exposed infusion of tannin (oak bark) in a day or two, they did not develop in the liquid deprived of tannin, even after eight days.

In 1889 the author compared a sumac infusion exposed to the laboratory air with a similar infusion sterilised and exposed to filtered air. In the first the common mould *Penicillium Glaucum* quickly developed, and in 23 days 80 per cent. of the tannin was destroyed. In the protected infusion after 23 days the total amount of tannin was 0.3 per cent. less than at the beginning of the experiment. This loss was no doubt due to the boiling (several times) required to sterilise the liquid.

In contradiction to the experiments of Kohnstein, two French chemists, Messrs. Collin and Benoit, in 1888 endeavoured to show that unsterilised hide always caused fermentation of the tan liquors, changing the tannin into gallic, butyric and lactic acids. They proposed to prevent this fermentation by the addition of minute quantities of mercuric iodide to the liquors.

Eitner, criticising their conclusions (Der Gerber, xv., 1, &c.), denies that the bacteria of unsterilised hide have the power to decompose tannin.

He added decomposing blood albumen to a tannin infusion, and found that only the tannin actually combined with the albumen, and thus precipitated, was removed from the solution, and that when moulds were excluded, the remaining tannin did not disappear, a proof that the putrefactive ferments in decomposing blood exercise no influence on the tannin. In hardly any instance do Eitner's experiments corroborate the statements of Collin and Benoit, indeed it seems certain that these bacteria instead of destroying the tannin actually preserve it, as moulds, which easily decompose tannins, refuse to grow in the presence of the bacteria.

The fermentation of tannin is not, however, due to moulds alone, several species of saccharomyces or wild yeasts attack tannin infusions, and I have isolated in a plate cultivation from sumac infusions a micrococcus which ferments them. This organism grows well in peptone and sugar solution, but will not grow in beef broth. I strongly suspect this to be the organism which ferments sumac and gallnut infusions with formation of gallic acid and evolution of CO<sub>2</sub>. Infusions of these materials ferment and lose their tannins very quickly without the presence of moulds or saccharomyces.

As far as I know, none of these organisms and their products have yet been investigated, and as different tannins ferment in different ways, some being susceptible to fermentation, like pine and oak barks, and divi-divi, others difficultly fermentable, like larch and hemlock, so the amount of work to be done before all these changes are understood is enormous.

We may conclude, however, that the presence of micro-organisms in tan liquors is essential to the proper progress of the tanning process, (1) by causing the formation of acids which are essential for many leathers; (2) by prevent-

ing the development of moulds. Besides those in the text, I give at foot<sup>1</sup> references to other researches on this subject.

Fig. 2.



Fig. 2.—Shows organisms observed by the author in tannin infusions (principally sumac) in 1886—7.

a. *Penicillium glaucum*.

b. Various saccharomyces.

c. Pale green transparent organism with longitudinal revolution, on one end coming in contact with one cell of c the whole organism trembles; the other end continuing to revolve until the filament breaks in two, forming two separate organisms d, d, which after a short time appear filled with granules, and show one or two nuclei. These rotate slowly after the fashion of volvox, and attach themselves to the cells c, assuming then a rapid vibratory motion. They seem fastened by a fine thread, and then struggle to detach themselves.

d. Another transparent organism undergoing changes on coming in contact with the granules c.

g and k. Bacteria and micrococci  $\times 600$ .

It is a cause for reproach to English tanners that most of these are by German and Austrian chemists; I am glad to say that we are now endeavouring to regain lost ground, and that the work now being carried on in the Leather Industries Laboratory at the Yorkshire College, Leeds, under the able management of Professor H. R. Procter, will, ere long, give us a place in the front rank of all nations.

#### DISCUSSION.

Mr. CARLELLA inquired if Mr. Woods had ever used a South American wood known as Quebracho, said to contain as much as 19 per cent. of tannin?

Mr. CLOWES remarked that in one of his earlier communications Mr. Wood had spoken of a certain organism which he had detected, and of the identity of which he was not certain. He would like to know if, upon further examination, the organism had proved to be a new one, or whether it had proved to be one already known. He believed Mr. Wood had had some communication on this matter with other gentlemen who were working in connection with micro-organisms; and numbers would be interested to know what the results of those communications were. The majority of the members present were placed in a very difficult position, as they knew but little of the work placed before them by Mr. Wood. Not only were they unfamiliar with the processes of the tanning industry, but they also were not in a position to learn the newer ideas

\* A loose term for soluble bodies accompanying tannins, some of which reduce Fehling's solution.

<sup>1</sup> Der Gerber, x., 1884, 54, 102; XI., 1885, 98, 112; XIII., 1887, 245; XVIII., 1892, 215. Dine Polyt. Journ., 256, 38, 42, &c. H. Trimble, Index to the literature of the Tannins, in 'The Tannins,' Philadelphia, 1892.

with regard to those processes which Mr. Wood had placed before them. That gentleman had done the section a great service in a highly summarising, as he had done, the new processes, and also in giving them references to the papers in which the different results had appeared. This communication would be very useful to those who wished to take up research work in connection with tanning. It would enable those who wished to get the latest information to acquire it with little trouble, and thus to avoid undertaking work only to find that it had already been done. As to the work which Mr. Wood said was being done at the Leeds College under Mr. Procter, he regretted that similar facilities were not afforded at the Nottingham University College.

The CHAIRMAN said it was Mr. Wood's good fortune to frequently visit the Continent, so that he was constantly coming in contact with the professors interested in these experiments, and the section were very fortunate in having him before them to give them his experiences.

Mr. Wood, replying to Mr. Canalia's question, said that a large quantity of Quebracho was used. It was used more on the Continent, however, than in England. The wool was estimated to contain about 24 per cent. of tannin, but it was so hard that special machines had to be used for cutting it up into shavings, so that the extract was in more general use. The extract contained about 45 per cent. of tannin.

With regard to Dr. Clowes' question about the bacteria to which he had made reference at a previous meeting, he had had some conversations with Dr. Frankland at the British Association meetings in November, as to its identity, and that gentleman advised doing the work by another method. It was now in a pretty forward state; he was working upon it in conjunction with Mr. Wilcox.

With reference to the Leeds Leather Laboratory, it was assisted by the Leeds Leather Trades' Association, besides a grant by the college. There were many more tanners there than in Nottingham.

The CHAIRMAN drew attention to the fact that it had been decided that members of the section should be at liberty to place interesting questions upon the agenda paper for discussion at the meetings. He hoped that good might result from the movement.

Meeting held at the Queen's Hotel, Leeds, on Monday, March 5th, 1894.

PROFESSOR ARTHUR SMITHHELLS IN THE CHAIR.

## ON THE MORDANTING OF WOOL WITH CHROMIUM. PART III.

By L. LIEFCHT AND J. J. HUMMEL.

(See also this Journal, 1893, 240 and 332.)

Among the various chromic oxide salts the first to be considered is chrome-alum,  $\text{Cr}_2(\text{SO}_4)_3 \cdot \text{K}_2\text{SO}_4 + 24\text{H}_2\text{O}$ . Although seldom or never used by woollen dyers, this salt has long been employed by calico-printers, who already, more than 40 years ago, recognised the necessity of applying chromium in some other form than that of potassium bichromate, wherever, indeed, the oxidising property of the latter was found to be injurious, or was not beneficial. At one time a very expensive chemical, and consequently excluded from use in practice, it has now for many years been the custom of the calico-printer's colourist to prepare chrome-alum by reducing potassium bichromate in the presence of sulphuric acid, with glucose, starch, glycerin, &c.

As early as 1851 C. Koechlin made experiments with chrome-alum, not only as a mordant for cotton, but also for wool. His results appear to have been very unsatisfactory, for he says: "L'alun de chrome est le moins convenable des sels pour fournir l'oxyde. Le nitrate donne de meilleurs résultats." For wool (probably woollen printing is meant) he recommends the use of chromium acetate with the addition of one equivalent of oxalic acid; he finds the oxalate and the nitrate to be the best mordants for wool.

Gehe recommends chrome-alum in place of bichromate of potash, because, he says, it is cheap, does not make the wool hard and brittle, and it gives level colours which do not rub off so much. Peisert also prefers chrome-alum to bichromate of potash, because he finds it to yield more level colours, and he recommends it to be used with the addition of a little sulphuric acid. Jolet, too, recommends chrome-alum for wool, and states that the fibre absorbs the salt as such, not as a basic salt or as a hydrate. He considers that the salt is decomposed in the dye-bath only, for although the chrome-alum is not washed out by cold water it can be removed by hot water. The addition of cream of tartar he deems to be unnecessary, although it is absorbed unchanged by the wool to the extent of one-fourth the weight of the chrome-alum employed. Curiously enough Jolet says that chrome colours are not very fast to light. Gauswilt and Kertesz recommend the use of cream of tartar as an assistant. Depierre considers all chromic oxide salts to be inferior mordants to bichromate of potash. According to Herzfeld green solutions of chrome-alum give up more oxide to the fibre than violet solutions, and he finds it desirable to employ large amounts of cream of tartar. Kay and Bastow find, on comparing bichromate of potash with chrome-alum as mordants, using equivalent amounts, that the latter gives up to the fibre twice as much  $\text{Cr}_2\text{O}_3$  as the former; they also observe that by employing larger amounts of chrome-alum, e.g., 20 per cent. instead of 10 per cent., less chromium is fixed, e.g., 53.07 per cent. instead of 63.87 per cent.; and with the use of 3 per cent. tartaric acid the amount fixed is reduced as low as 30.7 per cent. Koepp enumerates the following advantages in connection with the use of chromic oxide mordants (referring no doubt to the use of chromium fluoride): the wool preserves a soft handle and is readily wetted out and there is no fear of over-chroming. Knecht, Rawson, and Loewenthal consider that part of the chromium sulphate is probably simply absorbed by the wool fibre and is not fixed in a permanent manner, and thus account for the poor results obtained in dyeing. Cochenhausen recommends the use of 4 mols. cream of tartar; Witt also recommends tartar, but does not find chrome-alum to be an advantageous mordant. Previous experiments in the Yorkshire College dyehouse have shown that cream of tartar, tartaric and oxalic acids are all useful assistants with chrome-alum.

## Yorkshire Section.

Chairman: A. Smithells.

Vice-Chairman: T. Fairley.

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J. E. Bedford.	C. Rawson.
F. Branson.	F. W. Richardson.
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J. J. Hummel.	G. W. Shutter.
J. Lewkowsitch.	G. Ward.
W. McD. Mackey.	T. B. Wilkinson.

Hon. Local Secretary:

H. R. Procter, Yorkshire College, Leeds.

Notices of Papers and Communications should be addressed to the Hon. Local Secretary.

### SESSION 1893-94.

April 2nd:—

Messrs. Hummel and Perkin. "The Tinctorial Properties of some Indian Dyestuffs." Part I.  
Annual Meeting for Election of Officers.

May 7th.—Mr. H. R. Procter. "The Qualitative Recognition of Tanning Materials."



Such is a very brief *résumé* of the published references to chrome-alum as a mordant for wool.

The difficulties connected with the subject are not inviting, and yet further study seemed to us desirable, especially when it is remembered that bichromate of potash baths are only exhausted to the extent of one-third, and because a good non-oxidising chromium mordant is required for all these colours which are in any way sensitive to oxidising influences, *e.g.*, wooded colours, alizarin blue, &c.

Further, it appeared interesting to learn, if possible, more regarding the reactions which occur during the mordanting of wool, about which we still know so little, and which probably cannot be fully explained until we have a clearer knowledge of the chemical constitution of wool.

#### A.—CHROME-ALUM. $\text{Cr}_2(\text{SO}_4)_3 \cdot \text{K}_2\text{SO}_4 + 24\text{H}_2\text{O}$ .

The differences in the opinions expressed regarding the value of chrome-alum as a wool-mordant are due no doubt to two causes:—first, the different percentages, reckoned on the weight of wool employed; second, the concentration of the mordanting bath and the quality of the water. Here too, as in the case of the bichromate of potash mordant, little or no attention has been paid to the ratio which ought to subsist between the amounts of colouring matter and mordant employed.

In the following experiments 10 per cent. chrome-alum was usually taken, *i.e.*, about equivalent to 3 per cent. bichromate of potash. For each trial 10 grms. wool and 600 cc. Leeds water were employed.

**Experiment 1.**—Wool was mordanted  $1\frac{1}{2}$  hours with 10 per cent. chrome-alum. As the temperature of the bath rose, the colour of the solution gradually altered, the violet tint changing to green. This of course was not surprising, for it has long been known that on heating solutions of the violet modification of chrome-alum to a temperature of  $55^\circ$ – $65^\circ$  C. it changes into the green modification. If the solution was heated without any wool being immersed in it, the same colour changes were noticed, and yet a distinctly yellowish-green was exhibited by the solution containing the wool. Both baths remained clear to  $80^\circ$ – $82^\circ$  C., but at this temperature the bath containing wool began to show signs of turbidity, which soon rapidly increased so that the solution became quite opaque. The precipitate was excessively fine and had by no means the ordinary flocculent character of chromic hydrate. Now it has long been known that wool has the property of absorbing or attracting in a mechanical manner very finely divided insoluble matter in suspension, and this feature was strikingly shown in the present instance. Before the appearance of the turbidity the wool scarcely showed even traces of any green tint, but from this point it rapidly acquired a yellowish-green colour. At a temperature of about  $95^\circ$  C. the solution became clear again, and, after boiling a little, it was perfectly limpid and almost colourless, the wool having mechanically fixed nearly the whole of the precipitate. When the bath was allowed to cool, it contained only a small quantity of a pale green precipitate, and if the wool was kneaded and pressed in the liquor with the fingers a little of the adhering precipitate could be removed from it; still even with vigorous washing the major portion still remained attached to the wool, colouring it a pleasing shade of yellowish-green. The chrome-alum solution not containing wool gave no precipitate even after long boiling, and remained clear when cold.

**Experiment 2.**—The same experiment as the foregoing was repeated in every detail except that 10 grms. bleached cotton was employed instead of wool. In this case the bath remained perfectly clear both when boiling and when cold, and after washing, the cotton appeared perfectly white.

**Experiment 3.**—The same experiment was again repeated, this time using 10 grms. boiled-off silk. Here the same appearances were noticed as in the case of wool, although the decomposition of the chrome-alum was not so great, and the green colour of the silk was less intense than that of the wool.

In order to see if the silk was really mordanted properly or not, a portion was dyed with 12 per cent. alizarin (20 per cent. paste), when a fairly good claret colour was obtained.

The wool of experiment No. 1, and other similar pieces, were dyed with alizarin, logwood, and quercitron bark, but in each case a very poor and practically useless result was obtained.

An attempt was now made to determine analytically the nature of the change which had occurred. The waste liquors of experiments 1, 2, and 3, were analysed, No. 1 being of course filtered. The waste liquor of No. 2 was absolutely unchanged, it still contained the whole of the chromium in the form of chrome-alum.

With respect to the waste liquor of No. 1, however, the average of ten determinations showed that only 14.3 per cent. of the original chromic oxide remained still in solution, so that 85.7 per cent. was partly fixed on the wool and partly in the precipitate. On repeating the experiment with the addition of 10 per cent. sodium chloride to the bath, the decomposition was quite complete, for the waste liquor proved to be entirely free from chromium. Another experiment in which only 5 per cent. chrome-alum was employed, also showed mere traces of chromium in the filtered liquor. A certain relationship seems therefore to exist between the amounts of wool and of chrome-alum, so that, as already stated, when using 5 per cent. of the latter total decomposition is produced, while with 10 per cent. it is affected only to the extent of 85.7 per cent.

The precipitate adhering to the wool was removed as much as possible by kneading with water, then collected on a filter, washed thoroughly, and analysed. It proved to be very difficultly soluble in acids, and was therefore changed into sodium chromate by boiling with an alkaline solution of sodium hypochlorite, then acidified and reduced with alcohol. Analysis showed the precipitate to be a very basic chromic sulphate, the actual results obtained in four examinations being as follows:—

1.  $1(\text{Cr}_2\text{O}_3) + 0.836 \text{ SO}_3$
2. " + 0.819 "
3. " + 0.538 "
4. " + 0.360 "

The varying composition here shown is not surprising, and it is no doubt caused by the different lengths of time taken to heat the bath up to the point of initial turbidity.

Moreover, the last portions of chrome-alum may also become insoluble by long-continued heating at the boiling point.

First thoughts would lead one to expect to find the liberated sulphuric acid in the waste liquor along with the undecomposed chrome-alum. Strange to say, this is not the case entirely, for the facts compel the conclusion that the wool contains free sulphuric acid as well as the basic chromium sulphate.

Suppose we take the mean of the above analyses and consider the basic chromium sulphate to have the composition  $1(\text{Cr}_2\text{O}_3) + 0.666\text{SO}_3$  ( $= \frac{2}{3}$  sulphate). Such a compound is obtained, according to Gimelin, on heating a solution of  $\text{Cr}_2(\text{SO}_4)_3 \cdot 2(\text{OH})_2$  as a pale green precipitate having the composition  $2(\text{Cr}_2\text{SO}_4(\text{OH})_2) + \text{Cr}_2(\text{OH})_6$ , *i.e.*,  $(\text{Cr}_2\text{O}_3 + 0.666\text{SO}_3)$ . Now we have already seen that about 85.7 per cent. of the chrome-alum used is precipitated in the form of basic sulphate of the above composition. Omitting the  $\text{K}_2\text{SO}_4$ , the amount of chrome-alum used contains 0.153 grm.  $\text{Cr}_2\text{O}_3 + 0.240$  grm.  $\text{SO}_3$ , but since 85.7 per cent. of the  $\text{Cr}_2\text{O}_3$  (*i.e.*, 0.13112 grm.) require for the formation of the  $\frac{2}{3}$  sulphate, 0.0457 grm.  $\text{SO}_3$ , there must remain in the waste liquor 0.0219 grm.  $\text{Cr}_2\text{O}_3$  combined with 0.1943 grm.  $\text{SO}_3$ —

$$0.0219 : 0.1943 = 153 : x ; x = 1358. \quad \frac{1.55}{89} = 16.9.$$

therefore the remainder of the chromium must be found in the waste liquor as  $(\text{Cr}_2\text{O}_3 + 16.9 \text{ SO}_3)$ , *i.e.*, as a very acid sulphate. Experiments showed, however, that much

less free sulphuric acid was present than is here indicated. The following experimental data may be cited as typical of many which were made:—

$$\begin{array}{l} a. \quad \text{Cr}_2\text{O}_3 = 0.016, \quad \text{SO}_3 = 0.1033. \\ 0.016 : 0.1033 = 153 : x; \quad x = 188. \quad \frac{188}{80} = 12.35. \end{array}$$

There was, therefore, really present in the waste liquor an acid sulphate of the composition ( $\text{Cr}_2\text{O}_3 + 12.35 \text{SO}_3$ ).

On the wool and in the precipitate there should accordingly be ( $\text{Cr}_2\text{O}_3 + 1.91 \text{SO}_3$ ), calculated as follows:—

$$\begin{array}{l} 0.153 - 0.016 = 0.137 \text{ gm. Cr}_2\text{O}_3 \\ 0.240 - 0.1033 = 0.1367 \text{ gm. SO}_3 \\ 0.137 : 0.1367 = 153 : x; \quad x = 152.7; \quad \frac{152.7}{80} = 1.91. \end{array}$$

Such a compound, however, in spite of its basic character, should be perfectly soluble, an evident contradiction of the facts.

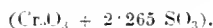
The 0.137 gm.  $\text{Cr}_2\text{O}_3$  present on the wool would require for the formation of  $\frac{2}{3}$  chromic sulphate 0.0477 gm.  $\text{SO}_3$ . By difference, however, there is found to be 0.1367 gm.  $\text{SO}_3$ , hence there must be on the wool 0.1367—0.0477, i.e., 0.089 gm.  $\text{SO}_3$  in the free state.

It seems somewhat difficult to imagine the presence of basic chromium sulphate and of free sulphuric acid in the wool at the same time, but the correctness of this view will be established by experiments to be cited later on. It may, however, be already stated here that long boiling of the green coloured wool with excess of hydrochloric acid did not dissolve off the basic sulphate.

b. On calcining another sample of wool mordanted in chrome-alum, it was found to contain 0.1385 gm.  $\text{Cr}_2\text{O}_3$  (containing a trace of  $\text{Fe}_2\text{O}_3$ ) and 0.164 gm.  $\text{SO}_3$ , from which the following calculation—

$$0.1385 : 0.164 = 153 : x; \quad x = 181.2; \quad \frac{181.2}{80} = 2.265$$

shows that the wool contained the compound—



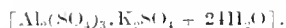
Since, however, the 0.1385 gm.  $\text{Cr}_2\text{O}_3$  would as  $\frac{2}{3}$  basic sulphate only contain 0.0482 gm.  $\text{SO}_3$ , there must have been 0.164—0.0482, i.e., 0.1158 gm. free  $\text{SO}_3$  in the wool, an amount which is somewhat larger even than that estimated above. In the waste liquor from the wool here analysed there was found 0.0145 gm.,  $\text{Cr}_2\text{O}_3$ , and 0.076 gm.,  $\text{SO}_3$ , which correspond to the compound ( $\text{Cr}_2\text{O}_3 + 10.02 \text{SO}_3$ ).

c. Another similar analysis showed the wool to contain ( $\text{Cr}_2\text{O}_3 + 1.745 \text{SO}_3$ ), while the waste liquor contained ( $\text{Cr}_2\text{O}_3 + 11.16 \text{SO}_3$ ). In this experiment the mordanted wool was extracted for a very long time with distilled water, which may account for the larger proportion of  $\text{SO}_3$  found in the waste liquor.

d. It would be too tedious to cite all the different analyses which were made, but another may be given in which a comparison was made between the behaviour of chrome alum and ordinary potash alum.

*Experiment 4.*—Wool was mordanted with—

a. 9.48 per cent. potash-alum—

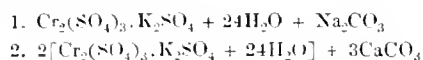


b. 10 per cent. chrome-alum.

The alum bath began to be slightly turbid at 73° C., and did not appear to change much afterwards; when cold a good deal of precipitate could be removed from the wool by kneading it with water. The chrome-alum bath began to be turbid at 82° C., the decomposition being specially marked at 85° C., and soon quite complete; 88.8 per cent. of the chrome alum was decomposed. On titration, (a) required 2.56 cc. normal NaOH; (b) required 0.93 cc. Analysis showed the wool to contain ( $\text{Cr}_2\text{O}_3 + 2.238 \text{SO}_3$ ), and the waste liquor ( $\text{Cr}_2\text{O}_3 + 9.01 \text{SO}_3$ ).

*Experiment 5.*—Although the foregoing experiments showed that no good result could be expected by employing basic chromic sulphate, one experiment was made with this

compound, more with the view of demonstrating how very differently wool behaves from cotton. The following two baths were prepared:—



both equivalent to 10 per cent. chrome-alum, and two wool patterns were mordanted as usual. As expected, both solutions decomposed very much, and the wool acquired a fine sea-green colour. The result reminds one forcibly of Chevreul's attempt to colour wool with finely divided carbon, ferric hydrate, &c., and this method of dyeing wool green may certainly be capable of practical application. Both patterns gave poor colours on being dyed and were therefore, as expected, badly mordanted.

*Experiment 6.*—To determine if the potassium sulphate present in chrome-alum exercised any influence, in connexion with the decomposition of the latter, 10 grms. wool were boiled with a solution of pure chromic sulphate equivalent to 1 gm. chrome-alum and prepared as follows: One gm. chrome-alum in solution was precipitated with the equivalent amount of NaOH and the chromic hydrate was well washed and at once dissolved in the equivalent amount of normal sulphuric acid. The appearances observed were exactly the same as in the case of chrome-alum. (The waste liquor contained 0.01346 gm.  $\text{Cr}_2\text{O}_3$  and 0.0774 gm.  $\text{SO}_3$ , therefore the wool and precipitate together contained 0.13954 gm.  $\text{Cr}_2\text{O}_3$  and 0.1626 gm.  $\text{SO}_3$ . Hence the waste liquor contained ( $\text{Cr}_2\text{O}_3 + 10.99 \text{SO}_3$ ), and the wool ( $\text{Cr}_2\text{O}_3 + 2.23 \text{SO}_3$ ).

After mordanting, the wool was well pressed, then steeped in cold distilled water, and boiled again with water repeatedly. It was impossible, however, even after many days, to remove the last traces of acid. The first wash waters were added to the waste liquor before making the analysis. In a subsidiary experiment, bichromate of potash, equivalent to 10 per cent. chrome-alum, was reduced by addition of 4 mols.  $\text{H}_2\text{SO}_4$ , and the necessary amount of starch and the solution was employed for mordanting 10 grms. wool. As was to be expected, the same appearances were observed as in the case of chrome-alum.

*Experiment 7.*—It seemed desirable to determine carefully the behaviour of chromium chloride and nitrate towards wool. These salts were prepared from chrome-alum in the same manner as the chromium sulphate, in order to be quite certain that exactly the same amount of  $\text{Cr}_2\text{O}_3$  was used in each case.

The chromium chloride bath became yellowish green on heating, but remained perfectly clear throughout. The wool acquired a peculiar greenish yellow tint, and had not the appearance of being mordanted at all. Analysis showed that 86.54 per cent. of the chromium remained in the waste liquor, so that only 13.46 per cent. was fixed on the wool.

The waste liquor contained  $\text{Cr}_2(\text{OH})_2\text{Cl}_4$ , hence the wool must have contained the compound ( $\text{Cr}_2 + 18.3 \text{Cl}$ ). This corresponds to ( $\text{Cr}_2\text{O}_3 + 9.15 \text{SO}_3$ ), hence in this case a very acid salt is fixed on the wool. The circumstance that here none of the chromium was precipitated as was the case with chrome-alum, is quite in accordance with the fact that both chromium chloride and nitrate in solution bear the addition of a large proportion of sodium carbonate (up to 2 mols.), without a precipitate being formed.

With chromium nitrate too, a very indifferent mordanting of the wool was effected, the material acquiring even a paler greenish-yellow tint. The waste liquor contained 0.1339 gm.  $\text{Cr}_2\text{O}_3$ , i.e., 87.52 per cent. of the original amount, so that the wool contained only 12.48 per cent.

Both samples of mordanted wool gave poor results in dyeing, a point to which reference will be made when dealing with the effect of adding various acids to chrome-alum mordant.

*Experiment 8.*—According to Havrez, wool may be mordanted to a certain extent even in the cold. In order to test this point, 10 grms. wool were steeped for a month in a solution of 1 gm. chrome-alum in 600 cc. water, and shaken up every day. After thorough washing, the fent was dyed, when it became at once evident that a partial

mordanting of the wool had really occurred. A titration showed that about 11 per cent. of the chrome-alum had been decomposed.

In all cases of mordanting wool with acid salts, as was already observed long ago by Hayez, and referred to more recently by Knecht, the presence of ammonia could be detected in the waste liquors, and this too was observed with certainty, even in the case of the wool mordanted in the cold. The ammonia, however, was not quantitatively estimated. It is well to point out here that in all the analyses of waste liquors given, a portion of the sulphuric acid reckoned as in combination with chromium is really combined with ammonia.

It is not unnatural therefore, to consider that the ammonia which is given off from the wool combines with some of the  $\text{SO}_4$  groups of the chrome-alum and so effects its decomposition. This undoubtedly occurs, but only to a limited extent, and cannot be accepted as a full explanation of the reaction which takes place, otherwise the analyses would not show any disappearance of  $\text{H}_2\text{SO}_4$  from the waste liquor. To avoid any misconception on this point, consider the following typical case, a little more in detail. When wool is boiled with 5 per cent. chrome-alum in solution, the decomposition of the salt is practically complete, as we have seen. A small amount (equal to  $\frac{1}{3}$  mol.  $\text{H}_2\text{SO}_4$ ) is combined with the precipitated  $\text{Cr}_2\text{O}_3$ , so that the waste liquor should contain  $3\frac{1}{3}$  mols.  $\text{H}_2\text{SO}_4$  capable of being estimated as barium sulphate. Such is, however, never the case, as numerous analyses have shown; invariably a large proportion of the  $\text{H}_2\text{SO}_4$  is wanting, and must, therefore, have been taken up by the wool. That such is really the case will be better understood in connection with other experiments still to be cited.

**Experiment 9.**—This experiment was made in order to confirm if possible with more certainty the truth of the conclusions, drawn from the analyses already made, the immediate question to be answered being: what becomes of the  $\text{K}_2\text{SO}_4$  of the chrome-alum. 10 cc. dilute  $\text{H}_2\text{SO}_4$  was exactly neutralised with  $\text{KOH}$ , then made up to 600 cc. with water, and 10 grms. wool were boiled in the  $\text{K}_2\text{SO}_4$  solution. The bath became faintly turbid through the presence of organic matter, but it remained perfectly neutral. The sulphuric acid determined gravimetrically in one half of the bath, amounted to 0.2135 gm.  $\text{SO}_3$ , therefore 0.427 gm. per 600 cc. waste liquor. A parallel determination was made with 10 cc. of the original dilute sulphuric acid employed, and it was found to contain 0.4266 gm.  $\text{SO}_3$ . This complete agreement in the numbers obtained, showed that the potassium sulphate was not absorbed by the wool but remained entirely in the bath. Hence, in all the analyses of waste liquors, the  $\text{BaSO}_4$  due to the presence of the  $\text{K}_2\text{SO}_4$  was invariably deducted, and only the residual  $\text{H}_2\text{SO}_4$  was reckoned as combined with the chromium.

**Experiment 10.**—This experiment was made in order to see if, during the mordanting process, the sulphur of the wool itself might not yield sulphuric acid.

Ten grms. of wool were boiled in a solution of sodium nitrate made by neutralising 10 cc. normal nitric acid with potassium hydrate and diluting to 600 cc. Barium chloride was added to the neutral and slightly turbid waste liquor after acidifying it with hydrochloric acid, but it produced only yellowish white flocks, not in the least like barium sulphate, and when calcined showing traces of iron to be present. This residue only weighed 0.017 gm., and was probably composed of  $\text{BaO}$  and  $\text{Fe}_2\text{O}_3$  rather than of  $\text{BaSO}_4$ . At any rate, such traces of  $\text{H}_2\text{SO}_4$  might be neglected.

It is usually considered that a chrome-alum solution which has become green by heating is dissociated, and this dissociation occurs between  $60^\circ$  and  $80^\circ$  C. Tieckhorne, however, has suggested that the chromatic change is due to basicity of the solution (Chemical News, 24, pp. 209, 211). When he allowed dilute chrome-alum solution to drop into boiling water he observed that dissociation occurred at once (see experiment 19), the solution became opaque and basic sulphate was precipitated, but it dissolved again on cooling. An attempt to repeat this experiment was not

successful, for the solution never exhibited the decided turbidity which invariably appeared whenever wool was present. According to Krieger the green solution contains  $(\text{Cr}_2\text{O}_3 \cdot 2\text{SO}_3)_2$ , while Stewart states that 8 mols. chrome-alum are decomposed into—



Again G. D. van Claef (1881) showed that green chrome-alum solutions were separable by dialysis into two parts, an acid sulphate diffused through the septum while a basic sulphate remained behind. Berzelius had previously shown a similar state of things to exist in connection with ferric salts. Lowel found the presence of green salts saturated only to the extent of  $\frac{1}{2}$ , which corresponds to a tetra-acid chromic salt. The latest work on this subject is that of Recoura, who says: under the action of heat, the oxide combined with three equivalents of acid undergoes a modification which alters its capacity for saturation, so that it can only remain in combination with two equivalents of acid, the third being in the free state, and the liquor becomes green. Since his chromo-sulphuric acid  $(\text{Cr}_2\text{O}_3 \cdot 4\text{SO}_3)_2$  is immediately decomposed on boiling, it can scarcely enter into consideration here. Recoura himself considers that the change from violet to green in the case of chrome-alum solutions depend on the formation of the basic sulphate  $(\text{Cr}_2\text{O}_3)_2(\text{SO}_3)_2$ , to which the corresponding hydrate would be  $\text{Cr}_2\text{O}_3(\text{OH})_2$ .

An experiment was made in which chrome-alum was titrated with normal  $\text{NaOH}$ , and it was observed that a first change of colour really occurred when one-sixth of the  $\text{SO}_3$  was neutralised, and a second change to a decided yellowish-green colour was noticed when one-third of the  $\text{SO}_3$  was saturated. These observations correspond exactly with the appearances noticed during the mordanting process, so that the decomposition of the chrome-alum during the mordanting of the wool can scarcely be explained in any other manner than by considering that the wool gradually absorbs the sulphuric acid from the dissociated green solution with the formation of a more and more basic salt, and that this process continues until the wool under the existing conditions becomes saturated. As above shown, this point occurs when about 8.57 per cent. of the 10 per cent. chrome-alum presented to the fibre is decomposed, and it has already been shown that 5 per cent. is completely decomposed by the wool.

**Experiment 11.**—This experiment was made in order to see if substances similarly constituted to wool would behave in a similar manner towards chrome-alum solutions. The whites of two eggs were beaten to a froth with cold distilled water, and then poured into boiling distilled water. The finely-divided coagulum thus obtained was pressed, well washed, and treated with dilute acetic acid to remove the last traces of alkali, and finally washed again. The coagulated albumen thus prepared was boiled for  $1\frac{1}{2}$  hours with 1 gm. chrome-alum dissolved in 600 cc. water, with the result that exactly the same appearances were observed as in the case of wool, and the albumen absorbed 74 per cent. of the acid present. In another experiment hide-powder was boiled with chrome-alum solution in a similar manner, and the same reactions occurred; the chrome-alum was decomposed with precipitation of a very basic sulphate.

**Experiment 12** was made in order to determine approximately how much sulphuric acid was absorbed by the wool at the point when turbidity of the bath began. 1 gm. chrome-alum was dissolved in 600 cc. water; the solution was heated to  $80^\circ$  C., and then rapidly, but carefully, titrated with normal sodium hydrate. Incipient turbidity was caused by the addition of 2.5 cc., which corresponds to the absorption of 1.25  $\text{SO}_3$ , so that at this moment the bath would contain  $(\text{Cr}_2\text{O}_3 - 1.75 \text{SO}_3)_2$ .

If now the above-mentioned view is correct, namely, that the almost total decomposition of the chrome-alum solution is caused by the absorption of its sulphuric acid by the wool, two modes of preventing it appear possible: (1) the employment of larger amounts of chrome-alum; (2) the addition of acid to the bath.

*Experiment 13.*—Wool was mordanted with 10, 15, 20, 25, 30, 35, and 40 per cent. chrome-alum in the ordinary manner. With increase of concentration of the baths their decomposition was diminished, as indeed was to be expected on physical grounds, so that the solutions containing the largest amounts remained perfectly clear. With the increase in the amounts of mordant used, the separation and fixing of insoluble basic sulphate gradually disappeared; the samples had the appearance of being better mordanted, showing signs of having fixed large quantities of chromium. Instead of the yellowish-green colour observed before, they had now a violet-greenish grey tint, and they gave one the impression that chrome-alum as such was in a state of solution within the wool-fibre.

The samples were dyed with logwood, alizarin, and Persian berries, and the colours obtained became darker and darker, corresponding to the increasing amounts of chrome-alum employed. When examined more carefully, however, the colours were seen to have an irregular, speckled, or seedy appearance, since separate wool fibres had evidently taken up different amounts of mordant, and therefore of colouring matter as well. A microscopic examination showed this peculiarity extremely well, and fibres of every degree of intensity of colour were to be seen, some being almost colourless, others very dark; indeed, similar differences were noticed even in one and the same fibre.

With chromic acid, *i.e.*, bichromate of potash, it is easy to mordant the wool evenly, so that when examined microscopically the individual fibres scarcely differ from each other in depth of colour. With the chromic oxide mordants however such regularity is difficult to attain and is only met with in the very best of this class. No doubt this difficulty has to some extent caused practical wool-dyers to discard chrome-alum. With the dyestuffs above alluded to (logwood, alizarin, and Persian berries) and with the increasing amounts of chrome-alum, the colours varied respectively from pale grey to deep bluish-black, from pale yellowish-chestnut to a dark dull purple, and from straw-yellow to a brownish-orange.

While the question of overchroming, referred to in the last paper, was under consideration, a few experiments were made to see if similar effects could possibly be obtained with chromic oxide mordants, and it may be well to refer to these here.

*Experiment 14.*—The usual 10 grms. wool, divided into six equal pieces, was mordanted with 40 per cent. chrome alum. No. 1 sample was removed from the bath just when it began to boil, No. 2 a quarter of an hour later, and so on; No. 5 was boiled 1 hour, and No. 6, 2 hours. The mordanted samples were well washed, and half of each was extracted several times with boiling water, then dyed with logwood at the boil and finally washed in boiling water. In no case was there formation of lake in the dye-bath, and no overchroming was perceptible. All the mordanted samples had a violet greyish-green tint similar to that of chrome-alum, and the baths showed no signs of dissociation. The dyed patterns varied from a medium to a deep shade of bluish-black with a slight reddish cast in the case of those patterns which had been washed in the ordinary manner with cold water after mordanting. Although chrome alum seems finally to give up a larger amount of chromium to the wool than does chromic acid, the latter gives up more than the former in a limited period.

After pursuing the study of the behaviour of chrome-alum up to this point, we were, through the kindness of Mr. R. Crenier (Verviers) enabled to consult an important though little known paper written by Paul Havrez, entitled "*Étude de l'alunage des laines à teindre*," and which appears in the *Bulletin du Musée de l'Industrie de Belgique*, May and June 1872. Havrez worked with ordinary alum, but in most points his results coincide with the foregoing, although his explanations of the reactions which occur differ from ours. He imitated the somewhat older work of Chevreul who neglected the influence exerted by the amounts of alum employed, and who indeed adopted conditions altogether useless from a practical point of view. For example, he treated 1 grm. wool in the cold with 25 grms.

of a 5 per cent. alum solution (therefore 125 per cent. alum) and then determined the amount of water, alum, &c. in 10 grms. of the bath. Still, Chevreul had already shown that wool absorbed 2.84 per cent.  $\text{H}_2\text{SO}_4$ , of which it could not be deprived; further, he observed the liberation of ammonia or of an ammoniacal product. He considered also that wool was capable of absorbing 1.26 per cent. alum. Previous to Chevreul indeed, Thénard and Roard found that wool could absorb alum as such, *i.e.* without undergoing decomposition.

Havrez studied specially the influence of the relative amounts of alum, wool, and water; then the influence of temperature, and the duration of the action. He worked at a tepid and also at a boiling heat, with amounts of alum varying from  $\frac{1}{100}$ —100 per cent., and after dyeing with various dye-woods, he determined by means of Chevreul's chromatic scale, the effect produced as regards shade, saddening and intensity. Unfortunately he seldom gives positive chemical data so necessary to the understanding of his assertions, and probably for this very reason his otherwise excellent work has for the most part been neglected by chemists.

Havrez observed the decomposition of the alum when employing small amounts, and at first was inclined to attribute it to traces of soda left in the wool from the scouring, the calcium carbonate of the water employed, and the ammonia resulting from the decomposition of the gelatinous principle of the wool; afterwards, however, he adopted a suggestion of the celebrated chemist Stas, and ascribed the cause to the dissociation of the alum. We find, however, that although an addition of acetic acid prevents dissociation, no good results are obtainable in this manner. Further, we disagree with his assertion that an excess of alum acts like an acid which dissolves the alumina, and gives shades which remind one of the reddening of litmus by acid, while small amounts give shades which correspond to the bluing of litmus by alkalis. On the other hand, Havrez rightly maintains that with large amounts of alum, there is fixed in the wool a compound of alumina and sulphuric acid. In our experiments the largest amount of chrome-alum employed was 40 per cent., which gave indeed the darkest colours, and there was therefore no sign of any oxide being dissolved off the fibre. Havrez also observed that wool mordanted for a long time in old alum baths became covered with a white, powdery precipitate, which he regarded as aluminium hydrate. In the case of chrome-alum, our conclusion is, as already stated, that the analogous precipitate is really a very basic sulphate. Havrez states that if a number of mordanting operations are effected in the same bath, the solution acquires an alkaline reaction towards litmus, if for 1 part alum more than 15 parts wool are employed. This does not occur with less than 15 parts wool to 1 part alum, the alumina then remains in solution, and purer, brighter colours are obtained in consequence of the absorption of free sulphuric acid and excess of alum. With less than 7 per cent. alum the action of the basicity of the bath becomes evident, the wool suffers, becomes brownish tinted through alkali, emits odorous gases, and gives only dull colours on dyeing. An addition of acid is beneficial with small proportions (1 per cent.) of alum; this statement of Havrez is also true, and shows how carefully he worked and observed.

Further on there will be an opportunity of showing that with smaller amounts of colouring matter one must use correspondingly smaller amounts of mordants, but at the same time increasing amounts of acid, in order to obtain bright normal colours.

Havrez declared that with small amounts of alum, boiling was not at all necessary, and that he could mordant wool with aluminium acetate at a low temperature. This last point we have not exhaustively studied, but up to the present we have invariably obtained poor results when mordanting at a low temperature. Certainly Havrez both mordanted and dyed in much more concentrated baths than ours, *viz.*, 1,500 parts water per 100 parts wool, a proportion which is probably seldom or never met with in practice. The mordanting action undoubtedly varies according to the concentration of the baths employed, and hence it is

difficult to bring into accord the various statements of different authors, since each has evidently worked under slightly different conditions.

We are unable, however, to confirm Havrez's statement that by using 2 per cent. sulphuric acid with 10 per cent. alum, the dyeing with logwood is hindered; we find that even with 3 and 4 per cent. acid with 10 per cent. chrome-alum, deep colours are obtained.

Havrez concludes, always judging merely from the appearance of the dyed colours, that with the use of 3-7 per cent. alum, a basic sulphate is fixed on the fibre; with more than 7 per cent. a little less alumina and more aluminium sulphate is fixed. The maximum amount lies between 10 and 15 per cent.; larger amounts are said to dissolve off the alumina already fixed, and thus yield paler colours; with 30-40 per cent. alum, deep colours are only obtained after very long boiling.

In explanation of his results, Havrez assumes in the first place that dissociation occurs, thus:—



and he considers the  $\text{Al}_2(\text{OH})_6$  to be present partly in a soluble modification. Further, he considers that the wool takes up these two forms of hydrate in unequal amounts along with free alum. Finally, when several lots of wool are mordanted in a mixture of aluminium sulphate and sulphuric acid, he concludes that the first lots of wool absorb relatively more sulphuric acid, since after some time the bath contains alum only and not any free sulphuric acid; "l'absorption fixe la dissociation, la rend permanente." A bath is poorer in sulphuric acid and richer in basic sulphate, the larger the quantity of wool which is worked in it, either all at once or in successive quantities.

Further reference here to Havrez's work is unnecessary, except to refer the reader to the abstract which occurs in Chas. O'Neill's *Textile Colourist*, Vol. III., 1877, p. 131, and to give Havrez's general conclusions, which are as follows:—

(1.) Strong doses of mordants act in solution as salts (or as salts and acids) after their absorption by wool.

(2.) Weak doses of mordants act upon wool as metallic hydrates, the formation of which is assisted by long boiling.

(3.) The unequal absorption by wool of the basic hydrates and dissociated acids, and its reactions upon each of them, cause these differences.

(4.) The excess of salt, it would appear, may be replaced by small quantities of acids, or of acid salts of potassium (bisulphate, binoxalate, tartar, &c.), which in water are resolved into free acid and neutral salt.

(5.) The augmentation of the metallic hydrate fixed is procured by addition of water, increase of heat, or prolonged contact.

(6.) The colour which pure wool takes in dyeing, confuses that due to the first portion of hydrated acid, or metallic hydrate absorbed by the wool. Acids act specially in purifying wool: they seem also to set the colouring matters free from glucosides. The metallic hydrates fix coloured lakes upon wool in a different manner.

## B. CHROME-ALUM WITH VARIOUS ACIDS.

### 1. Chrome-Alum and Sulphuric Acid.

The foregoing series of experiments have shown that although the dissociation of chrome-alum may be prevented by employing large amounts, still level dyeing with the use of this mordant is not to be obtained in this manner, and hence experiments were now tried with the addition of acids.

**Experiment 15.**—Wool was mordanted with 10 per cent. chrome-alum + 0, 1, 2, 3, 4 mols.  $\text{H}_2\text{SO}_4$ .

In baths Nos. 1 and 2 strong dissociation occurred, in No. 3 there was much less, but in Nos. 4 and 5 the solutions remained perfectly clear; the waste liquor of No. 1 appeared almost colourless, but the rest showed a green coloration, which gradually became more intense towards No. 5. Nevertheless the dyed patterns showed that from Nos. 2-5 the amount of chromium fixed gradually increased, Nos. 4 and 5 especially giving dark colours.

Here, too, however, a microscopic examination showed very unequal dyeing of the different fibres, and the patterns themselves had a "seedy" appearance, just as when large percentages of chrome-alum were employed. In individual fibres the tips always appeared darker than the roots, i.e., the younger part of the fibre. The same observations were made in the case of unspun lamb's wool secured in the laboratory, which was tried perchance the woollen cashmeres employed contained more than one quality of wool. Here, too, the use of bichromate of potash gave level colours, the irregularities only appearing with the employment of the chrome oxide salt. The difference in the general behaviour of cotton and wool towards mordants is here shown in a striking manner. With the former, as is well known, we have to employ basic salts; whereas with the latter the presence of free acid seems to be absolutely essential.

The amounts of sulphuric acid recommended many years ago by Peissert were 1.66-2.5 mols. per 1 mol. chrome-alum. When dyed in alizarin, pattern No. 5 gave indeed a very deep bluish Bordeaux, but the colour was not full and it had a peculiar greyish hue, and differed entirely from the normal Bordeaux obtained from alizarin.

No chromium determinations were made, but the titration of the waste liquor of bath No. 5 showed an absorption by the wool of 0.288 grm.  $\text{SO}_3 = 0.3528$  grm.  $\text{H}_2\text{SO}_4$ , so that the mordant fixed upon it was very acid.

The poor result as regards colour here obtained accords entirely with that obtained some years ago by Liechti and Schwitzer, when it was shown that the use of sulphuric acid as an assistant cannot replace organic acids (*Mittheilungen des technologischen Gewerbe-Museums, Wien, III., p. 56*).

### 2. Chrome-Alum and Acetic Acid.

If acetic acid proved to have the power of preventing the dissociation of the chrome-alum, good results in dyeing might be expected, but experiment showed this not to be the case.

**Experiment 16.**—Wool was mordanted with 10 per cent. chrome-alum, + 0, 3, 6, 8, 10, 12, 14 mols. acetic acid, also + 2, 4, 6 mols. sodium acetate. Baths Nos. 8, 9, 10 became turbid first; after 35 minutes, baths Nos. 2, 3, 4 became also turbid, but more slightly; at 95° C. bath No. 5 became turbid, but to a still smaller extent; while baths Nos. 6 and 7 remained clear. When washed and dyed with 25 per cent. logwood, poor, "seedy" greys only were obtained, little calculated to inspire further experiment in this direction. A note of Havrez gave hope that better results might be obtainable by mordanting at a lower temperature. The experiment was therefore repeated, employing the amounts of Nos. 2-7 and a temperature of 60-70° C. In this case all the baths remained clear, but although the dissociation of the chrome-alum had thus been prevented, the dyed result was even worse than before. When microscopically examined some of the fibres showed a deep bluish grey colour, but others were almost colourless.

This result appears to indicate that a more acid compound than  $\text{R}_2(\text{SO}_4)_3$  must necessarily be present on the fibre in order to produce the colour lake within its substance; acetic acid being a volatile acid, is apparently not adapted for this purpose.

The last three mordants, Nos. 8, 9, 10, with sodium acetate, were included in order to see if a salt less rich in  $\text{SO}_4$  than chrome-alum could be fixed upon the wool, but, as expected, the results were very poor. Already in 1886 Liechti and Schwitzer found that pure chromium acetate gave unfavourable results when employed as a mordant for wool.

Before referring to further mordanting experiments, it is necessary to cite briefly a few experiments made with the view of determining the importance or otherwise of certain possible sources of error. As is well known to the analytical chemist, certain kinds of glass are not wholly unattacked by boiling water, and there was a possibility, therefore, that the chrome-alum might be to a certain extent decomposed by the glass. One of the glass dye-vessels containing 600 cc. water was, therefore, heated in the course of an hour to the boiling-point and boiled one hour, with the addition of a little methyl orange, just reddened with a trace of hydro-

chloric acid, and afterwards allowed to cool. On comparing the tinted water which had been boiled with a small sample of it which had been set aside previously, no indications of any neutralisation could be seen. Another similar experiment was made by boiling 600 cc. water with the addition of an amount of sulphuric acid (6 cc. normal  $H_2SO_4$ ) equivalent to 1 gm. chrome-alum; to neutralise the liquid afterwards 6 cc. normal NaOH was required, so that here, too, it was shown that the alkali of the glass dye-vessel had exercised no action in the mordanting experiments.

Another possible source of error might of course be the faint alkalinity of the Leeds water, as well as that of the wool itself, due to the scouring process. For 600 cc. Leeds water there was required 0.21 cc. normal acid to neutralise it, and for the 10 grms. wool, 0.79 cc.; therefore together 1 cc. acid. A sample of 10 grms. wool was therefore mordanted with 10 per cent. chrome-alum in 600 cc. water, with the addition of 1 cc. normal sulphuric acid, but the same appearances as those already described were again observed. It is therefore, as we believe, conclusively proved that the decomposition of the chrome-alum is brought about by the wool itself absorbing the sulphuric acid from the dissociated chrome-alum in the mordanting bath.

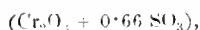
**Experiment 17.**—It seemed possible to prevent the decomposition of the chrome-alum, if the wool's affinity for acid were satisfied *before* the mordanting process. To this end two samples of wool were boiled for  $1\frac{1}{2}$  hours with 600 cc., the one with the addition of 1 gm. sulphuric acid, and the other with its equivalent, 1.88 grms. cream of tartar. After washing, the samples were mordanted as usual in a separate bath with 10 per cent. chrome-alum. Even here, however, though to a less extent, turbidity and decomposition of the chrome-alum ensued. On dyeing with logwood better bluish-black colours were obtained than with the use of chrome-alum alone, but they were not altogether satisfactory either in colour or evenness. Examination by the microscope showed both deep black and pale bluish slate-coloured fibres. On titrating the waste liquors it was found that an absorption of 0.2832 gm.  $SO_3$  and cream of tartar corresponding to 0.1276 gm.  $SO_3$  had occurred, so that the amount of the latter substance absorbed by the wool was 45 per cent. of the former.

**Experiment 18.**—A special experiment showed that "chlorinated" wool may be well mordanted with chrome-alum without any decomposition being noticeable in the bath. A 10-grm. sample of ordinary wool was heated with 600 cc. water and 2 grms. sulphuric acid, then well squeezed and mordanted with 10 per cent. chrome-alum, and in this case no decomposition in the mordant bath was noticeable.

If after the treatment with acid the wool is steeped for a quarter of an hour in a cold dilute solution of bleaching powder, then washed and mordanted with chrome alum, no decomposition of the chrome-alum occurs in the bath, but one observes an interesting formation of chromic acid.

Apart from the effect of the oxidation of the wool, possibly the good results obtained on "chlorinated" wool, with some colouring matters at least, may depend to some extent upon the acid absorbed by the wool. In the case of the above two samples, when dyed with alizarin, the non-chlorinated sample gave a garnet-red colour, pointing evidently to the effect of the acid, absorbed by the wool, whereas the second one gave a bluish Bordeaux colour, which probably contained lime.

It has already been stated that the wool takes up the free sulphuric acid from the dissociated green chrome alum solution, with the formation and precipitation in the bath and on the wool of the basic compound—



and it has been shown that the same reaction takes place to a less extent even in the cold, if the operation is prolonged sufficiently, hence the idea occurs to one that even in the case of the violet chrome-alum solution, there may be a partial or incipient dissociation.

Here the first question to be determined is whether or not free sulphuric acid is present upon wool mordanted with chrome-alum.

**Experiment 19.**—A piece of chrome-alum mordanted wool was divided into six parts, which was then dyed with logwood—

- |      |   |
|------|---|
| (1.) | Without addition.                             |
| (2.) | With addition of 1 cc. normal sulphuric acid. |
| (3.) | " " 1 " " oxalic acid.                        |
| (4.) | " " 1 " " tartaric acid.                      |
| (5.) | " " 1 " " sodium acetate.                     |
| (6.) | " " 1 " " calcium acetate.                    |

The colours were of course poor, "seely," brownish-greys, and even with the naked eye one could detect among the black fibres dirty pale brown ones. The patterns Nos. 2—4 had more of a reddish-brown cast, so that the colour was certainly not improved by an addition of acid to the dye-bath. This result accorded entirely with the negative one of another experiment, in which the attempt was made to correct an already decomposed chrome-alum bath. Patterns Nos. 5 and 6 showed plainly the beneficial effect of neutralising the free sulphuric acid in the wool by the sodium or calcium acetate, the latter seeming indeed better than the former. The colours of these patterns were dark bluish-greys, but even here the microscope revealed almost colourless fibres alongside of others which were deep blue.

Why individual wool fibres should behave so differently, as is here shown, is a moot point; whether it is due to a mixture of wool or whether the mature fibres behave differently to those of more recent growth, with respect to their power of absorption of the mordant constituents, is not yet determined. It always appeared, however, as if the coarser fibres had a deeper colour than the thinner ones. In any case it is very striking that similar marked differences are never met with in wool mordanted with bichromate of potash, and explains to some extent the preference of practical dyers for this mordant. O. N. Witt has also referred to this matter in connection with the dyeing of wool with indulin, and dyers meet with it frequently in ordinary practice; it is certainly one of considerable importance, and there is no doubt that in many cases in practice irregular colours have been caused by the spinner rather than by the dyer.

The different behaviour of various qualities of wool towards many colouring matters is often met with, especially in the case of azo-colours, and when two or three colours are being applied simultaneously. Sometimes, at a given temperature, one colour is already entirely taken up from the dye-bath while the others have been scarcely attracted at all, but only certain parts of the mixture of wools had become dyed, the others dyeing afterwards at higher temperatures, the end result being that the dyed wool contains fibres of different colours, and the general colour is bad because of the irrational mixing of different qualities of wool.

This is not the time to enter into this question further, but it seems possible that here, too, the unequal attraction for acid by the different wool fibres lies at the root of the matter. The observation of Breinl that wool treated with dilute sulphuric acid and then steamed, dyes deeper colours than untreated wool, is explained most simply by ascribing it to the absorption of acid.

**Experiment 20.**—Another method of showing the presence of absorbed free acid may be based upon the behaviours of the acid azo colours. Wool, after mordanting with chrome-alum and washing, was dyed with 3 per cent. Fast Red E (BASAR). The bath was almost completely exhausted, and the wool was dyed a deep red; the shade was, of course, considerably dulled by the presence on the fibre of the basic chromic sulphate, but otherwise the dye was quite normal. A microscopic examination revealed no undyed fibres, but there were all shades from pale to dark red. The waste dye liquor had a neutral reaction. Unmordanted wool dyed in a similar manner only acquired a pale flesh tint.

**Experiment 21.**—Two samples of wool, 10 grms. each, were dyed simultaneously with 600 cc. distilled water, 0.3 gm. Fast Red E, the one with addition of 6 cc. normal sulphuric acid, the other with 1 gm. chrome-alum. The one with sulphuric acid exhausted the bath completely; the



chrome-alum bath became gradually turbid and was not fully exhausted; the colour of the wool was of course duller, of a bluer red, and not so deep as the other. The paler colour is explained by the fact that a portion of the  $\text{SO}_3$  could not assist in developing the Fast Red, since it was present in the precipitated basic chromic sulphate. Taking this into account, to have had equal amounts of sulphuric acid in action, one ought to have employed 1.28 grms. chrome-alum. For the rest, 6 cc. normal sulphuric acid are equivalent only to 0.83 grm.  $\text{NaHSO}_4$ , of which usually 10 per cent. is recommended to be used with acid colours, *i.e.*, 1 grm. in the present instance. The basic chromic sulphate precipitated in the bath had not attracted any colouring matter. Here, too, the dyeing shows clearly the absorption of sulphuric acid from the chrome-alum by the wool.

**Experiment 22.**—This is to some extent a repetition of experiment 21 (employing an equivalent amount of chromium oxalate instead of the sulphate), with the object of preventing the formation of a precipitate. By chance Bordeaux S was used instead of Fast Red. The bath remained clear, but was only about two-thirds exhausted. On addition of  $\text{NaOH}$  to the waste liquor, pure chromic hydrate was precipitated. It would appear from this result that wool has a greater attraction for sulphuric acid than for oxalic acid from chromium salts.

The experiments already referred to point to the necessity of determining the behaviour of wool towards various acids before continuing the study of the use of chrome-alum with the addition of organic acids. Such experiments were not originally intended when planning the work, but the interesting behaviour of wool towards chrome-alum seemed to compel this step, although, perhaps, little new could be expected from it.

In the first instance it seemed interesting to determine whether a relation exists between the amounts of acid absorbed respectively from a chrome-alum bath and one of free sulphuric acid. Should this be the case it would be an excellent proof that the decomposition of the chrome-alum is a direct result of the absorption of the sulphuric acid by the wool. Here, too, the experiments were always conducted in such a manner that 10 grms. wool were boiled for  $\frac{1}{2}$  hours with 600 cc. water containing the acids selected. After allowing the baths to cool and making up to the original volume, a definite portion was titrated and calculated for 600 cc.; the deficiency of acid was considered to have been absorbed by the wool. In this manner alone comparable results are to be expected, since it is practically impossible to wash out equally different samples of wool boiled with different acids, since the latter appear to be retained by the wool in different proportions. With respect to cream of tartar, amounts were always taken in which the free acid was equivalent to the other acids. According to Roard and Thenard, namely, wool decomposes cream of tartar into free tartaric acid which is absorbed by the wool and neutral potassium tartrate which remains in the bath. Kuhlmann, too, states that it is only the free tartaric acid which is active, and hence recommends that dyers should invariably liberate the whole of the tartaric acid by an addition of sulphuric acid. It may be incidentally mentioned that in all the waste acid liquors the presence of ammonia could be detected.

The early experiments on this subject of the absorption of acids by fibre, seem to be absolutely worthless for our present purpose, because they were made under conditions absolutely different from those imposed in practice, *e.g.*, those of Chevreul (1833) and Bolley (1859), as well as of Mills and Takamine (Journ. Soc. Chem. Industry, March 1883). More important for us are the recent experiments and analyses of Knecht (Journ. Soc. Dyers and Colourists, 4, 104). He found that wool boiled with 5 per cent. sulphuric acid, absorbed 3.6 per cent.; by subsequently extracting the fibre three times with boiling water 1.26 per cent. could be removed, so that 2.34 per cent. was permanently absorbed or neutralised by the wool. The amount of other acids equivalent to this would be 3 per cent. oxalic acid, 3.6 per cent. tartaric acid, 8.98 per cent. cream of tartar, 7.54 per cent. potash-alum, 7.94 per cent. chrome-alum. Now, according to Knecht, wool boiled

with 10 per cent. tartar absorbs 4.28 per cent. which corresponds to 1.111 grms.  $\text{H}_2\text{SO}_4$ , *i.e.*, only 17.5 per cent. of the above 2.34 per cent. With the use of only 5 per cent. alum (1.55 per cent.  $\text{H}_2\text{SO}_4$ ) total absorption of the sulphuric acid occurred; the more alum used the less relatively is the amount of sulphuric acid absorbed, as already previously observed by Havrez, and later by us in the case of chrome-alum. With the use of 20 per cent., 15 per cent., 10 per cent. alum, the amounts of  $\text{SO}_3$  found by Knecht to be absorbed were 11.0 per cent., 14.6 per cent., 22.1 per cent. of the 25.42 per cent.  $\text{SO}_3$  theoretically present. The cause of this is ascribed by him to dissociation. With 15 per cent. alum there was 7.1 per cent.  $\text{SO}_3$  permanently absorbed or neutralised, and there is probably fixed on the wool a basic sulphate. If the mordanted wool was not extracted with boiling distilled water there was found to be absorbed 14.60 per cent.  $\text{SO}_3$  instead of 7.1 per cent. From Knecht's data we cannot draw the same conclusions exactly, no doubt because in these cases titration does not suffice to give one a clear insight into the matter, and one ought so to titrate as to determine in an acid bath first the free acid and then that combined with the alumina.

Knecht presented to the wool—

$\text{Al}_2\text{O}_3$ , 0.0807 grm., and  $\text{SO}_3$ , 0.1898 grm.;

there remained in the bath—

$\text{Al}_2\text{O}_3$ , 0.0355 grm., and  $\text{SO}_3$ , 0.1367 grm.;

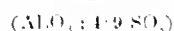
there was therefore on the wool—

$\text{Al}_2\text{O}_3$ , 0.0452 grm., and  $\text{SO}_3$ , 0.0531 grm.

From these data one may calculate as follows:—

$$0.0452 : 0.0531 = 102 : x; x = 119 \text{ and } \frac{119}{80} = 1.487$$

that is to say, the wool eventually contains the basic aluminium sulphate ( $\text{Al}_2\text{O}_3 : 1.487 \text{ SO}_3$ ), and if calculated in the same manner the bath is shown to contain—



If Knecht's data respecting the temporarily fixed compound are calculated in a similar way, the conclusion arrived at is that both on the wool and in the waste mordant liquor there is present ( $\text{Al}_2\text{O}_3 : 3\text{SO}_3$ ), this is—

Presented to wool—

$\text{Al}_2\text{O}_3$ , 0.0807 grm., and  $\text{SO}_3$ , 0.1898 grm.;

in the bath—

$\text{Al}_2\text{O}_3$ , 0.0355 grm. (?), and  $\text{SO}_3$ , 0.0837 grm.;

therefore on the wool—

$\text{Al}_2\text{O}_3$ , 0.0452 grm. (?), and  $\text{SO}_3$ , 0.1061 grm.

In both cases the figures work out to ( $\text{Al}_2\text{O}_3 : 3\text{SO}_3$ ).

According to the amount of alum employed in mordanting, as well as that of the water and the duration of the boiling, different results are obtained, and it is impossible, therefore, to give exact formulae which are generally applicable. The dissociation becomes less the larger the amounts of alum employed, and seems to us to become nil with the use of 35 per cent. chrome-alum. Knecht found that with increasing amounts of acid or alum, proportionally diminishing amounts are fixed, *e.g.*, 1.13 per cent. with addition of 20 per cent.  $\text{H}_2\text{SO}_4$ , and only 4.82 per cent. with 40 per cent.  $\text{H}_2\text{SO}_4$ .

In the case of alum no further decomposition will of course occur after the wool's need for acid has been satisfied. With the use of sufficiently large amounts of alum the baths must therefore remain clear, since only a very faintly basic sulphate is formed.

Knecht does not seem to have observed the actual formation in the solution of insoluble basic sulphate, although it is somewhat striking (especially in the case of chrome-alum). His conclusions on the question are as follows:—

"When boiled with 15 per cent. alum, wool elects a partial decomposition of the salt, leaving free acid in solution. The amount of the latter is, however, very small. The  $\text{Al}_2\text{O}_3$  and  $\text{SO}_3$  appear to be absorbed in about equivalent proportions."

#### Absorption of Various Acids by Wool.

In all these experiments it was found impossible to obtain concordant results. The results varied, for example, according to the amount of acid presented to the fibre, as noticed



also by Knecht, although we never employed such large amounts of acid as he. They varied, too, according to the concentration of the baths.

It is well perhaps to refer here to the difficulties connected with the equal heating and evaporation of the several racks, although every precaution was taken. To avoid gross errors, the baths were maintained at a constant level, and, after cooling thoroughly, were again made up to the mark (600 cc.) with water, and well stirred with the wool some time until a state of equilibrium had been established, before taking a portion of the liquid for analysis.

#### Absorption of Sulphuric Acid.

*Experiment 23.*—(1.) When 10 cc. normal  $\text{H}_2\text{SO}_4$  were used, i.e., 0.49 gm.  $\text{H}_2\text{SO}_4 = 0.40$  gm.  $\text{SO}_3$ , the following amounts were absorbed:—

$\text{H}_2\text{SO}_4$		$\text{SO}_3$
Grm.		Grm.
0.3467	=	0.283
0.3325	=	0.287
0.3469	=	0.283
0.3136	=	0.256
0.3386	=	0.276
0.2640	=	0.240
0.3332	=	0.272
Average 0.332	=	0.271

(2.) When 6 cc. normal  $\text{H}_2\text{SO}_4$  were used, i.e., 0.294 gm.  $\text{H}_2\text{SO}_4 = 0.240$  gm.  $\text{SO}_3$ , and equivalent to 1 gm. chrome-alum, the following amounts were absorbed:—

$\text{H}_2\text{SO}_4$		$\text{SO}_3$
Grm.		Grm.
0.25382	=	0.2072
0.24696	=	0.2016
0.2400	=	0.2000
Average 0.2439	=	0.2029

The above 10 determinations, made at different times, show therefore that a reduction in the amount of acid presented to the wool from 10 cc. to 6 cc. causes the fibre to absorb less acid, in the proportion of 100:74.3.

To determine the influence of the concentration of the liquid, three amounts of solution were employed, viz.: (a) 333 cc., (b) 666 cc., (c) 1,000 cc., each containing 10 cc. normal  $\text{H}_2\text{SO}_4$ . The amounts of  $\text{H}_2\text{SO}_4$  absorbed by the wool were 0.31605, 0.30331, and 0.27881 gm. respectively; that is, with increasing concentration the amounts absorbed had the ratio 100:108:113. Simultaneous with the last three determinations, the behaviour of wool towards hydrochloric and nitric acids was examined, 10 cc. of the normal acids being employed in each case in 666 cc. water. The amounts absorbed, expressed in terms of  $\text{H}_2\text{SO}_4$ , are as follows:—

For  $\text{HCl}$ , 0.2793 gm.; for  $\text{HNO}_3$ , 0.28175 gm.; that is a little less than in the case of  $\text{H}_2\text{SO}_4$ , a deviation which is possibly due to the volatility of these acids.

Parallel experiments boiling wool with 1 gm. chrome-alum and 6 cc. normal  $\text{H}_2\text{SO}_4$  respectively, i.e., equal amounts of  $\text{SO}_3$ , gave the following amounts of  $\text{H}_2\text{SO}_4$  or  $\text{SO}_3$  absorbed:—

#### For chrome-alum—

- (a.) 0.24696 gm.  $\text{H}_2\text{SO}_4 = 0.2016$  gm.  $\text{SO}_3$ , equiv. to 0.8400 gm. chrome-alum.
- (b.) 0.26068 gm.  $\text{H}_2\text{SO}_4 = 0.2128$  gm.  $\text{SO}_3$ , equiv. to 0.8866 gm. chrome-alum.
- (c.) 0.25676 gm.  $\text{H}_2\text{SO}_4 = 0.2096$  gm.  $\text{SO}_3$ , equiv. to 0.8733 gm. chrome-alum.
- (d.) 0.24813 gm.  $\text{H}_2\text{SO}_4 = 0.2028$  gm.  $\text{SO}_3$ , equiv. to 0.8459 gm. chrome-alum.

#### For sulphuric acid—

- (a.) 0.25382 gm.  $\text{H}_2\text{SO}_4 = 0.2072$  gm.  $\text{SO}_3$ , equiv. to 0.8630 gm. chrome-alum.
- (b.) 0.24696 gm.  $\text{H}_2\text{SO}_4 = 0.2016$  gm.  $\text{SO}_3$ , equiv. to 0.8566 gm. chrome-alum.

These figures are sufficiently concordant to show that the decomposition of the chrome-alum by the wool is really due to the absorption of acid by the fibre.

As previously stated, this decomposition also takes place to a small extent even in the cold, and in accordance with this fact it was found that 10 grms. wool steeped for a month in 600 cc. water containing 10 cc. normal  $\text{H}_2\text{SO}_4$ , absorbed 0.28837 gm.  $\text{H}_2\text{SO}_4$ , equivalent to 0.235 gm.  $\text{SO}_3$ .

We are inclined to share Knecht's view, as expressed in his conclusions, viz., that the acid is not combined permanently with the wool, although it is certainly very difficult to remove the last traces of acid from the fibre. A sample of wool boiled with a solution (600 cc.) containing 10 cc. normal  $\text{H}_2\text{SO}_4$  absorbed 0.3136. When the squeezed wool was extracted as completely as possible with water, and this was neutralised with normal  $\text{NaOH}$ , the wool still lying in the liquid, the latter invariably became acid again after some time, but by neutralising again and again after similar rests, it was possible to show the presence in the liquid of the full amount of acid originally employed. In another repetition experiment, the wool, having absorbed 0.31684 gm.  $\text{H}_2\text{SO}_4$ , was boiled with water for two hours; the liquid was then neutralised with normal  $\text{NaOH}$ , and the operations were repeated till no further quantity of sulphuric acid could be extracted; here all the sulphuric acid absorbed could be accounted for. Indeed in both cases a little more than 10 cc. normal  $\text{NaOH}$  was required, it is namely very difficult to titrate with exactitude in protracted and intermittent operations such as those above, and during the intervening time the indicators may have altered slightly.

#### Absorption of Organic Acids and Cream of Tartar.

The following acids, &c. were compared with each other, and the amounts absorbed by the wool are expressed in terms of sulphuric acid ( $\text{H}_2\text{SO}_4$ ):—

- 10 cc. normal sulphuric acid gave up 0.3386 gm.
- 10 cc. normal oxalic acid gave up 0.3258 gm.
- 10 cc. normal tartaric acid gave up 0.2964 gm.
- 1.88 grms. cream of tartar gave up 0.1536 gm.

Whereas, therefore, oxalic, tartaric, and sulphuric acids are absorbed by the wool in almost equivalent amounts, cream of tartar gives up very much less, namely, 45.4 per cent. only of the amount absorbed from sulphuric acid, this being the mean of five determinations.

In concluding this part of our communication, we venture to say that it is evident from the above-cited experiments that the behaviour of the wool fibre towards metallic salt solutions is very different from that of the cotton fibre.

In the case of cotton mordanted with metallic salts, we understand the fibre to be charged with hydrates or insoluble basic salts, and the mode of mordanting cotton is adapted to the precipitation of these, the fibre merely acting the part of a carrier. With wool it is different; basic salts may certainly be readily precipitated, but the wool thus mordanted gives poor results when dyed. How is this? We hope to deal with this question in our next communication. Meanwhile, it seems evident that the comparative instability of the wool under the ordinary conditions of mordanting imposed, its amide character, the liberation of ammonia, and its strong absorptive power for acids, each play their part in the mordanting process with metallic salts.

(To be continued.)

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Members who desire to read Papers, exhibit Apparatus, &c., or introduce Discussions during Session 1893-94 are requested to give early notice to the Hon. Local Secretary.

### SESSION 1893-94.

Glasgow:—

May 1st.—Prof. E. J. Mills and Mr. Saweys. "On the Action of Alkalis on Gelatin."

Edinburgh:—

April 10th.—Dr. J. B. Readman. "The Effect of the Saline Constituents of Water on the Character of Beer."

Meeting held in the Philosophical Society's Rooms, 207, Bath Street, Glasgow, on Tuesday, March 6th, 1894.

PROF. E. J. MILLS, F.R.S., IN THE CHAIR.

## THE PRODUCTION OF GAS FROM PARAFFIN OILS, AND FROM PURE MEMBERS OF PARAFFIN AND TERPENE SERIES.

BY JAMES F. TOCHER, F.I.C., F.C.S.

THE production of gas from oil, which is receiving special attention at the present time, was first undertaken in 1815, and was carried on successfully for some time, when it was superseded by coal-gas. In 1876 its manufacture was resuscitated for the purpose of lighting railway carriages, and since that time it has been continuously used, not only in the illumination of carriages, but also of lighthouses and buoys. The various forms for the decomposition of the oils were described by Professor Armstrong in his paper on the "Manufacture of Gas from Oil," read before this Society in 1884.

Within the last few years oil-gas has excited the widest interest, owing to its being adopted as an enricher of coal-gas. Two years ago the subject was brought under my notice by Mr. John Mackay, late manager of the Peterhead works, who read a short paper on "Enriching Coal-Gas by Paraffin Oils" at the annual meeting of the North British Association of Gas Managers held at Stirling in July 1892. Owing to the increased price of candles, Mr. Mackay, in common with many others, tried the utility of oil as an enricher. It being undesirable to erect any special plant, he utilized the ordinary  $\square$ -shaped fire-clay retorts, and passed the oil into the empty retort in the form of a spray by injection at high pressure. His results, given in the paper above referred to, justified the trial he had made, but as the oil was decomposed at a far higher temperature than that employed by the Pintsch and other special processes, and gave different results, an investigation into the conditions under which the various oils could be destructively distilled to advantage was suggested to me.

The earliest experiments in oil-gas making were conducted either in narrow tubes or in retorts filled with coke or iron filings, thereby giving a large heated surface in proportion to cubical capacity, and resulted in the production of much tar and little gas.

Dr. Stevenson Macphail's experiments (1872) were conducted in retorts filled with coke, while Lotz (1877) experimented with a tube 4 ft. long and 2 in. diameter. Lieberman and Burg (1878) used tubes 1 m. long and 1 in. diameter, and found that the yield of gas was greater when the tube was empty than when filled with charcoal. Professor Lewis, in his paper on "The Production of Oil-gas from Russian Petroleum" (this Journal, 1892, 584-586), gave the results of cracking petroleum in retorts of various sizes which show the greatest sperm value in using retorts where the surface is small in proportion to the cubical capacity. In carrying out the following experiments three retorts were used:—(1) 36 in.  $\times$  6 in.; (2) 36 in.  $\times$  5 in.; (3) 8 in.  $\times$  3 in., where  $\frac{\text{surface}}{\text{capacity}}$  = 8.6, 10.3, and 14.1 sq. ft. surface per cubic foot respectively. A  $\square$  clay retort was also used, in which the ratio was 3.75. In giving results from these, I shall also compare, in the latter part of the paper, various retorts in use, showing how far the different ratios of surface and capacity, &c., affect the cracking up of oils.

The retorts were set in the furnace with a fall 4 ft. The mouths of the two larger ones were closed by iron plates, which were secured by bolts. A stopcock was placed in the centre of each plate, which served the double purpose of determining the temperature and noting the colour of the gas during decomposition of the oil. The oil was fed into retort by means of a pipe inserted with a bend to upper end of retort, and led to within 2 in. of the lower end, where a small flat sheet of iron was placed in order to provide for the proper dispersion of the oil dropping from the pipe. The gas was passed from upper retort into a small cylindrical vessel, where the residuals were collected (and which was utilised to return residuals to retort in determining the ultimate sperm value of 0.884 oils), thence through small purifier and meter to small holder. The gases from other retorts were passed through separate vessels for collection of residuals and thence to holders. With the exception of fire-clay retort, the temperatures were determined by means of Siemen's water pyrometer, the point selected in the retort being the top of iron plate, on which the oil dropped. The temperature of fire-clay retort was about 1,200° C., or bright orange, that degree being usually employed in the manufacture of coal-gas.

The illuminating power was determined in the usual way, with the exception that suitable burners were selected and the rate adjusted to suit the quality of each gas, the results being then calculated to 5 cubic feet of gas and 120 grains sperm per hour. Each gas was analysed prior to determination of candle power:  $\text{CO}_2$  by  $\text{KOH}$  sol., O by alkaline pyrogallol, olefines, &c., by  $\text{Br}$  (br vapour removed by  $\text{KOH}$ ), and CO by cuprous chloride. A portion of the residual gas was mixed with excess of O and exploded,  $\text{CH}_4$ , H (and N) being estimated after explosion in the usual way. The carbon and hydrogen densities, when such are given, were determined by exploding a portion of the original gas in excess of oxygen and calculating the densities from the following formulae, where—

A =  $\text{CO}_2$  in 100 vols. of gas.

B =  $\text{CO}_2$ , due to  $\text{CH}_4$  (CO and  $\text{CO}_2$  if present) in 100 vols. of gas.

a = O required for complete combustion of 100 vols. of gas.

$\beta$  = O required for combustion of H,  $\text{CH}_4$ , and C of  $\text{C}_n\text{H}_m$  hydrocarbons.

C = Percentage of  $\text{C}_n\text{H}_m$  hydrocarbons in 100 vols. of gas.

$$(1) \frac{A - B}{C} = \text{carbon density:}$$

$$(2) \frac{a - \beta}{C} = \text{hydrogen density:}$$

*Paraffin Oils.*

The first series of experiments were conducted in retort 6 in. × 6 in., with the following grades of paraffin oils,

viz.:—(1) Mineral naphtha, sp. gr. 0.730; (2) burning oil, sp. gr. 0.807; (3) light mineral oil, sp. gr. 0.847; and (4) heavy mineral oil, sp. gr. 0.884. The following results were obtained:—

TABLE I.

Temperature.	Mineral Naphtha. Sp. Gr. 0.730.		Burning Oil. Sp. Gr. 0.807.		Mineral Oil. Sp. Gr. 0.847.		Mineral Oil. Sp. Gr. 0.884.			
	600° C.	850° C.	600° C.	850° C.	600° C.	800° C.	1,100° C.	500° C.	600° C.	850° C.
Cubic feet per gallon of oil ..	72	100	75	93	64	82	95	34	59	108
Colour of gas .....	White	White	White	Brownish-white.	White	Light brown.	Dark brown.	Light yellow.	Light yellow.	Brown.
Composition of gas:—	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
C <sub>12</sub> H <sub>22</sub> hydrocarbons .....	31.2	29.8	32.8	43.1	33.7	46.2	21.5	44.7	49.5	35.7
C <sub>18</sub> H <sub>32</sub> +2 hydrocarbons .....	47.6	48.7	57.0	47.2	50.1	39.4	49.2	40.1	48.2	46.3
Hydrogen .....	17.4	19.1	7.1	7.6	13.7	11.9	28.4	12.7	8.1	16.1
Carbon density of C <sub>12</sub> H <sub>22</sub> .....	2.67	2.71	3.93	2.92	2.88	2.75	2.41	3.11	2.95	2.79
Hydrogen density .....	..	..	..	..	1.87	5.22	5.16	5.38	5.54	4.46
Illuminating power:—										
In candles corrected to 5 cubic feet of gas and 120 grains sperm per hour .....	43.5	42.2	48.7	61.4	52.5	61.8	36.2	63.8	57.7	49.0
Candles per gallon .....	626	844	730	1,112	672	1,062	688	434	681	1,058
Pounds sperm per ton .....	3,292	4,437	3,482	5,447	3,096	4,815	3,119	1,885	2,938	4,596
Enriching value (Table III), pounds sperm .....	..	..	..	6 per cent. mixture gave 10.171. Enrichment figure = 118.	..	5 per cent. mixture gave 6.872. Enrichment figure = 93.5.	..	..	..	..
Percentage of residuals .....	11.4	5.1	21.4	7.5	25.5	12.2	18	62.3	41.5	9.4

An uniform rate of flow was maintained throughout, and was, as nearly as possible, the same in all these experiments. On taking the temperature of the retort from time to time, after decomposition of the oil had been effected, it was found to have fallen on an average 50°–100° C.

The results obtained in decomposing the naphtha at 600° and 800° are similar so far as the composition and candle-power of the gas are concerned, but at 600° the yield was less, giving a smaller sperm value, while the percentage of residuals was double that found at 800° C.

The burning oil at 600° C. gave only 75 cubic feet of 48.7 candle-gas, but on raising the temperature to 800° C. a yield of 93 cubic feet of 61.4-candle gas was obtained. The unsaturated hydrocarbons in the gas had increased by 10.3 per cent., while the volume of residuals had decreased by one-third.

A comparison of the results from 0.847 oil show that any degree of temperature much above 800°–900° C. reduces the sperm value of the gas. At 600° C., 64 cubic feet of 52.5-candle gas were obtained, equal to 659 candles per gallon; at 800° C. a yield of 82 cubic feet of 64.8-candle gas was obtained, equal to 1,062 candles per gallon, while on decomposing the oil in a fire-clay retort at 1,100°–1,200° C. (bright orange), the yield rose to 95 cubic feet, the candle-power being 36.2, equal to 688 candles per gallon, a figure similar to that obtained at 600° C. The percentage of hydrogen in the gas was double that found at 800° C., while the unsaturated hydrocarbons were reduced to less than one-half, showing that a highly heated surface tends to the production of free hydrogen at the expense of the heavier hydrocarbons.

The first experiment with 0.884 oil was performed at a temperature approaching 500° C. The yield was small, only 34 cubic feet per gallon, while the percentage of residuals amounted to 62.3 per cent. The candle-power was high, viz., 63.8, and the gas contained 44.7 per cent. of heavy hydrocarbons with a carbon density of 3.11. At 600° C. the yield rose to 59 cubic feet per gallon, while the

candle-power fell to 57.7, and the residuals to 41.5 per cent. A yield of 108 cubic feet of 49-candle gas was obtained at 850° C., equal to 1,058 candles per gallon. The residuals amounted only to 9.4 per cent.

The tar obtained from the decomposition of naphtha at 800° C. was fractionally distilled, 35.1 per cent. distilling over between 70°–100° C., while 39 per cent. of the naphtha itself distilled over between 60°–100° C.; burning oil-tar gave 23 per cent. up to 100° C., 0.847 oil-tar at 800° C. gave 13.5 per cent., 0.884 oil-tar at 600° C. gave 1.1, and 6 per cent. at 850° C.; while nothing was obtained from 0.847 oil-tar at 1,100° C. until 190°–200° C.

From the results given in Table I., it is apparent that by the method of decomposition employed, the degree of temperature giving the highest sperm value ranges from 800°–850° C. In the Pintsch, Patterson, and Keith retorts, where the method of cracking the oil is similar in principle to that employed in these experiments, the temperature is higher, viz., 900°–1,000° C., while the sperm values obtained are lower. It is clear that if a temperature lower than 800° C. was adopted in practice, the conditions under which the oil is to be decomposed must be entirely different. It is the special feature of the new process of manufacturing oil-gas patented by Messrs. Young and Bell last year that a low temperature is employed, while the residuals (which we have seen from results at 600° C. amount to over 60 per cent.) are run back into retort to be decomposed. The actual temperature employed is, I believe, lower than 600° C., and therefore a larger volume of material must pass over unchanged—at least ungasified, but as it is returned to retort, the whole of the material is utilised, with the result that there are no liquid residuals, but instead, a solid residue is left in retort.

The following experiment, undertaken to ascertain the sperm value obtainable when a sample of oil is wholly converted into gas and solid residue, has also afforded an opportunity of expressing the decomposition of the oil in clearer terms. The cylindrical vessel, which in the previous

PERCENTAGES OF OILS AND RESIDUALS DISTILLING EVERY 1° FROM 60°—200° C.

	60	70	80	90	100	100-110	110-120	120	130	130-140	140	150	150-160	160	170	180	190	200
Naphtha 0.730...	6	4	14	15	15	17	11	8	10	..	..	..	..	..	..	..	..	..
Naphtha residual at 800° C.	..	7	21	7	3.5	3	2.5	2	1.5	..	..	..	..	..	..	..	..	..
Burning oil 0.803	..	..	..	..	..	..	..	..	..	2	4	7	7	..	..	..	..	..
Burning oil residuals at 850° C.	..	3	2	18	9	4	3.8	3.5	3	3	4	2.2	1.8	1.1	..	..	..	..
0.847 mineral oil.	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	2
Residuals at 800° C.	..	..	2.5	11	2.5	2.5	2.5	2.5	2.5	3	2.8	2.5	2	2.5	3	4	..	..
Residuals at 1,100° C.	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	2	2	..
0.884 mineral oil.	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..
Residuals at 600° C.	..	..	..	1.1	1.5	2	1.6	1.5	1.5	1.5	1.5	1	..	..	..	..	..	..
Residuals at 850° C.	..	..	1	5	4.7	4.5	4	4	3.8	3.7	3.7	4.5	4	3.5	..	..	..	..

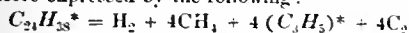
experiments served also as a washer, was utilised. A small pipe was fixed to lower edge of vessel and carried to a point about an inch above funnel through which the oil was introduced to retort. Eight pounds of 0.884 mineral oil were placed in the vessel. The retort being heated to about 600° C., oil was allowed to pass into retort by opening stopcock attached to end of pipe. No fresh oil was introduced; the oil, mixed with tar formed, being simply allowed to flow into retort at slow rate until the whole was converted into gas and solid residue. The candle-power of the first portion of the gas was 66.1, while that of the final portion was 51.5. The specific gravity of the oil, taken three times during the course of the experiment, was successively 0.889, 0.881, and 0.895. A yield of 81 cubic feet of 61.1-candle gas was obtained, equal to 1,094 candles per gallon, or 131,280 grains sperm per gallon. This may be conveniently termed the ultimate sperm value, seeing that it is the highest attainable figure in experimentally converting the whole of the oil into gas and carbon. There was a solid residue of 24.9 per cent.; the percentage composition of the gas was as follows:—

	Per Cent.
CnHm hydrocarbons .....	42.9
CnHn+2 .....	43.5
Hydrogen .....	13.3

Carbon density = 2.89, hydrogen density = 5.12. In order to trace out the decomposition of the oil, the percentages of carbon and hydrogen present in the oil were determined by combustion. The following is the mean of two determinations:—

	Per Cent.
Carbon .....	86.68
Hydrogen .....	11.67

The average molecular weight of the substances of which the oil is composed, was determined by noting the depression of the freezing point of benzene. Two determinations were made, the figures being 318 and 322. The average formula for the oil is therefore represented by  $C_{24}H_{38}$ . On converting the results obtained from cracking up the oil into per cent. by weight of oil taken, the following figures are obtained:—Hydrogen 0.7, methane 19.8, CnHm 50.7, carbon 24.9, and loss 3.9. The decomposition of the oil is therefore expressed by the following:—



	Theory.	Found.
	Per Cent.	Per Cent.
H <sub>2</sub> .....	0.6	0.7
4CH <sub>4</sub> .....	19.6	19.8
4(C <sub>3</sub> H <sub>8</sub> ) .....	50.3	50.7
4C <sub>2</sub> .....	29.5	24.9
Loss .....	..	3.9
	100.0	100.0

\* These denote no definite chemical compounds, but simply average formulae.

The heavier mineral oils are said to consist almost entirely of olefines and pseudo-olefines. The average molecular formula obtained for 0.884 oil, although it affords no proof as to the constitution of the components of the oil, tends to bear out that the oil consists of compounds rich in carbon, like the olefine and acetylene series. The difference in percentage composition between these oils and various paraffins and terpenes is not very great, as is shown by the following:—

	Paraffins.		Turpentine Oil.	Olefines.	0.884 Mineral Oil.
	Octane, C <sub>8</sub> H <sub>18</sub>	Decane, C <sub>10</sub> H <sub>22</sub>	C <sub>9</sub> H <sub>18</sub>	C <sub>10</sub> H <sub>18</sub>	C <sub>10</sub> H <sub>16</sub> (found.)
C	84.2	84.5	85.2	88.2	86.68
H	15.8	15.5	14.8	11.8	11.67

Octane, Decane, and Turpentine Oils.

The experiments with octane, decane, and turpentine were undertaken in order to ascertain how far the results differed, in destructively distilling at a moderately high temperature, liquids containing approximately the same percentages of C and H, but widely differing in constitution and in complexity of molecular structure. Octane, prepared from octyl iodide and having a boiling point of 122° C., was decomposed at 600° C. and 800° C. in the 8 in. × 3 in. retort. Decane from iso-amyl iodide, and boiling at 156.5° C., was decomposed in a similar manner. Turpentine oil (portion distilling = 92 per cent. 154°—158° C.) was decomposed in 6 in. × 36 in. retort. The results are embodied in Table II.

Norton and Andrews, on decomposing pentane and hexane, by passing the vapours of these paraffins through a red-hot tube, found that decomposition set in about 600° C. The products obtained at that temperature were ethylene, propylene, butylene, and paraffins. Benzene was formed from hexane at higher temperatures. From the results on Table II., it will be seen that, while octane gave at 550° C. 23 per cent. of unsaturated hydrocarbons, equivalent to 30 per cent. of ethylene, at 800° C. only 12.3, equivalent to 11.7 per cent. ethylene, were obtained. Similarly, decane at 550° C. gave 27.4 unsaturated hydrocarbons, equivalent to 32.1 per cent. ethylene, and at 800° C. 13.4, equivalent to 14.2 per cent. ethylene. These results bear out that at low temperatures octane and decane are decomposed into ethylene and higher olefines, methane, and hydrogen, while at high temperatures no higher olefines are formed, the gaseous products being simply ethylene, methane, and hydrogen.

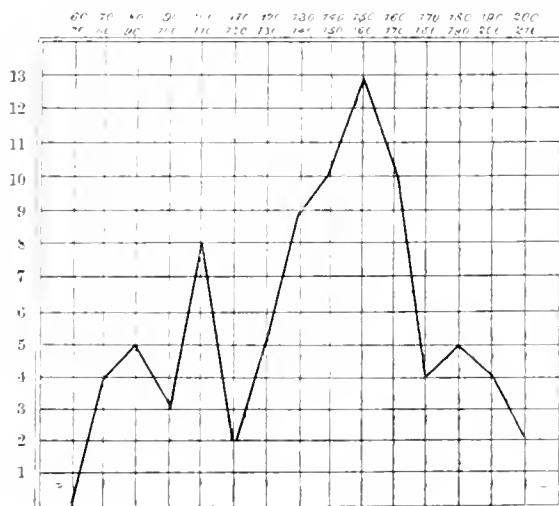
It has been shown by Professor Tilden (Journ. Chem. Soc. 1884, page 410) that the products obtained from

TABLE II.

Temperature.	Octane, $C_8H_{18}$ .		Decane, $C_{10}H_{22}$ .		Turpentine Oil, $C_{10}H_{16}$ .	Naphtha, $C_{10}H_{18}$ .	Mineral Oil, 0.884.	
	Boiling Point, 122° C.		Boiling Point, 156.5° C.		Boiling Point, 154°-158° C.	0.730.		
	550° C.	800° C.	550° C.	800° C.	800° C.	850° C.	500° C.	850° C.
Yield.....	18 litres per 100 cc.	12 litres per 100 cc.	21.6 litres per 100 cc.	17.5 litres per 100 cc.	63.3 c.f. per gall.	10.6 c.f.	34 c.f.	0.108 c.f.
Colour of gas.....	White.	White.	White.	White.	White.	White.	Light yellow.	Brown.
Composition of gas—								
$C_nH_{2n+2}$ hydrocarbons.....	23.5	12.3	27.4	13.4	19.1	20.8	44.7	35.7
$C_nH_{2n+4}$ hydrocarbons.....	39.4	35.4	39.0	50.1	51.1	48.7	40.1	46.3
Hydrogen.....	35.7	52.8	35.7	36.5	29.8	19.1	12.7	16.1
Ethylene equivalent of $C_nH_m$ .....	3.00	11.7	32.4	44.2	30.4	55.2	69.5	49.8
Carbon density.....	2.56	1.91	2.35	2.42	3.20	2.71	3.11	2.79
Illuminating power, in candles per 5 c.f.	18.0	..	20.2	12.0	39.1	42.2	63.8	49.0
Residuals, per cent.....	22.0	A few drops	50.0	None	16.6	5.1	62.3	9.4
			Composed almost entirely of unbranched Decane.					

ordinary turpentine oil at a low red heat, consist chiefly of *pinene*, *heptene*, *metaxylene*, *cymene*, and *terpene*, with small quantities of benzene and toluene. The results embodied in the foregoing table show that at 800° C., turpentine oil is capable of yielding a large volume of gas, 63.3 cubic feet, with an illuminating value of 39.1 candles, having been obtained. The residuals amounted to 16.6 per cent., and when fractionally distilled gave a total distillate up to 100° C. of 12.2 per cent.

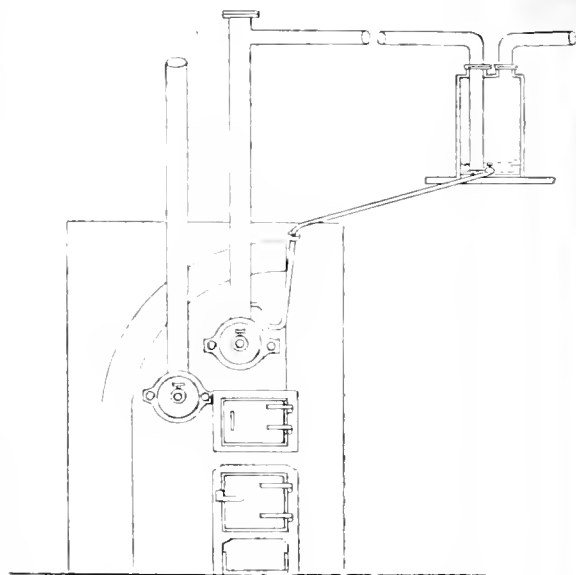
The percentages which came over every 10° degrees up to 210° C. are shown by the following diagram:—

PERCENTAGE OF RESIDUE FROM TURPENTINE OIL,  
DISTILLING EVERY 10° FROM 60° TO 210° C.

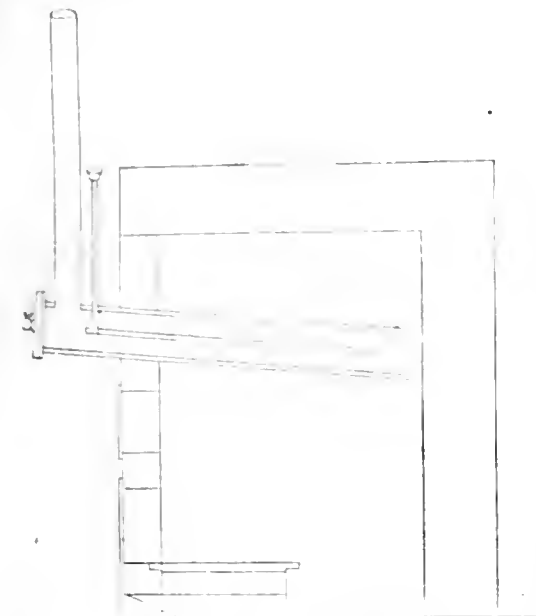
The portion distilling below 106° C. was nitrified, the amount of benzene found being 3.7 per cent. of total residue. The distillate between 110°-120° C. consisted principally of toluene, while the portion distilling between 130°-170° C. (42.2 per cent.) is due to *cymene* and isomeric *terpene*.

Summarising the results on Table II., it has been found that on submitting members of paraffin, olefine, and terpene series to destructive distillation about ordinary temperature

employed in oil-gas manufacture, they tend invariably to split up into simpler members, with the evolution of gaseous members of the olefine and paraffin series and free hydrogen. The rupture between carbon and hydrogen is greater as the temperature rises. A glance at the percentages of

FORM OF APPARATUS USED IN DETERMINING ULTIMATE  
SPERM VALUE.

unsaturated hydrocarbons found will show what each series is capable of. Lower paraffins give a smaller percentage than terpenes, while terpenes again have a less percentage than higher paraffins. As might naturally be expected, the percentage is highest in liquids where the higher olefines form the greater part. From this circumstance, heavy mineral oils are more suitable for oil-gas making than Russian petroleum, and the latter than American petroleum, the olefines being highest in Scotch oil, next in the Russian, and least in the American.



FORM OF APPARATUS USED IN FIRST SERIES OF EXPERIMENTS.

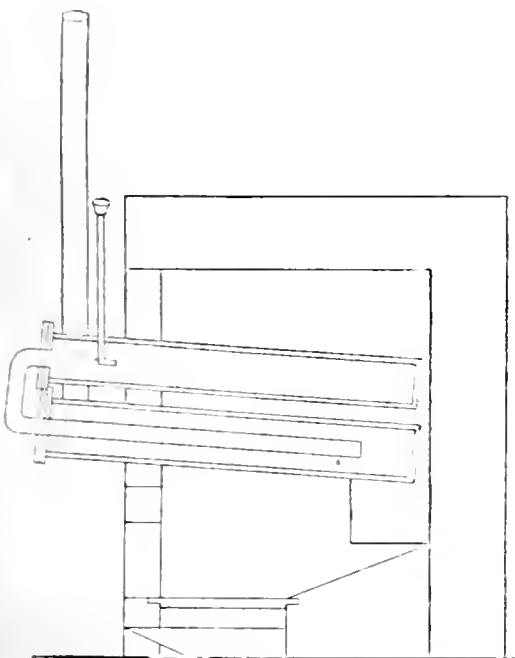


DIAGRAM SHOWING UPPER AND LOWER RETORTS CONNECTED.

#### Oil-Gas Enrichment.

In 1884, Professor P. F. Frankland read a paper on "The Illuminating Power of Ethylene when Burnt with Non-Luminous Combustible Gases" (*Chem. Soc. Jour.* 4530). He determined the illuminating value of pure ethylene when burnt alone to be equivalent to 68.5 candles per five cubic feet, but on mixing ethylene with various proportions of hydrogen and methane, he found that the intrinsic

illuminating power of the ethylene (calculated to five cubic feet per hour irrespective of the composition of the mixture) was much greater. When marsh-gas was used as a diluent, the intrinsic value of ethylene did not increase to any extent until the proportion present in the mixture reached 50 per cent., when the value rapidly rose as the proportion of methane was increased until 90 per cent. had been added, when the intrinsic value of ethylene reached 170-180 candles.

This fact is of great value when it is desired to enrich a poor coal-gas by the addition of oil-gas of high illuminating power. The proportion employed in practice where oil-gas is used to enrich coal-gas is about 15-20 per cent., and in order to determine the intrinsic value of oil-gas, experiments were made, using varying proportions of oil and coal-gas. The illuminating values of oil and coal-gas being separately determined, various known proportions were run into the large experimental holder. Prior to determining the candle-power each mixture was allowed to stand 14-18 hours to allow complete diffusion of the mixed gases.

The following results were obtained (for Table III, see next page).

#### Size and Shape of Retorts.

It will perhaps be of interest to glance into the conditions (other than temperature and nature of oil used) affecting the production of gas from oil. It is evident that the following factors enter into the question. (1.) Temperature of retort (affected by rate of flow and diameter of retort). (2.) Rate of flow into retort (the length of retort, and temperature modify the effect of rate of flow). (3.) Length of retort in proportion to its diameter (the time during which the vapours and gases are exposed to heat being of course directly proportional to length of retort). (4.) The ratio of total surface to cubical capacity. (The temperature in the centre of a narrow retort corresponds closely to surface temperature. Given a proportionate length, and the wider the retort is, the further is the divergence of temperature between centre and surface, owing to surface exposed being less in relation to cubical capacity.) (5.) The oil used.

The following experiment is an illustration of how a continuous and uniform rate of flow, by reducing the temperature in the interior of retort, affects the composition yield and candle-power of the gas. The oil was sprayed into the fire-clay retort at a rate of 0.42 gallon per minute. The gaseous products at the end of the first, third, and fifth minutes were examined separately, with the following results:—

	Yield.	Candle Power.	Composition of the Gas.		
			CuHm.	CuHn <sub>2</sub> + 2	H.
	Cub. Ft. per Gall.				
End of first minute.	126	24.7	10.5	47.7	41.8
End of third minute.	93	20.5	15.3	50.6	34.1
End of fifth minute.	79	16.6	28.7	48.1	23.1

The effect of different rates of flow is shown by the following:—

Retort.	Time.	Amount of oil used.	Yield per Gallon.	Candle Power.
36 in. x 6 in.	Mins.	Gallon.	Cub. Ft.	
	60	0.25	26	41.1
"	18	0.25	71	58.7

In order to note the effect of prolonging the action of heat on the vapours and gases produced by running the oil into upper retort, an experiment was tried where the upper and

TABLE III  
OIL-GAS IN VARIOUS PROPORTIONS OF COAL-GAS.

Proportions.		Candles observed.	Rate.		Candle-Power at 5 Cubic Feet per Hour and 120 Grains Sperm.	Theoretical Figure for the Mixture, Calculated from Observed Candle- Power of Separate Gases	Enrichment Figure found = Intrinsic Illuminating Value of Oil-Gas $P_1 = \frac{P_3 - P_2}{P_1 - P_2} + P_3$	Increase in Candle-Power Per Cent. of Oil-Gas taken $C_1 = \frac{P_3 - P_2}{P_1}$
Oil-Gas.	Coal-Gas.		Gas per Hour.	Grains Sperm per Hour.				
100	..	11.22	2.08	117	32.8	..	..	..
..	100	10.20	5.00	115	19.5	..	..	..
75	25	10.25	2.30	125	45.4	44.2	55.3	0.36
60	40	10.84	2.55	121	42.9	39.3	58.5	0.39
40	60	7.40	2.12	118	34.2	32.7	56.2	0.57
24	80	9.32	3.02	117	29.9	26.1	71.5	0.52
13	87	11.94	4.42	122	27.4	24.8	80.2	0.62
100	..	..	..	..	57.7	..	..	..
..	100	..	..	..	20.5	..	..	..
5	95	12.4	5.00	118	24.3	22.3	96.5	0.76
100	..	..	..	..	61.4	..	..	..
..	100	..	..	..	20.5	..	..	..
6	94	10.5	4.01	121	23.4	22.9	118.8	0.48

$P_1$  = candle-power of oil-gas

$P_2$  = candle-power of oil-gas as shown by enrichment.

$P_3$  = candle-power of coal-gas.

$P_4$  = candle-power mixture found.

$P_5$  = theoretical candle-power of mixture.

$y$  = coal-gas taken.

$x$  = oil-gas taken.

$C_1$  = candles per cent. of oil-gas.

$$P_5 = \frac{(x \times P_1) + (y \times P_2)}{x + y}$$

$$P_4 = \frac{y(P_3 - P_2)}{x} + P_3$$

lower ones were connected by means of a pipe passing to back of lower one. In this case the ratio between surface and capacity was little altered from first series of experiments, but the time during which the vapours were exposed to the action of heat was doubled. Two experiments were made, (1) with 0.884 oil, and (2) with a sample of coal-tar (splint), sp. gr. 1.17, the rate of flow in each case being the same. The results obtained were as follows, the results from 0.884 oil at 800° C. (given in Table I.) being inserted for comparison:—

Temperature.	0.884 Mineral Oil.		Coal Tar.	
	800° C.	800° C.	960° C.	960° C.
No. of retorts used	1	2	1	2
Yield per retort, .....	108	119	19	51
Percentage composition of gas CO <sub>2</sub>	..	..	..	0.6
Calim. ....	35.7	13.5	17.7	8.8
Calim <sub>2</sub> + 2 .....	46.3	51.6	50.1	34.5
H <sub>2</sub> .....	16.1	34.9	25.7	45.4
CO .....	..	..	5.2	8.2
Candle-power .....	19.0	29.9	..	22.5
Per cent. residuals	..	..	35 per cent. liquid residuals and some pitch.	62.5 per cent. solid residue (coke).

The following table shows the relationship between (1), length and diameter; (2), surface and capacity of some retorts used experimentally and in practice.

	Lewes's Experiments.			Pintsch.
	Patterson. 3 6" x 19.5"	10" x 4"	4" x 2"	2 (76)" x 10"
Length = $h$	4.4	2.5	27	15.2
Diameter = $2r$	..	..	..	..
Sq. ft. of surface per cub. ft.*	5.6	14.4	24.4	5

	Young.		Own Experiments.	
	9" x 2 5/8"	3" x 8"	6" x 36"	(13" x 17 1/2" x 8 1/2")
Length = $h$	4	2.6	6	(6.7)
Diameter = $2r$	..	..	..	..
Sq. ft. of surface per cub. ft.	2	19	8.6	(6.37)

$$\text{* Ratio: } \frac{\text{Total surface of cylinder}}{\text{Total capacity}} = \frac{2\pi rh + 2\pi r^2}{\pi r^2 h} = \frac{2(h+r)}{r^2 h}$$

The results given by Professor Vivian B. Lewes (in his paper on Russian petroleum previously referred to) clearly show the effects of varying the forms of retorts.

It is evident that increasing the length of a retort affects the products of decomposition equally as much as increasing the ratio of square surface to cubical capacity, while the latter ratio is of course little altered. In the Patterson and Young retorts the surface ratios are smallest, while in the Pintsch and other double retorts used in practice, the length



ratio is largest, rendering a large rate of flow necessary for the production of rich gas.

In conclusion I have to express my thanks to Mr. Mackay, lately gas manager at Peterhead, for the facilities afforded in undertaking these experiments, and in a similar manner to his successor, Mr. James Kincaid, for affording me every facility and assistance in carrying them out.

## NOTES ON THE OXIDATION OF ANILINE. (I.)

BY PROF. E. J. MILLS, F.R.S., AND  
WALTER MACFARLANE.

ALTHOUGH the oxidation of aniline has been for many years carried out upon the large scale, it has never been submitted to continuous measurement. The investigation proves, in fact, to be beset with serious difficulties, which we can by no means pretend to have entirely overcome; but we have succeeded in eliciting certain results which may perhaps be of interest.

The substance which we in the first place selected as an oxidiser was mercuric nitrate, one of the substances by which magenta was at one time prepared in a technical scale from aniline. Our sample was made by Morson of London; and was found by a mean of several determinations to have the percentage composition—

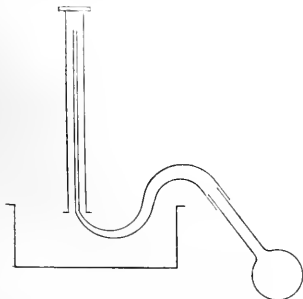
Mercuric oxide.....	70.02
Nitric anhydride.....	28.68
Water.....	2.13
	100.83

Mercury was estimated by Hempel's process. Nitric anhydride was determined by treatment with excess of hot baryta-water, passing carbonic dioxide into the solution, filtering, and precipitating the filtrate with hydric sulphate: from the weight of the precipitate, the weight of nitric anhydride could be calculated. Water was estimated by heating to 120° with pure litharge in a current of dry air.

The aniline which we employed had been distilled out during the manufacture of aniline by the arsenic process. Of this, 93 per cent. was volatile at 110°—183°; and the first third of this, an additional distillation, constituted the stock which we used for our experiments.

We had occasion to observe that either pure aniline or "aniline for red," when heated with arsenic pentoxide or mercuric chloride, always gave rise to a little ammonia. Also, on distilling pure aniline with ammoniac nitrate, we readily found that aniline can expel ammonia from its salts.

The measurement of the amount of any one colour formed during the action of mercuric nitrate on aniline is practically out of the question, the reaction being complicated by the variety of coloured products. Fortunately, however, we noticed that nitrogen was always evolved. We therefore arranged the following apparatus for investigating the reaction by collecting this gas under varied conditions as to mass of nitrate.



The apparatus consisted, as shown in the figure, of a cohobation-flask having a long neck, ground into a wider S-tube, itself terminating in a quill delivery; the gas was collected over a pneumatic trough in a graduated cylinder, partially occupied at the outset by air.

In each experiment, 20 cc. (= 20.601 grms.) of aniline were used, the amount of nitrate being varied. The two substances were allowed to remain in contact for 40 minutes, and the necessary readings of volume of air, barometer, and thermometer were made. The aniline was then heated to cohobation (but not so as to pass into the bend) for 210 minutes; and, after remaining over night, the readings were repeated. The results were as follow:—

TABLE I.

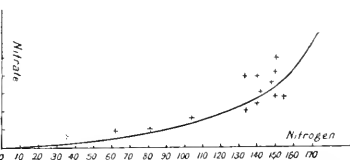
No.	Nitrate.	Normal Nitrogen.	No.	Nitrate.	Normal Nitrogen.
	Grms.	Cc.		Grms.	Cc.
1	0.5533	55.8	9	3.0176	111.6
2	0.8101	61.3	10	3.4532	116.4
3	1.0179	83.3	11	3.9183	130.3
4	1.4883	104.6	12	4.0077	133.4
5	2.1132	134.9	13	4.0733	131.0
6	2.4988	138.9	14	4.9929	152.4
7	2.9767	154.3	15	5.7243	137.6
8	2.9067	150.9	16	6.7436	117.8

During the cohobations the colour of the aniline changed to orange, deep orange, and intense mauve-purple. Abundance of mercury was liberated. The product of the reaction was always freely fluid, with more or less discrete solid in suspension. It was evident that there was always an excess of aniline.

The table shows that the interaction, somewhat energetic at first—becomes slower in proportion as the nitrate increases. Experiments 15 and 16 seem to indicate that, when the nitrate exceeds a certain amount, it acts as a retarding agent to its own normal effect. There is clearly a useful limit to the amount of oxidiser that can be used, even with "excess" of aniline. This also is noticed in the ordinary method of manufacturing magenta. In the annexed drawing we have plotted out our experiments and drawn through them the curve.

$$y = \frac{157.8}{1 + 7.5x}$$

(y being nitrogen and x nitrate).



The curve is calculated from the range 3—14 inclusive, the first and last two experiments evidently being not fairly within what we may term the "normal" interaction. The probable error of a single experiment is 4.3 cc.; and reflects well the known difficulty of obtaining regular results as between a solid and a liquid.

According to the equation, no amount of nitrate would in a "normal" experiment, have produced more than 229.3 cc. of gas. The useful limit seems to be when  $\text{Hg}_2\text{N}_2\text{O}_6$  gives rise to an evolution of  $1\frac{1}{2}$  N, which according to the same equation, would correspond to 3.3099 grms. nitrate and 149.2 cc. of gas.

As regards the actual chemical change, we may suppose that  $\text{Hg}_2\text{N}_2\text{O}_6 = \text{Hg} + \text{O}_2 + \text{N}_2\text{O}_4$ . One of the products of

the action of nascent oxygen on the aniline would be water; and one of the products of the action of nitric peroxide and aniline would be a diazo compound. Thus, nitrogen and phenol would be eventual products.

The failure of this process technically was doubtless due in the main to the expense of the reagent. We can now see, however, that the loss of nitrogen and the large excess of aniline required to be present throughout, to say nothing of collateral nitrations and other reactions, must in any event have led to its abandonment.

## NOTES ON THE OXIDATION OF ANILINE. (II.)

BY PROFESSOR MILLS AND GEORGE WATSON, JUN.

A sample of purified aniline from the stock referred to in the preceding note, was cohobated for six hours with mercuric oxide in order to destroy residual toluidine (if any); it was then distilled in a current of steam and dried over caustic potash.

Pure mercuric oxide was prepared by dissolving very pure mercury in aqueous hydric nitrate, and precipitating with caustic soda freshly made from sodium; the washing was for the most part performed in a silver basin. Of two preparations, that used in our first series of experiments was found to contain 0.1 per cent. of impurity, consisting of lime, silica, and alumina; the one used in our second series contained 0.07 per cent. of impurity, chiefly consisting of argentic oxide.

In the course of some qualitative experiments, we found that when mercuric oxide is treated with aniline, neither nitrogen nor ammonia is formed. The principal product was a brown body, apparently of an induline character. Associated with this, when small quantities of oxide were used, were tangible amounts of a red product, which did not seem to be formed when the mass of the oxide was large.

We measured the course of oxidation in the following manner:—Into a flask of about 50 cc. capacity, 10 cc. of aniline were always introduced, and a known but varied amount of oxide. A cohobating tube was attached to the flask, which was then heated for  $3\frac{1}{2}$  hours continuously; the time being reckoned from the commencement of ebullition. The contents of the flask were then digested with alcohol in the cold, and further diluted to a convenient depth of tint. The relative amounts of brown colouring matter were ascertained by colorimetric comparison with one such solution taken as an arbitrary or intrinsic standard (100). These comparisons were made by means of an instrument essentially identical with the "detached colorimeter" designed by one of us (Phil. Mag. June 1879). We found no difficulty in attending to the brown colour solely when making these estimations.

TABLE I.

No.	Oxide.	Colour.	No.	Oxide.	Colour.
	Grms.			Grms.	
1	6.2923	126.9	7	2.5059	58.7
2	5.9709	100.0	8	2.0219	38.8
3	5.1700	102.0	9	1.5490	36.6
4	4.1145	67.6	10	1.1823	24.0
5	3.5410	60.9	11	0.8520	16.9
6	2.9933	55.3			

The results in this table, if graphically represented, lie approximately on a straight line—

$$\text{Oxide} \times 19.55 = \text{colouring matter.}$$

The equation has a probable error of 5.9. We are disposed to regard this set of experiments as somewhat abnormal on account of the presence of water or (which

would come to the same thing) a more easily oxidable matter than aniline. We had, in fact, noticed that the cohobation was unduly concussive.

In the following set of experiments we used another sample of aniline, and ebullition went on quietly.

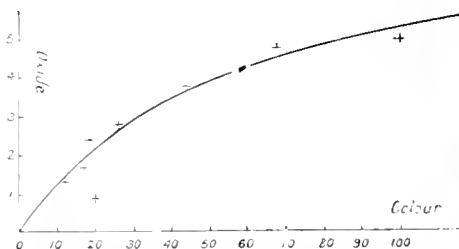
TABLE II.

No.	Oxide.	Colour.	No.	Oxide.	Colour.
	Grms.			Grms.	
1	5.1700	100.0	5	2.4359	18.8
2	4.8909	67.4	6	1.7495	17.1
3	3.8554	43.8	7	1.3233	12.4
4	2.8188	24.3	8	0.9179	19.7

The results correspond to a hyperbola, whose equation is—

$$y = \frac{6.73x}{1 - .12x}$$

( $y$  = colour,  $x$  = oxide). We subjoin a drawing of this curve, and have plotted round it the experimental quantities. The probable error of a single determination is 5.2.



The equation has a critical value when  $x = 8.3$ . The aniline and oxide are then very nearly in the proportion,  $3C_6H_5N:11HgO$ , and any excess of oxide above this would lead theoretically to a negative result.

In the experiments with the nitrate, it will be remembered that the useful limit appeared to be  $3HgN_2O_6:4N$ . The mathematical relations of the curves show that one of these reactions is distinctly inverse to the other.

## DISCUSSION.

The CHAIRMAN said that arsenic oxide should still be used in making magenta on the large scale, was interesting because it was undoubtedly the first of the methods discovered by Dr. Medlock. It seemed remarkable that oxide of mercury should not evolve nitrogen from aniline, and he asked if other oxidisers behaved similarly? Could Professor Mills give any details regarding the brown substance which he had observed?

Professor MILLS had not separated the brown substance. It was so mixed with other colouring matters as to make its separation a very difficult operation. The research was rather intended as an instance of the mode of measuring, on a small scale, a technical operation. Stirring was effected by means of cohobation, while in the technical operation a screw-stirrer is employed in a horizontal cylinder. So far as he had been able to ascertain, nitrogen had never been observed in this manufacture.

Discussion on Mr. Tocher's paper was postponed until May 1st.

## Journal and Patent\* Literature.

Class.	Page
I.—General Plant, Apparatus, and Machinery .....	239
II.—Fuel, Gas, and Light .....	241
III.—Destructive Distillation, Tar Products, &c. ....	243
IV.—Colouring Matters and Dyes .....	244
V.—Textiles: Cotton, Wool, Silk, &c. ....	248
VI.—Dyeing, Calico Printing, Paper Staining, and Bleaching .....	248
VII.—Acids, Alkalis, and Salts .....	249
VIII.—Glass, Pottery, and Enamels .....	251
IX.—Building Materials, Clays, Mortars and Cements ..	251
X.—Metallurgy .....	255
XI.—Electro-Chemistry and Electro-Metallurgy .....	255
XII.—Fats, Oils, and Soap Manufacture .....	257
XIII.—Pigments and Paints; Resins, Varnishes, &c.; India-Rubber, &c. ....	262
XIV.—Tanning, Leather, Glue, and Size .....	263
XV.—Manures, &c. ....	263
XVI.—Sugar, Starch, Gum, &c. ....	263
XVII.—Brewing, Wines, Spirits, &c. ....	265
XVIII.—Chemistry of Foods; Sanitary Chemistry and Water Purification; Disinfectants .....	269
XIX.—Paper, Pasteboard, &c. ....	—
XX.—Fine Chemicals, Alkaloids, Essences, and Extracts ..	272
XXI.—Photographic Materials and Processes .....	276
XXII.—Explosives, Matches, &c. ....	276
XXIII.—Analytical Chemistry .....	277

## I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

*Improved Method of Setting Up Vessels for Boiling.*  
G. A. Mangoldt. Chem. Zeit. 17, 1899.

WHEN vessels are employed in chemical manufactures heated by direct firing, a frequently occurring inconvenience is that the substances heated are apt to "burn" on the bottom, thereby leading to considerable loss and damage to the rest of the product. To avoid this, the vessel may be conveniently heated by means of one or more circular flues round the sides, through which the flame plays before reaching the bottom. In this way the temperature of the gases is sensibly reduced before they come in contact with the lowest part of the boiler, where dregs and solid impurities, &c., naturally tend to settle; the chance of burning on the sides of vertical and conical boilers is much less than on the bottom. Cylindrical vertical distilling apparatus thus heated are found to form markedly lessened amounts of products of decomposition by overheating; whilst conical soap kettles heated by direct firing, produce a soap less browned and otherwise deteriorated in quality by the burning of insoluble limesoaps, &c., on the bottom. In order to collect such precipitated impurities, the base of the kettle may conveniently be provided with a "hat," or small well at the base, protected from direct contact with the flame. The solid dregs, &c., settle into this. The same arrangement answers well when saline matters "salt out" in consequence of concentration by evaporation.

—C. R. A. W.

*Use of Asbestos for the Filtration of Sugar, Juices, and Syrups.* P. A. Maignen. Bull. de l'Assoc. des Chim. 10, 881.

See under XVI., page 264.

\* Any of these specifications may be obtained by post by remitting 8d.—the price now fixed for all specifications, postage included—to Sir Henry Reader Lack, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

## PATENTS.

*Process for the Distillation of Substances having a High Boiling Point.* Benno Jaffé and Darmstaedter, Charlottenburg, Berlin. Eng. Pat. 1468, January 23, 1893.

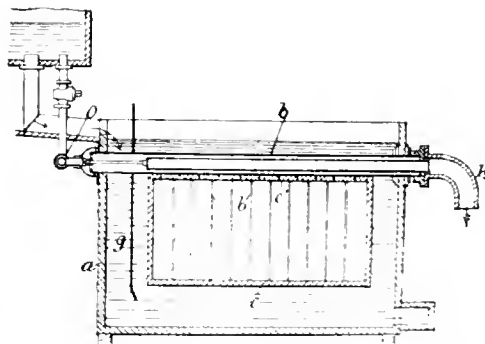
THE use of superheated steam for distilling substances not readily volatilised is general, partly because of the assumption that the steam vaporises the particles of the substances that come in contact with it, envelopes the same, and carries them along with it, so as to effect distillation at a temperature below the boiling-point of the substance treated; and partly because it is supposed that if air were employed instead of steam, oxidation or decomposition would be more readily brought about. This, however, is incorrect as regards glycerol; by passing air through glycerol, distillation can begin at 120° C., and is active at 170–180°; the distillate obtained is purer, as no decomposition is brought about at the lower temperature used; the yield is quite as large as that obtained by means of steam. The current of air is propelled by any suitable air-forcing apparatus, the glycerol being heated from the outside. The distillate is free from aqueous particles, and is collected in suitable refrigeration recipients, one or more of the last of which contains some water, so as to remove the last traces of glycerol from the escaping air which forms the vehicle. Aniline and other substances can similarly be readily distilled in a current of any permanent gas that has no detrimental effect upon the substance treated.—C. R. A. W.

*Improvements in and relating to Filtering Apparatus.*  
H. J. E. Jensen, Hamburg, Germany. Eng. Pat. 1780, January 26, 1893.

THE patented improvement refers to filters in which the filtering material, such as asbestos and other fibrous material, is in the first instance mixed with the liquid and deposited upon perforated screens by the aspirating action of the liquid as it is drawn through the said screens. The improvement consists in causing the liquid to approach and pass through the screens at an equal speed over all the exposed surfaces, with a view to obtaining an equal thickness of filtering material throughout, and to preventing its disturbance or washing away afterwards.

For this purpose the inventor proposes to apportion the areas of the holes through the tubes which carry away the filtered liquid, in such a way as to induce equal currents through all parts of the filtering surfaces, one method of arranging them being shown in the accompanying illustrations.

Fig. 1.



*a* is an open cistern into which the liquid to be filtered is run from the shoot *a*, the partition *g* causing it to descend and to reach the filtering units *e* from below. The units consist of frames covered with wire netting, hung from the pipes *b*, with which they communicate through the openings *b*, *c*.

Within the pipe *b* is secured centrally a second pipe *i*, closed at one end and communicating at the other with the outlet opening *h*.

Fig. 2.

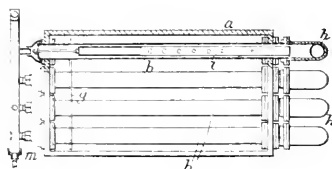
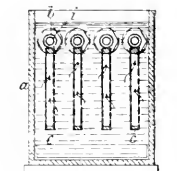


Fig. 3.



The inner pipe is perforated with openings diminishing in area towards the outlet end, which is claimed by the inventor to produce the desired equality in the current through the filtering surfaces.—B.

*Improvements in and connected with Gas Producers.* J. J. and T. F. Meldrum, both of Manchester. Eng. Pat. 4076, February 24, 1893.

See under II., page 242.

*Apparatus for Driving Off Heavy Vapours of Solvents.* The Parent Syndicate Oil Extractor Co., Lim., and H. A. A. Dombrain, Bradford, Yorkshire. Eng. Pat. 3040, March 8, 1893.

THE material, containing the solvent, is fed into the lower end of an inclined trough and distributed along it by a pair of worm conveyors. The trough is surrounded by a sheet-metal casing communicating by means of pipes with a water-cooled conduit below, and furthermore is heated by a steam-jacket depending from its under side, whereby the solvent is evaporated and the heavy vapour, escaping into the water-cooled conduit, is condensed.—E. G. C.

*Improvements in the Manufacture or Restoration of Filtering Media.* J. Wetter, London. From M. Weinrich, St. Louis, U.S.A. Eng. Pat. 5375, March 13, 1893.

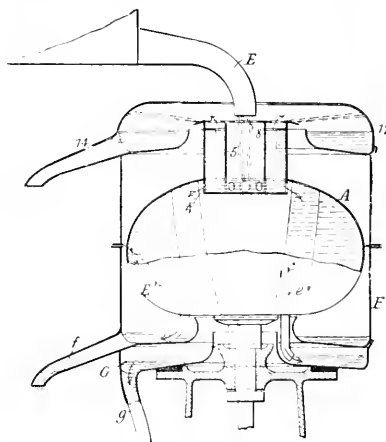
TO regenerate spent bone-charcoal, whereby it may be used over again for filtering purposes, the bone-charcoal or bone-char is first washed with pure hot water, and, if necessary, treated with such purifying agents as muriatic acid, soda, or ammonia. The wash-water is afterwards drawn off, and a hot solution of crude bone gelatin or dissolved glue is run on to the bone-char, in the proportion of 160 to 240 lb. of solution to 1,000 lb. of bone-char. Steam is now applied to bring the temperature of the mass up to boiling point, and it is kept boiling until the water in the mass is so far reduced in amount, that the gelatin sticks to the particles of bone-char. The mass is now removed, dried, and then charred in suitable kilns, thereby covering the old ineffective surface of the particles with a

new coating. The burned and cooled bone-char is now washed with pure hot water, and is then ready for use. A mixture of gelatin and fresh blood, or fresh blood alone, may be used in place of the gelatin or glue.—E. G. C.

*Improvements in Centrifugal Machines for Separating Liquids.* F. Ludloff, Berlin. Eng. Pat. 5493, March 14, 1893.

THE inventor states that in ordinary centrifugal machines used for separating cream from milk, it is necessary that the speed should be kept even. If slower than the supply warrants, the full quantity of cream is not extracted and the contents are likely to run over. If worked faster than necessary a loss of power ensues.

The patentee proposes to remedy this defect by introducing an automatic arrangement by which, for a reduced speed, a part of the milk under treatment is thrown into a vessel from which it is returned to the original reservoir for a second treatment. The illustration indicates one of



the arrangements, in which *A* is the separating drum, here shown of somewhat spherical shape. The milk reaches it from the spout *E* through the hole *8* in the upper part of the cylindrical neck attached to the drum *A*, inside of which the liquid, on rotation, takes the parabolic form, having the skim milk near the larger diameter of the drum and the cream more towards the centre. The skim milk escapes through the tubes *E'* into the casing *F*, leaving the latter by the spout *f*. The cream-collecting tube *e'* discharges into the lower chamber *G* in a similar manner. If the speed of the drum decreases, the issue through holes *4* diminishes, which causes the supply to rise in the upper cylindrical chamber and finally to overflow into the splash-pan *12*, issuing at *14*, and thus indicating a necessity for increased speed. At too high a speed no such stream of milk is seen at the outlet *14*, and the speed may be diminished until just giving a proper balance. The various holes indicated are originally adjusted for the proper speed desired.—B.

*Improvements in Filtering Sewage.* B. Rushford and J. Edwards, Aldershot. Eng. Pat. 5689, March 16, 1893.

See under XVIII. B., page 271.

*Improvements in Apparatus for Cooling and Evaporating Liquids, such as are used in Breweries and Distilleries, and for such like Purposes.* H. J. Worsam, London. Eng. Pat. 6316, March 24, 1893.

See under XVII., page 269.

*Improvements in Self-acting Apparatus for Raising or Forcing Liquid by Steam, Compressed Air, or other Elastic Fluid under Pressure.* P. Kestner, Lille, France. Eng. Pat. 6165, March 27, 1893.

This invention refers to improvements in the details of an automatic valve arrangement under the previous patent, No. 15,648, 1892 (this Journal, 1892, 394), whereby it is sought, by means of a body suitably arranged in the liquid, that when the receptacle is filled with liquid this body will cause or allow the escape valve to close and the steam valve to open, but when the liquid has been discharged more or less completely from the receptacle, the weight of the said body will effect the opening of the discharge valve and close the steam valve, without requiring first to effect a reduction of pressure in the receptacle.—B.

*Improvements in Vessels for Containing Liquefied Sulphur Dioxide for Use as a Disinfectant and Vermin Destroyer, and for other Medical and Sanitary Purposes.* E. Soy, London. Eng. Pat. 12,276, June 23, 1893.

See under XVIII. C., page 271.

*Improvements in the Method and Apparatus for Producing Gas.* A. Kitson, German Town, Penn., U.S.A. Eng. Pat. 19,521, October 17, 1893.

See under II., page 243.

*An Improved System and Apparatus for Removing Impurities from and Refining China Clay.* W. W. R. Nicholls, W. Nicholls, and F. H. Nicholls, St. Austell. Eng. Pat. 19,699, October 19, 1893.

See under VIII., page 251.

*An Improved Method or Process and Apparatus for Deodorising Oils and Fats.* H. J. Bull, Bergen, Norway. Eng. Pat. 20,413, October 28, 1893.

See under XII., page 261.

*Improvements in Apparatus and Furnaces for Concentrating Sulphuric Acid and other Liquids.* J. Bradbury, Manchester. Eng. Pat. 23,327, November 23, 1893.

See under VI., page 251.

*Improvements in and connected with the Process of and Machinery for the Drying or Heating of Gypsum, Clay, Sand, Grain, Slaughter-house Refuse, and other Materials.* R. Haddan, London. From F. D. Cummer, Cleveland, Ohio, U.S.A. Eng. Pat. 23,045, November 30, 1893.

The apparatus consists of an inclined rotating cylinder, supported at each end on "spiders," one "spider" being operatively connected with the cylinder for driving it by means of suitable mechanism. The material to be heated or dried is fed in at the upper end of the cylinder, and is agitated by means of longitudinal stirrers, fixed to the sides of the cylinder; the material, after passing through the cylinder, falls into a screw conveyor for delivery at any required point. Around the cylinder is built the combustion chamber of a furnace, thereby permitting the hot air and gases to circulate round the cylinder, and also to enter at the lower end and pass through the cylinder to the upper

end, from which they are conveyed away to the flue. An exhaust fan is placed within the flue to cause a current of air through the apparatus, to promote the heating and drying action, and also to more effectively control it than would be the case with a natural draught. Pockets are provided to receive material which may escape from the cylinder, between the "spiders" and the cylinder shell, and this material is afterwards discharged through a suitable drain-pipe.—E. G. C.

*Improvements in or relating to Machinery or Apparatus for Treating, Deodorising, Drying, and Utilising Excreta, Nightsoil, Town's Refuse, and the like.* S. Smithson. Eng. Pat. 23,082, December 1, 1893.

See under XVIII. B., page 271.

*Improvements in the Concentration or Vaporisation of Sulphuric and Acetic Acids, and in Apparatus therefor.* A. J. Boulton, London. From S. C. Penchen and P. Clarke, Toronto, Canada. Eng. Pat. 24,739, December 23, 1893.

See under VII., page 251.

*Improvements in the Manufacture of Sugar.* H. H. Lake. From P. H. Vander Weyde and O. Lugo, New York, U.S.A. Eng. Pat. 136, January 2, 1894.

See under XVI., page 265.

## II.—FUEL, GAS, AND LIGHT.

*General Characters of Boghead Cannel.* C. E. Bertrand and B. Renault. Comptes rend. 1893, 117, 593—596.

THE following are the conclusions drawn from a study of boghead from Autun, kerosene shale from Australia, and Torbane Hill mineral:—

A very simple class of coal exists, which is formed by the accumulation of the fronds of a single species of algae in an ulmic deposit. These vegeto-ulmic deposits indicate tranquil periods during which the algae have multiplied so as to cover the surface of the water, whilst an abundant terrestrial vegetation has scattered clouds of pollen or spores. These vegetable accumulations have been produced on the spot where the algae have lived. The ulmic acid is precipitated under the influence of calcareous water, the algae sink to the bottom and carry with them the soaked pollen or spores, whilst any small floating debris is simultaneously carried down with the ulmic substances. No change, no decomposition has taken place in these vegeto-ulmic deposits. In the case of the three boghead cannel, these deposits have become penetrated by brown infiltrations probably of a bituminous nature: the bitumen possibly owes its origin to the decomposition of a mass of vegetable matter not far removed.

The boghead cannel may frequently be associated with ordinary coal, which they sometimes precede, sometimes succeed: occasionally they occur as intercalated beds in the coal measures, and not infrequently the coal occurs in the boghead cannel. The cleavage of the coal does not extend to the masses of boghead cannel.

The three boghead cannel owe their special characters to the algae composing them, namely, the Autun boghead to *Pila bibraetensis*, kerosene shale to *Reinschia australis*, and Torbane Hill mineral to another *Pila*. If all other conditions are present but the algae are wanting, the coal-bed will not possess the character of a boghead cannel.

Amongst the yellow bodies present in coal are the substances contained in the pollen grains and spores, also those produced by the walls of the fronds of the *Reinschia* and *Pilas*. Those formed by the gelatinised fronds are of a different kind from those produced by the sound fronds.

Then, again, there are yellow particles of animal origin, produced from cartilage, from coprolites, and even from the soft parts of animals embedded in the ulmic deposits.

Diatomaceæ were not found in the three boghead canals.  
—A. K. M.

*Persian Coal.* A. F. Stahl. Teheran. Chem. Zeit. **17**, 1596.

A CONSIDERABLE amount of coal is found in Persia, but for the most part of somewhat poor quality. Owing to the want of proper facilities for transport, but little is actually mined; thus, only 6,000 to 7,000 kharvar (1 kharvar = 280 kilos.), or some 1,500 tons, are yearly brought to Teheran, and sold at a price averaging about 40 kran (1 kran = about 6d.) per kharvar, or something like 4l. per ton. Naturally, industries requiring fuel are not greatly developed under such conditions. Various samples of Persian coal yielded the following figures on proximate analysis:—

No.	Volatile Matter lost on Destructive Distillation.	Coke.	Ash.
	Per Cent.	Per Cent.	
1	12	88	20 per cent.
2	20	80	14 per cent.; also 16 per cent. of stones.
3	19	81	50 per cent. ash and stones.
4	31	69	9 per cent.
5	28	72	27 "
6	27	73	10 per cent.; also 2 per cent. of stones.

As a rule such coal is very brittle and disintegrates spontaneously on exposure to air. In some cases a considerable admixture of pyrites is present, whilst the amount of stony matter is often excessive, owing to defective hand-picking.—C. R. A. W.

*Estimation of Sulphur in Recovered Gas-purifying Material.* F. Meyer. Chem. Zeit. **17**, 1319.

See under XXIII., page 283.

*A Coal containing Vanadium.* A. Mourlot. Comptes rend. 1893, **117**, 546.

See under Analyt. and Scient. Notes, page 289.

## PATENTS.

*An Improvement in Incandescent Gas Burners.* O. Imray, London. From J. Moeller, Paris. Eng. Pat. 124, January 3, 1893.

This invention relates to the kind of burner described in Eng. Pat. 15,286, 1885, in which a network hood of metallic oxide is heated to incandescence by a Bunsen flame. The improvement consists of making the hood of oxide of thorium, to which not more than 2 per cent. of the oxide of other rare metals, such as "uranium, cerium, terbium, neodymium, praeodymium, samarium, yttrium, or lanthanum," has been added.—R. B. P.

*Improvements in or relating to the Manufacture or Production of Block Fuel, Gas and other useful Products from Refuse Material.* J. C. W. Stanley, Middlesex. Eng. Pat. 2247, February 1, 1893.

THE process described in this specification relates to a method of treating what is known as "dust-bin" refuse,

from which waste paper, &c. has been removed in the manner described in Eng. Pat. 1510, 1889, so as to separate the various materials it contains, in order that they may be subsequently utilised.

The process consists of a series of siftings, washings, and filterings. The final products obtained are stated to be, "unburnt coal, burnt coal (floats and cinder), mixed coal and cinder, clean-washed sand, filter-pressed cakes, mud containing potash and nitrogen.—R. B. P.

*An Improved Method of Preparing Peat for Heating, Gas Generating, and other Purposes.* T. A. Dillon, Dublin. Eng. Pat. 3554, February 17, 1893.

This invention consists in mixing peat with mineral, or vegetable oils, fats, or resinous substances, by aid of an alkali, and thus making even moist peat into a satisfactory fuel.—R. B. P.

*An Improvement in Apparatus for the Manufacture of Water-Gas.* J. R. Paisley, Colnbrook. Eng. Pat. 3815, February 21, 1893.

This invention relates to an improvement in the cooler of the apparatus described in Eng. Pat. 15,797, 1889, and has for its object the prevention of the cooling pipes becoming choked by the dust which enters with the gas. This is effected by prolonging the lower ends of the cooling pipes into a trough, which forms a water seal. Passages for the gas from one pipe to the next are provided just above the water seal. By these means, whilst the gas flows through the cooler in the usual way, the dust entering with it, falls through the prolongations of the cooling pipes into the trough forming the water seal, and choking of the pipes is prevented.—R. B. P.

*Improvements in or connected with Gas Producers.* J. J. and T. F. Meldrum, both of Manchester. Eng. Pat. 4076, February 24, 1893.

This invention relates to producers closed at their lower end by a water seal, and into which air is introduced by means of a steam blower through a tuyère situated near the lower part of the producer. Figs. 1 and 2 show a sectional elevation and plan respectively, of the apparatus.

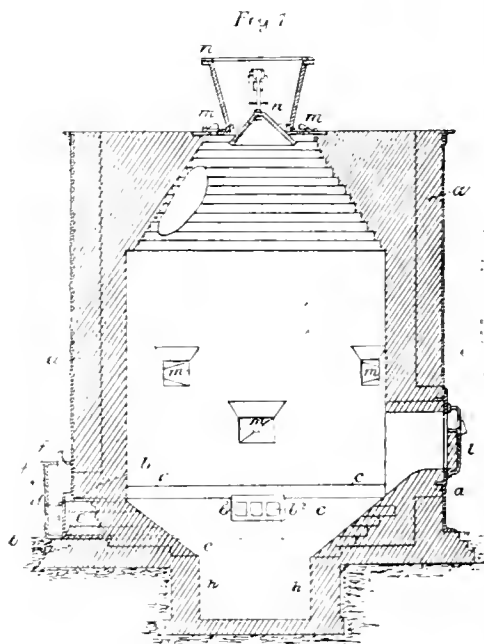
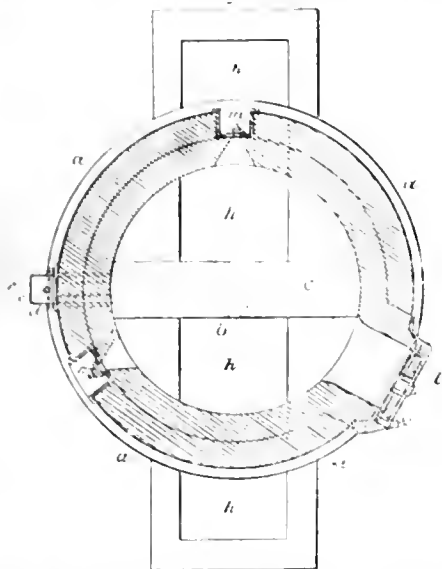


Fig. 7



*b* is the tuyère, resting on a raised wall *c* (Fig. 2), and *c* the blower. *h h*, are pits forming the water seal, from which ashes can be withdrawn. —R. B. P.

*Improvements in the Manufacture of Illuminating Gas, Artificial Fuel Blocks, and Coke for Metallurgical Purposes.* G. de Velna, Paris. Eng. Pat. 6951, March 21, 1893.

This process consists in adding hydrocarbons formed into an emulsion to any kind of coal, whereby a better quality and yield of gas is obtained. The emulsion is prepared by suitably mixing together and heating the following substance:

	Parts.
emulsion of, or any fatty acid .....	2 to 10
residual ley .....	0.1 to 1
Tar of petroleum or schist .....	89 to 98

—R. B. P.

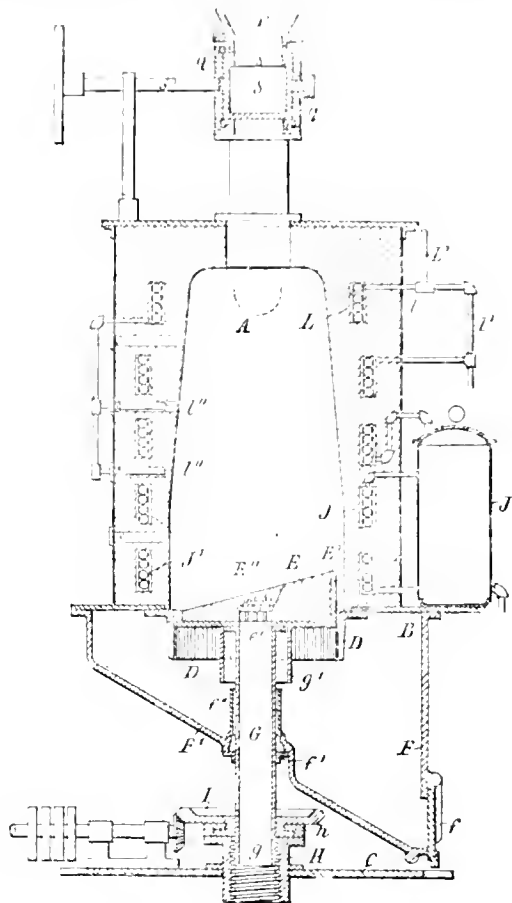
*Improvements in and connected with Means or Apparatus for Drying, more especially intended for Peat.* J. D. Brunton, Westminster. Eng. Pat. 6770, March 30, 1893.

In this process the peat is first moulded into bricks by a suitable machine, which delivers them on to a table consisting of a series of rotating parallel rollers, carried on a trolley. The mechanism is such that the bricks are regularly delivered on to suitably-heated drying floors from the moulding machine. It not only causes the passing of the peat bricks across the drying floor, but, as the bricks are turned over each time they pass from one strip to the next, it assists in the proper drying of them. The last strip delivers the dried bricks on to a suitable conveyor. —R. B. P.

*Improvements in the Method of and Apparatus for Producing Gas.* A. Kitson, German Town, Penn., U.S.A. Eng. Pat. 19,521, October 17, 1893.

The distinguishing feature of this apparatus is an inclined hearth which can be simultaneously rotated and raised, or lowered, whereby the fuel is kept in a uniform state of compactness, its surface clean, and the formation of clinker prevented. *E* is the inclined hearth, which is supported by the hollow shaft *G*. *G* is rotated by gearing and caused to rise or fall by the screw and nut at its lower end. *J* is a water-circulating coil connected to the reservoir *J*, as is also the steam-superheating coil *K*. *L* is an oil-vaporising coil connected at *L'* with the oil-supply pipe, and at *L''* with the

superheating coil, steam from which forces the oil through *L*, in which it is subjected to the heat of the furnace, and then into the furnace chamber through the openings *l' l''*. Air is supplied to the fuel in *A* through *G*, at the upper



end of which is a grating *c*. Over *c* are placed pieces of fire-brick or other refractory material. The air is heated before entering, by causing it to pass along a jacket surrounding the gas outlet from *A*. *S* is a hollow rotating plug for feeding in the fuel without allowing any gas to escape. —R. B. P.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Petroleum in Persia.* A. F. Stahl. Chem. Zeit. 17, 1102.

PETROLEUM is known in many places in Persia, but has not as yet been satisfactorily examined. An English company possessing the mining rights for most minerals in Persia, has undertaken during the last three years, boring experiments in Daliki, about 90 km. north-east of the harbour Bushire and on Kishm Island in the Persian Gulf, but without success. Traces of oil may be found all along the west and south-east coast, and in the interior of Persia, pointing to the probability of prolific oil beds. The oil wells east of Kutweis and Ahwaz, on the River Karoon, have yielded for many years a light-coloured oil, which the inhabitants obtain in a very primitive manner.



In November 1891, the author discovered a petroleum well south-east of Semnoon, on the road between Teheran and Meshed, in the Great Salt Desert, which he considers would be well worth boring trials.

After giving a geological sketch of the district, he goes on to show that on sinking 6 metres in the bluish clay, he obtained an output of 40–50 gallons oil per day, mixed with concentrated salt water. This oil has a specific gravity of 0.911. A distillate was obtained at 250° C. of sp. gr. 0.853. Later, another distillate at 200° C. showed a specific gravity of 0.815. The use of 15 per cent. of sulphuric acid failed to remove the yellow colour of the oil, which the author attributes to the high percentage of paraffins present.

The oil beds seem to be near the surface, and the geological structure points to a larger yield. There is no lack of inducement for boring experiments, and, as the use of oil in Persia for lighting purposes is increasing, there is no reason to doubt that an important industry may be developed.

—H. M.

*The Estimation of Paraffins.* R. Holand. Chem. Zeit. **17**, 1893, 1473, 1483.

See under XXII., page 286.

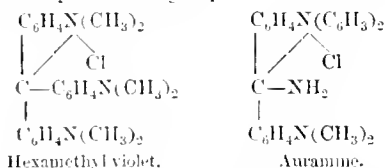
*Cresol as an Antiseptic.* Lauroix Hunkiahyendiau. J. Pharm. Chim. **23**, 34.

See under XX., page 273.

#### IV.—COLOURING MATTERS AND DYES.

*The Auramines.* A. Stock. J. prakt. Chem. **47**, 401–413.

The author has prepared and investigated the following auramines: Acetylphenylauramine, methylphenylauramine, diphenylauramine, and pentamethylene-auramine, and, on account of the existence of the di-substituted derivatives, he considers that they are derived from triphenylmethane compounds in such a way that a benzene ring is eliminated and replaced by an amido-group.

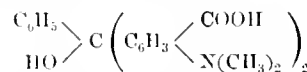


The colour of these compounds depends on the basicity of the amido-group. If this be strongly basic the colour is yellow, and as the basicity gradually diminishes the colour passes through yellowish-red into red. Alkalis as well as acids decompose the di-substituted auramines with the production of the corresponding ketone and amine. By the action of dry ammonia the amido-group is replaced by  $\text{NH}_2$ , the corresponding secondary amine being split off. Aqueous ammonia plays the part both of an alkali and of dry ammonia. As the basicity of the amido-group increases the latter reaction predominates.—J. S.

*The Carboxyl Derivatives of Dimethylaniline.* C. Lauth. Comptes rend. 1893, **117**, 581–583.

The author has examined the three isomeric dimethylanilidobenzoic acids  $(\text{CH}_3)_2\text{N} \cdot \text{C}_6\text{H}_4 \cdot \text{COOH}$ , with the view of determining the influence of the carboxyl group and of its position in the molecule. It is found that by the action of oxidising agents such as cuprous chloride or chloranil, the *ortho*-compound yields a reddish-violet tetramethylrosanilinecarboxylic acid which differs from the ordinary colouring matters of the triphenylmethane

series in being soluble in alkalis, from which solutions it is precipitated by acids. *o*-Dimethylanilidobenzoic acid gives a condensation product with tetramethyldiamidodiphenylcarbinol, which, when oxidised by means of lead oxide, gives a violet blue hexamethylrosaniline-carboxylic acid; it also yields a blue colouring matter—



with benzaldehyde and its metanil derivatives. *Para*-dimethylanilidobenzoic acid also yields condensation colouring matters, but the carboxyl group becomes eliminated. *Meta*-dimethylanilidobenzoic acid gives no colouring matter on oxidation, none with the aldehydes, and only a trace with tetramethyldiamidodiphenylcarbinol.—A. K. M.

*The Colouring Principles of Rubia Sikkimensis.* A. G. Perkin and J. J. Hummel. Proc. Chem. Soc. 1893 [128], 200.

The authors have separated both purpurin and munjistin or purpuroxanthinecarboxylic acid from this root; they point out that the results show that it is nearly identical as regards colouring principles with the closely allied *Rubia munjistin*.

*The Colouring and other Principles contained in Chara Root.* A. G. Perkin and J. J. Hummel. Proc. Chem. Soc. 1893 [133], 201.

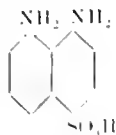
CHAY root is the root of *Oldenlandia umbellata*, and is used as a dye-stuff in India; a large number of distinct products were isolated by extracting it either with an aqueous solution of sulphurous acid, or with lime-water, alcohol, and other solvents, viz., two substances of the formula  $\text{C}_{16}\text{H}_{12}\text{O}_5$ , both of which are shown to be autragalloldimethyl ethers; a monomethyl ether of alizarin crystallising in orange needles melting at 178°–179°; a resinous yellow substance, possibly an isomeric methyl ether of alizarin in an impure form; methoxyanthraquinone; a red substance melting at 141°, which yields alizarin with sulphuric acid; ruberythric acid; rubichloric acid; alizarin; a wax of the formula  $(\text{C}_{30}\text{H}_{15}\text{O})_2$ , melting at 87°–88°; and cane sugar.

#### PATENTS.

*Improvements in the Production of Diamido-naphthalene-sulpho Acid and Amidonaphthol Sulpho Acid, and Colouring Matters from the latter.* S. Pitt, Sutton. From L. Cassella and Co., Frankfort-on-the-Maine, Germany. Eng. Pat. 4613, March 2, 1893.

By suitably nitrating and reducing naphthalene  $\beta$ -sulphonic acid, it is converted into a 1'1'-diamido-naphthalene derivative (Eng. Pat. 6972 of 1891; this Journal, 1892, 345). If the  $\alpha$ -acid be treated in a similar manner the main product formed during the mono-nitration is the 1'1'-nitro-naphthalene sulphonic acid, whilst the 1'4'-acid is only formed in small quantity. As, however, it is only the latter which is of use for conversion into the 1'1'4'-diamido-naphthalene sulphonic acid, another method has been devised for the preparation of the 1'4'-acid:—173 kilos. of  $\alpha$ -nitronaphthalene are converted into mono-sulphonic acid. After sulphonation the melt is cooled to 10° C. and 150 kilos. of nitrosulphuric acid, containing 63 kilos. of  $\text{HNO}_3$ , are run in at 15–20° C. The whole is then allowed to stand for 24 hours, and afterwards poured into 3,000 litres of brine. The greater part of the 1'1'4'-dinitro-naphthalene sulphonic acid separates as a brownish crystalline precipitate, which, after filtering, is washed with brine until free from acid. In order to reduce it, the dinitro acid is dissolved in 2,000 litres of water and heated with iron and a little acetic acid. After the reduction is complete the iron is precipitated with sodium carbonate, and 400 kilos. of sulphuric acid are added

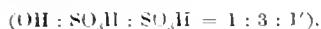
to the filtrate. On cooling, the sulphate of the diamido acid crystallises out in colourless needles. The acid has the following constitution.—



It combines with one or two molecules of a diazo compound and yields characteristic salts with mineral acids. It only combines with one molecule of nitrous acid, forming an azimide. The free acid is nearly insoluble in water, and may be obtained by carefully acidifying a solution of its sodium salt. The sodium salt may be salted out by means of common salt. By heating with mineral acids or caustic alkalis the acid is converted into 1'-amido-naphthol sulphonic acid, the best method being as follows:—10 kilos. of diamidonaphthalene sulphonic acid are heated for 12 hours "in a covered vessel" with 100 kilos. of dilute sulphuric acid containing from 20 to 30 per cent. of  $\text{H}_2\text{SO}_4$ . The amidonaphthol sulphonic acid separates in colourless needles from the cold solution. The acid dissolves sparingly in cold water. When treated with nitrous acid it dissolves with a yellow colour, forming an unstable diazo compound. The colour is changed to violet-red by the addition of sodium carbonate. The salts of the new acid are very soluble and show a violet fluorescence in an aqueous solution. The acid is capable of combining with one or two molecules of a diazo compound. The combinations with tetrazo compounds can take place in an acid or alkaline solution, producing different shades, and the usual variations in the components can be made. The colouring matters produced dye shades which lie towards the blue end of the spectrum, and certain of the combinations yield blue and greenish-black dyes.—T. A. L.

*The Manufacture or Production of Direct-dyeing Colouring Matters.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 4756, March 4, 1893.

By combining the diazo compounds of dehydrothio-*p*-toluidine, dehydrothio-*m*-xylidine, dehydrothio-*z*-cumidine, primuline base or their sulphonic acids with the monoalkylated 1'-1'-dihydroxynaphthalene sulphonic acids, direct-dyeing cotton colouring matters of bluish-red shades are produced (Eng. Pat. 3495 of 1893; this Journal, 1894, 145). On further investigating the behaviour of  $\alpha$ -naphthol sulphonic acids towards the above diazo compounds, it has been found that the colouring matters derived from the 1-3 acid give bluish-red shades fast to acids and alkalis, whereas the colouring matters from the 1-2, 1-3', or 1-2' acids only yield dull shades. The colouring matters produced are also superior to the dyestuff known as "Erica," which is formed by combining diazotised dehydrothio-*m*-xylidine with  $\alpha$ -naphthol-6-disulphonic acid—

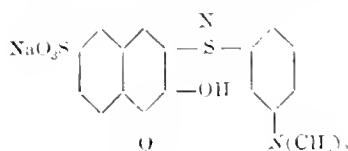


The formation of the colouring matter from dehydrothio-*p*-toluidine sulphonic acid is described as follows:—34.2 kilos. of sodium dehydrothio-*p*-toluidine sulphonate are dissolved, together with 6.9 kilos. of sodium nitrite, in about 300 litres of water, and hydrochloric acid is allowed to run in until the liquid is strongly acid. After about 12 hours, the diazo compound thus produced is added to a solution containing 24.6 kilos. of the sodium salt of the 1-3-naphthol sulphonic acid made alkaline with sodium carbonate. After standing 12 hours the mixture is heated to about 96° C., and the dyestuff is salted out and filtered off. It gives bluish-red shades on unmordanted cotton.

—T. A. L.

*The Production or Manufacture of Colouring Matters and Materials therefor.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 4757, March 4, 1893.

The first part of this specification relates to the production of alkylated *p*-phenylene diamine thiosulphonic acids, which subsequently are employed for the preparation of colouring matters. By adding two molecular proportions of sodium thiosulphate dissolved in water to a solution of one molecular proportion of a nitroso compound of a tertiary or secondary amine made acid with two molecular proportions of acetic acid and subsequently heating the resulting mixture to the boil, the yellow colour of the solution disappears, but no sulphur separates, and after a short time the liquid is of a slightly brownish colour, and the thiosulphonic acid derivative thus formed can be employed in the production of dyestuffs. The following quantities are given for the preparation of one of the thiosulphonic acids referred to:—34.2 kilos. of the sodium salt of nitroso-ethyl-benzyl-aniline sulphonic acid and 36 kilos. of a 33 per cent solution of acetic acid are dissolved in 200 litres of water and added to a solution of 50 kilos. of sodium thiosulphate in 200 litres of water. The liquid is heated to boiling until the original yellow colour has become fawn. The solution then contains the thiosulphonic derivative of *p*-amido-ethyl-benzyl-aniline sulphonic acid and may be used directly for the production of dyestuffs. It nitroso-dimethylaniline hydrochloride be employed instead of the nitroso-ethylbenzyl-aniline sulphonic acid, the resulting compound is *p*-amido-dimethylaniline thiosulphonic acid. The amido-methyl-*o*-toluidine thiosulphonic acid obtained from nitroso-methyl-*o*-toluidine forms granular crystals. The production of colouring matters from these thiosulphonic acids is an extension of the method described in Eng. Pat. 825 of 1893 (this Journal, 1894, 31). The solution of the thiosulphonic acid from the 34.2 kilos. of nitroso-ethyl-benzyl-aniline sodium sulphonate described above, is, after boiling, mixed directly with 28 kilos. of  $\beta$ -naphthoquinone *p*-sulphonic acid. The dyestuff commences to form at once, and the reaction is completed by heating the mixture for several hours to 80—100° C., when the colouring matter separates in bronzy crystals, which can be filtered off. The product is identical with that obtained from  $\beta$ -naphthoquinone *p*-sulphonic acid or from 1-2,4-dihydroxynaphthalene sulphonic acid and nitroso-ethyl-benzyl-aniline sulphonic acid in the presence of thiosulphates, as described in Eng. Pat. 825 of 1893. A dyestuff from dimethyl *p*-phenylene diamine thiosulphonic acid is produced by dissolving 25 kilos. of it and 25 kilos. of crystallised sodium acetate in 750 litres of hot water and mixing this with a solution of 28 kilos. of the potassium salt of  $\beta$ -naphthoquinone 3'-sulphonic acid dissolved in 200 litres of water. The whole is then heated for several hours to 70—100° C. until the condensation is complete. The product probably possesses the following formula:—



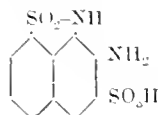
The colouring matter is very similar to the dyestuff obtained by sulphonating the product from nitroso-dimethylaniline, sodium thiosulphate, and  $\beta$ -naphthoquinone 4-sulphonic acid and gives blue shades on wool mordanted with chromium salts. Other blue dyestuffs are obtained by condensing dimethyl-*p*-phenylene diamine thiosulphonic acid with  $\beta$ -naphthoquinone-disulphonic acid. One of these, the  $\beta$ -naphthoquinone-2'-1'-disulphonic acid, can be obtained by oxidising one molecular proportion of 1-2-amido-naphthol-2'-1'-disulphonic acid with one molecular proportion of bromine in a suitable diluent or solvent and subsequently precipitating the acid as potassium salt by the addition of potassium chloride. By condensing  $\beta$ -naphthoquinone-4'-1'-disulphonic acid or its salts with dialkyl-*p*-phenylene diamine thiosulphonic acid, the same

dyestuffs are formed as those obtained from  $\beta$ -naphthoquinone-1'-sulphonic acid, as in the former case the one sulphonic acid group is split off during condensation. The  $\beta$ -naphthoquinone-1'-disulphonic acid is obtained by reducing an azo dyestuff from  $\alpha$ -naphthol-4'-disulphonic acid and oxidising the 2'-amido-naphthol-4'-disulphonic acid produced with one molecular proportion of bromine and salting the product out with potassium chloride. The potassium salt thus formed yields orange-red crystals sparingly soluble in a solution of potassium chloride.

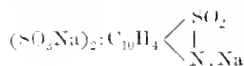
—T. A. L.

*The Manufacture and Production of Sulpho Acids of the Naphthalene Series and of Colouring Matters Obtainable therefrom.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 4979, March 7, 1893.

Isocyanides of the naphthol sulphonic acids are known as "sultones." Hitherto, only one of the corresponding anhydrides from an amido-naphthalene sulphonic acid has been described by Berntsen (Ber. 23, 3094), who produced it by heating the colouring matter from diazotised xylydine and  $\epsilon$ -naphthol sulphamidosulphonic acid with a dilute solution of sodium carbonate, and subsequently reducing the dyestuff thus formed. The resulting acid has the following constitution:—



Bodies of this type are produced from these naphthylamine sulphonic acids which contain an amido and a sulphonic group in the 1'- or peri position, by heating them with fuming sulphuric acid, and the products obtained are known as "naphthasultame sulphonic acids." Occasionally simultaneous sulphonation takes place. The following perinaphthylamine sulphonic acids can be used:—1'-naphthylamine sulphonic acid, 1'-4'-naphthylamine disulphonic acid (both of Ger. Pat. 40,571), 1'-4'-naphthylamine disulphonic acid, 1'-2'-naphthylamine disulphonic acid (Ger. Pat. 45,776), and 1'-2'-3'-naphthylamine trisulphonic acid. The method employed for producing the anhydride is, stirring the acid sodium salt of the acid into twice its weight of fuming sulphuric acid containing 40 per cent. of anhydride and heating the solution at 80°–90° C., until a test no longer gives a diazo compound or forms a dyestuff with diazobenzene sulphonic acid. The melt, which has become solid, is then diluted with ice water, neutralised with lime, filtered, and the solution evaporated down. The sodium salt when dried at 160° C. has the following composition:—



It is very easily soluble in water, but almost insoluble in absolute alcohol. Its solution in water is yellow and exhibits a strong greenish fluorescence similar to fluoresceine. The yellow colour and fluorescence disappear on adding an acid, even acetic acid, and when salt is added to this solution, white needles of the di-sodium salt crystallise out. These naphthasultame sulphonic acids remain unaltered by boiling with alkali lye, ammonia, or dilute acids, differing in this respect from the sultones, which are hydrolysed by this treatment. By fusing with  $2\frac{1}{2}$  times their weight of potash or soda and one-fifth their weight of water, they are converted into amido-naphthol sulphonic acids, which are known as peri amido-naphthol sulphonic acids. They are to be employed as components in the various methods for the production of azo dyestuffs.—T. A. L.

*Improved Manufacture of Colouring Matters.* C. D. Abel, London. From "The Actien Gesellschaft für Anilin Fabrikation," Berlin, Germany. Eng. Pat. 5039, March 8, 1893.

The 1'-1'-amidonaphthol disulphonic acid of Eng. Pat. 2984 of 1893 (this Journal, 1891, 32) can combine with various diazo compounds to form azo colouring matters. If tetrazo compounds be employed the usual modifications in the methods of combination may be carried out with the new amidonaphthol disulphonic acid. The shades obtained comprise reds, violets, and blues.—T. A. L.

*Improvement in the Production of Disazo Dyestuffs from Gamma "Amidonaphthol" Sulpho Acid.* S. Pitt, Sutton. From L. Cassella and Co., Frankfurt-on-the-Maine, Germany. Eng. Pat. 5141, March 9, 1893.

This is an extension of Eng. Pat. 16,699 of 1889 (this Journal, 1890, 935), and refers to the substitution of *p*-diamidodiphenylamine for the *p*-diamines there described. The dyestuffs obtained are very similar, being a little bluer in shade. According to this specification a colouring matter which dyes unmordanted cotton a bluish-black is obtained by suspending 30 kilos. of *p* diamidodiphenylamine sulphate in 400 litres of water and 40 kilos. of hydrochloric acid, diazotising this quantity with 14 kilos. of sodium nitrite at 0° C., and pouring the solution of the tetrazo compound, which is of a yellowish colour, into 1,000 litres of water in which 60 kilos. of  $\gamma$ -amidonaphthol sodium sulphonate are dissolved, the whole being kept alkaline by an excess of soda. A dark violet precipitate separates, which after 12 hours changes to a dark blue. After heating to about 80° C. the dyestuff is salted out. Other combinations may be obtained in a similar manner.

—T. A. L.

*Improvements in the Manufacture of Coloured Marking Inks and Marking Ink Pencils.* J. Hickisson, Hackney. Eng. Pat. 5316, March 11, 1893.

For use with a pen, an ink is prepared by dissolving one of the "oil-anilines" in castor oil, adding a small amount of india-rubber solution and a suitable mordant, and thinning down the mixture with turpentine. The colour may be brightened by the addition of a dye if desired. For pencils, one part of gum tragacanth, one part of china clay, one quarter part of borax, and three of oil-aniline are mixed together; the fabric is greased, mordanted (if no mordant is present in the pencil), and written on, and finally a hot iron is passed over the mark. The materials prescribed in Eng. Pat. 15,320 of 1887, are recommended as mordants.

—F. H. L.

*The Production of New Disazo Colouring Matters.* S. Pitt, Sutton. From L. Cassella and Co., Frankfurt-on-the-Maine, Germany. Eng. Pat. 5407, March 13, 1893.

The colouring matters described are obtained by combining tetrazo compounds with the alkylated derivatives of amidonaphthol disulphonic acid II. The preparation of this acid is described in Eng. Pat. 1742 of 1891 (this Journal, 1891, 918). For the production of the ethyl derivative 36 kilos. of the neutral sodium salt of 1'-1'-2'-3'-amidonaphthol disulphonic acid (acid II) are heated in an autoclave with 11 kilos. of ethyl bromide, 60 kilos. of calcium carbonate, and some dilute alcohol of 50 per cent. for 20 hours to 60° C. After filtering, the liquid is made acid with 12 kilos. of hydrochloric acid, and, after separation from a small quantity of unaltered acid, the filtrate is concentrated, and on cooling, the ethylamido-naphthol disulphonic acid II separates in thin colourless needles. The acid is easily soluble in water, less so in strong alcohol. It gives an easily soluble nitroso-amine with nitrous acid. The methyl or benzyl derivatives are obtained in a similar manner. All these compounds combined in an alkaline solution with the tetrazo derivatives of *p*-diamines

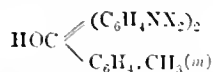
such as benzidine, tolidine, diamido ethoxy-diphenyl, diamidodiphenylether, &c., give blue dyestuffs for cotton of a very greenish shade. They differ from the products obtained by treating with halogen alkyls and an alkali those diazo derivatives of the amidonaphthol disulphonic acid II which are obtained in an acid solution and contain a free hydroxyl group. The technical value of the derivatives from the new acid consists in their greenish-blue shades, and the acid may therefore be used with advantage as a second component in the case of mixed diazo compounds. The following example shows the method employed:—The tetrazo compound from 18.4 kilos. of benzidine is cooled to 0° C. and added quickly to a solution of 42 kilos. of sodium carbonate in 400 litres of water. Immediately afterwards a neutral solution of 37 kilos. of 1.1'-2.3-ethyl-amido naphthol disulphonic acid is added, the whole being well stirred. The resulting intermediate compound separates as a blue-black precipitate, and a neutral solution of 24 kilos. of  $\gamma$ -amidonaphthol sulphonic acid is added. The intermediate compound gradually dissolves, and the dyestuff separates as a dark-coloured precipitate. It dyes unmordanted cotton a blue-black. The methyl or benzyl compound may be employed in place of the ethyl derivative without altering the properties or shades of the dyestuffs produced.—T. A. L.

**Production of Colouring Matters.** O. Imray, London. From "The Farbwerke vormals Meister, Lucius, and Brünig," Höchst-on-the-Maine, Germany. Eng. Pat. 5690, March 16, 1893.

AN extension of Eng. Pat. 19,820 of 1892 (this Journal, 1893, 922), substituting for the dinitrochlorobenzene there mentioned, picryl chloride, dinitrochlorobenzenes, nitrobenzyl chlorides, &c., and reacting with them on tetra-ethyl- or tetra-methyl-*m*-amido phenolphthalein to form dyestuffs. The colouring matters are easily soluble in alcohol and acetic acid, and some of them also in water, and those from tetra-methyl-*m*-amido-phenol separate from their solutions as small crystals. The dyestuffs produce bluer and faster shades than rhodamine on mordanted cotton, whilst the shades on wool are yellower. A solution of 25 kilos. of picryl chloride and 44 kilos. of tetra-ethyl-*m*-amido-phenolphthalein in 75 kilos. of alcohol is boiled for a few hours, when the alcohol is distilled off and the colouring matter, which forms a tough mass, is either dried as such, or after having been dissolved in hot dilute hydrochloric acid, is filtered and the dyestuff precipitated from the filtrate by adding salt.—T. A. L.

**Manufacture of Acid Colouring Matters, being the Sulphonic Acids of Alkyl Derivatives of the Diamido-diphenyl-m-tolyl-carbinol.** O. Imray, London. From "The Farbwerke vormals Meister, Lucius, and Brünig," Höchst-on-the-Maine, Germany. Eng. Pat. 5691, March 16, 1893.

THESE colouring matters are obtained by sulphonating the alkyl derivatives of diamidodiphenyl-*m*-tolyl-carbinol of the general formula—



The leuco bases are produced by condensing *m*-tolyl aldehyde ( $\text{C}_6\text{H}_4 \cdot \text{CH}_2 \cdot \text{COH}$ , b.p. 199–200° C.) with secondary or tertiary aromatic bases. These leuco bases are sulphonated, and the leucosulphonic acids oxidised, or the same products may be obtained by first oxidising the leuco base and then sulphonating the carbinol thus produced. The following example gives the quantities employed:—59.6 kilos. of diethylaniline, 22 kilos. of concentrated sulphuric acid, and 24 kilos. of *m*-tolylaldehyde are heated for three days in a lead-lined vessel to 100° C. with constant agitation. After having been made alkaline, the melt is distilled with steam, and the leuco base remaining behind is boiled with water until no longer alkaline, and then dried at

100° C. In order to sulphonate it, it is dissolved in five times its weight of sulphuric acid monohydrate and the solution thus obtained is heated for some time to 80–90° C. until a sample dissolves completely in ammonia. The melt is then poured into water and converted into the calcium salt. The leuco calcium salt is dissolved in 50 times its weight of water, sulphuric acid (three equivalents) is added, and the solution is oxidised with the necessary quantity of lead peroxide. After filtering off the lead sulphate the blue solution is evaporated to dryness. Instead of first sulphonating and then oxidising, the leuco base may be oxidised, and the carbinol, a green colouring matter, afterwards sulphonated. In place of the diethylaniline employed above, the following secondary and tertiary aromatic bases may be used:—monomethyl or ethylaniline or *o*-toluidine, dimethylaniline or *o*-toluidine, diethyl-*o*-toluidine, methyl- or ethyl-benzylaniline.—T. A. L.

**Manufacture of Thionine Colouring Matters Dying on Mordants.** O. Imray, London. From "The Farbwerke vormals Meister, Lucius, and Brünig," Höchst-on-the-Maine, Germany. Eng. Pat. 5692, March 16, 1893.

THE process consists in blowing air through an alkaline solution of gallic acid or analogous bodies and the thio-sulphonic acids, mercaptans or disulphides, produced, according to Ger. Pat. 15,879, from the following diamines:—*p*-phenylene diamine, dimethyl-, diethyl- mono-ethyl-*p*-phenylene diamines, or ethyl-*p*-tolylene diamine: 2.5 kilos. of *p*-amido dimethylaniline thio-sulphonic acid and 1.7 kilos. of gallic acid are dissolved in 40 litres of water, heated to 60–80° C., and mixed with 4 kilos. of sodium hydrate as soda-lye. Air is then blown through the mixture until the formation of the colouring matter is complete, when it is precipitated by adding salt. The sodium salt is decomposed by acetic acid, and the acid colouring matter, which dissolves with difficulty, is made into a paste. It forms small bronze-coloured crystals, easily soluble in dilute hydrochloric acid or alkalis. The colouring matters give reddish-violet to blue shades on chrome mordants, according to the diamine employed, and are said to be fast to soap and fulling.—T. A. L.

**Improvements in the Manufacture of Auramine.** J. Y. Johnson, London. From the "Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 6249, March 23, 1893.

THIS is an extension of Eng. Pat. 12,549 of 1889 (this Journal, 1890, 498) and of Eng. Pat. 16,666 of 1890 (this Journal, 1891, 762). The methods there given describe the preparation of auramine from tetramethyl-diamido-diphenyl-methane or its derivatives, the corresponding hydrol and thio-hydrol, by treating them with sulphur and ammonia. It has now been discovered that tetramethyl-diamido-diphenyl dimethyl-methane can also be converted into auramine by treating it with sulphur and ammonia, under suitable conditions. This compound can be obtained by condensing acetone with dimethylaniline (Ber. 12, 813; 6, 345). The following quantities are given for the preparation of auramine according to the present invention:—About 14 kilos. of tetra-methyl-diamido-diphenyl-dimethyl-methane and 6 kilos. of sulphur are mixed with 120 kilos. of salt and 7 kilos. of ammonium chloride, and a stream of ammonia gas is passed through the mixture for eight hours at 175° C. The melt is first washed with cold water to dissolve away the salt and ammonium chloride, and is then dissolved in water at 70° C., filtered, salted out, pressed, and dried.—T. A. L.

**Manufacture of New Colouring Matters.** J. C. L. Dand, D. E. Huguenin, and A. J. J. d'Andron, St. Fons, Rhone, France. Eng. Pat. 24,892, December 23, 1893. Date under International Convention, July 4, 1893.

THESE colouring matters are obtained by the action of phenols and their derivatives on the oxazines and gallo-cyanines, and subsequent oxidation and sulphonation of

the substances produced. The colouring matters obtained are bodies soluble in alkalis, giving blue to violet solutions, and forming violet blue lakes with metallic mordants. 10 kilos. of gall-cyanine, obtained by condensing gallic acid with nitroso-dimethylaniline hydrochloride, 15 kilos. of resorcinol, 20 litres of water, and 90 kilos. of commercial hydrochloric acid are heated together on the water-bath until the bluish-violet coloration has disappeared and greenish-yellow crystals have separated. After diluting with water and washing free from acid, the green substance is oxidised with air in an alkaline solution, giving a violet-blue colouring matter after precipitation with hydrochloric acid much bluer than the original gallo-cyanine. This can then be sulphonated in the usual manner. The condensation product may also be sulphonated by dissolving in sulphuric acid, and subsequently oxidised by adding ferric oxide; or sulphuric acid may be used alone to act as both condensing and oxidising agent, as at 100° C. it acts as an oxidiser. Whichever method be employed, the product of the reaction is precipitated by water and washed free from sulphuric acid. If higher sulphonic acids have been formed it may be necessary to separate them as calcium salts, as these acids are soluble in water.—T. A. L.

and highly polished. When of sufficient thickness, the film is transferred on to the permanent fabric by passing it and the cloth face to face between rollers.—F. H. L.

*Improved Manufacture of Detergent for Washing and Cleansing Dirty Linen.* Annalie Gross, Szederjes, Austro-Hungary. Eng. Pat. 23,398, December 5, 1893.

See under XII., page 261.

## VI.—DYEING, CALICO PRINTING, PAPER-STAINING, AND BLEACHING.

*Note on the Different Theories relating to Dyeing.*

Leo. Vignon. Bull. Soc. Ind. Mulhouse, 1893, 407—410.

THE absorbing power that textiles, hide, and metallic oxides exercise on colouring matters, has been attributed to different causes, and generally explained by the mechanical and chemical theories; the former limiting its facts to that of absorption, whatever its cause; the latter explaining the production of dyeing by chemical agencies.

It has been stated that in the phenomena observed in dyeing, no chemical combination ensues between the coloring matter and the textile; the dyed result does not present the essential characters of a chemical compound. It is also stated that the colouring matters are not combined with the textiles, but in some way dissolved in them, this opinion suggesting the substitution for the term "absorption" by that of "solution," a view standing midway between the existing chemical and mechanical theories.

The author is of opinion that dyeing may be considered as involving chemical phenomena, in which dissociation occurs, and that dyeing only takes place when certain chemical functions are exerted by the absorbing body and the colouring matter; moreover, there are two facts not to be lost sight of, namely, the high molecular weight of the textile in comparison with the colouring matter, and dissociation. Although the molecular weight of the textiles is not known, there is every indication of its being very high, and it is difficult to conceive that a molecule weighing 4,000 units fixes 1, 2, . . . ,  $n$  molecules of a body, the molecular weight of which is relatively low; it is difficult to establish the law of definite proportions.

The phenomena of dissociation are frequently produced by the action of solvents; for example, aluminium chloride, carbonates, borates, alums, and ammoniacal salts are dissociated at the ordinary temperature by means of water; even in contact with absolute alcohol, hypochlorite of calcium is decomposed, chloride of calcium dissolving and lime remaining in suspension, this phenomenon being quite analogous to that of the coloration of alcohol by a magenta-dyed fabric.

In every case of dyeing there is inferred a system consisting of three elements, the absorbent, the colouring matter, and the solvent; and between these three a state of equilibrium is arrived at, determined by the chemical force and the condition of dissociation of the system; it may appear then that the law of definite proportions is not applicable to the phenomena of dyeing.

In the author's opinion the great majority of dyeing phenomena are of a chemical nature, chemical action sufficing to explain them, especially when we also consider the part that dissociation plays.

The general character of dyed substances is marked by the formation of a chemical compound, more or less dissociated by different solvents (this Journal, 1894, 120—127).—W. R.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

### PATENTS.

*A New or Improved Process for the Treatment of Textile Vegetable Fibres, and Apparatus connected therewith.* A. F. B. Gomess, London. Eng. Pat. 18,358, October 13, 1892.

THE invention relates to the preparation of textile fibres from the cortex of plants, particularly those of the Urtica family. Lime is first removed by steeping in a cold solution of sodium thiosulphate, whereby soluble calcium thiosulphate is formed. The epidermis is also deoxidised and rendered more soluble. A thorough washing follows, which finally removes the calcium compound. The fibre is next boiled with caustic soda and zinc dust, and again washed, then soaked in a dilute solution of sodium hypochlorite and hydrochloric acid, washed, and passed through weak ammonia to remove any traces of chlorine. Finally, it is washed and boiled with soap.

The apparatus employed in some instances consists of a hollow revolving drum, inside which a number of pegs are fixed, and into which hot air can be passed.—R. B. B.

*Improvements in or relating to Open Soaping, Washing, Boiling, and other similar Machines for Operating upon Textile Fabrics.* C. L. Jackson, Bolton, and E. W. Hunt, Manchester. Eng. Pat. 209, January 16, 1893.

THREE or more triangular beaters of wood or metal are arranged in such a manner above the level of the liquid in the washing machine that the cloth is struck and shaken three times during each revolution, the effect being as if it were suddenly pulled lengthwise between the top and bottom guiding rollers. These beaters remove hard and thick substances from the cloth.

The machines are charged with soap solution or other liquid by a pipe entering at the bottom of the vessel, and the liquid overflows at the top. The substances beaten out from the cloth are thus carried away at once, and precipitation is prevented.—R. B. B.

*Improvements in the Manufacture of Rubber coated Waterproof Fabrics.* W. Abbott and W. R. Wilks, London. Eng. Pat. 6769, March 30, 1893.

A COATING of rubber is prepared on a temporary support, consisting, for instance, of prepared rubber cloth, vulcanised

*Report on the Previous Note.* M. A. Rosenstiehl. Bull. Soc. Ind. Mulhouse, 1893, 411.

THE forces contributing to the dyeing process are of two kinds—

1. *Adherence or cohesion*, and this is always present. It is the mere attraction of matter by matter.

2. Chemical attraction, which often happens, but is not indispensable.

Neither the chemical nor mechanical theory by itself explains dyeing satisfactorily. It may be defined as the operation which consists in covering the textile fibre with a perfectly adherent layer of a coloured substance, which often is a combination of a portion of the substance of the fibre with a colouring matter.—W. R.

*Reports on Modified Starches prepared by Siemens and Halske.* Berlin. Bull. Soc. Ind. Mulhouse, October—November 1893, 363.

See under XVI., page 264.

*Soaps most Useful for Scouring Woollen Goods.* J. Lewkowitsch. J. Soc. Dyers and Colorists, 1894, 12—33.

See under XII., page 258.

## PATENTS.

*Improved Detergent Compound or Preparation for the Washing or Scouring of Textile Fabrics or other Articles.* W. Brothers, Rawtenstall, Lancashire. Eng. Pat. 6123, March 22, 1893.

See under XII., page 260.

*Improvements in the Method of and Means employed for Bleaching, Dyeing, and otherwise treating Flax, Hemp, and other Fibres.* A. McMeekin, Doagh, Ireland. Eng. Pat. 6600, March 29, 1893.

THE flax &c. in the form of "rove" is wound on perforated bobbins or tubes. The bleaching or other liquid, or hot air, is forced by pressure into the centre of the bobbins, and can find no other outlet save the perforations in the bobbins, through which it passes, and thence through the fibre, whereby the latter becomes thoroughly penetrated.

—R. B. B.

*Improvements in Dyeing Machines.* L. Weldon, Amsterdam, New York, U.S.A. Eng. Pat. 21,185, November 7, 1893.

THE machine described is adapted for dyeing loose wool or cotton, and consists of an open-work cage divided into two or more compartments, revolving in the dye liquor in such a manner that the fibre is alternately immersed in the dye bath and carried out of it. The particular improvement here is an arrangement whereby the rolling and consequent matting of the fibre during the revolution of the cage is minimised. In each compartment longitudinal ribs and "horns" extend from the axis of the cage part way towards its periphery. Opposite to these horns, and attached to the inside of the cage, are hooks, capable of swinging freely in one direction. The combination of "horns" and hooks is so arranged that the fibre in passing from one side of the compartment to the other, as it must do when the cage is revolving, does so in a gradual and gentle manner, thereby decreasing the matting effect, and increasing the evenness of dyeing.—R. B. B.

## VII—ACIDS, ALKALIS, AND SALTS.

*Production of Chemically Pure Caustic Soda and Hydrogen from Sodium.* M. Rosenfeld. J. Prakt. Chem. 48, 599—601.

THE explosions which occur when water and sodium interact, are attributed by the author to the formation within the metal of sodium hydride, which afterwards dissociates suddenly and violently. As this compound can only be produced when an atmosphere of hydrogen surrounds the sodium, the author has been led to try the action of a current of steam on sodium, the hydrogen being removed as it is formed. He finds that the reaction proceeds very smoothly without any tendency to explosion, and that pure solid caustic soda and hydrogen free from oxygen may easily be obtained in this way. The apparatus he uses consists of an iron crucible with two side tubes (for the admission of steam and the delivery of hydrogen, respectively) and a lid fastened down by means of a stirrup and screw. The caustic soda obtained always contains some finely-divided iron, probably from the formation of a sodium-iron alloy in the first stages of the reaction.—J. W.

*The Use of Electrical Heat for the Concentration of Sulphuric Acid.* A. H. Bacherer. Chem. Zeit. 17, 1597—1598.

IN order to produce 100 kilos. of 66° vitriol, 117 kilos. of 60° acid are requisite; taking this as having the temperature 18° C. to begin with, and the concentrated acid as being at 330° C. to end with, and further assuming the specific heat of the acid to be 0.33, the heat consumption during the heating up and evaporation will be as follows:—

	Calories.
1. Heating 60° acid from 18° to 330° C. ....	16,296
2. Heating acid, evaporation of 17 kilos. water, ..	12,283
3. Heat evolution during union of 60° acid with 1½ H <sub>2</sub> O, forming 66° acid, .....	10,000
	<hr/> 32,679

Hence  $32679 \div 4.2 \times 10^3$  Watts per second are required, or  $\frac{32679 \times 4.2 \times 10^3}{736 \times 3600} = 44.2$  horse-power per hour; or, allowing for loss of heat by radiation, &c., from the glass retorts, about 50 horse-power per hour. Admitting the practical cost of the current to be 0.04 mark per hour horse-power (0.01 shilling), the cost of concentrating one ton (1,000 kilos.) will be 20 marks (about 1*l.*).

IN order to calculate the dimensions of a platinum wire capable of furnishing the requisite heat, it is assumed that the mean temperature of the wire is 150° C. higher than that of the acid, i.e., is 480° C.; this amounts to supposing that with this excess of temperature of metal over that of acid, the rate of passage of heat into the fluid is just as rapid as its rate of generation by the current. If the difference of potential between the ends of the wire be 5 volts, a current of 364 amperes will develop in a wire 0.5 cm. diameter and 77 cm. long, 2.4 electrical units; hence, in the course of 5 hours, 12 units are produced, competent to concentrate 24 kilos. of 66° acid (starting at 60°) in a vessel suitably jacketed to minimise loss by conduction, &c. Whether, however, it will prove cheaper in practice to use a weaker current for a proportionately longer time, or *vice versa*, is a matter which cannot be decided *a priori* without actual trial. The calculations indicate at least the probability that electrically generated heat may be economically employed in the concentration of oil of vitriol, and that the process would not involve any special technical difficulty, but on the other hand would in various ways be more convenient than the methods now in ordinary use.—C. R. A. W.

*The Inertness of Quicklime. II. The Interaction of Chlorine and Lime.* V. H. Veley. Proc. Chem. Soc. 1893, [128], 209.

THE author has ascertained by synthetical and analytical methods that no appreciable action takes place between dry chlorine and quicklime below 300°; above this temperature, a partial displacement of oxygen is effected by the chlorine; the interaction is thus analogous to that of baryta and chlorine, not specially dried, and at ordinary temperatures.

*Tests of Purity of Wood Vinegar.* G. Buchner. Chem. Zeit. 17, 1319.

See under XXIII., page 285.

*The Determination of Formic Acid.* A. Lieben. Monats-h. Chem. 14, 746.

See under XXIII., page 288.

*The Action of Calcium Sulphite and of Potassium Bisulphite on Alcoholic Fermentation.* F. Ravizza. Le Staz. Sperim. agr. ital. 24, 1893, 593.

See under XVII., page 266.

#### PATENTS.

*Improvements in the Manufacture of Alkali, and in Apparatus therefor.* J. Hargreaves, Farworth-in-Widnes, and T. Bird, Cressington, Liverpool. Eng. Pat. 18,871, October 21, 1892.

ACCORDING to these improvements, relating in part to Eng. Pat. 18,039, 1892, alkali is obtained by electrolysis from chloride of sodium solution. The anode compartment, only containing the electrolyte, is bounded by a porous partition, in contact with which, on the outer side, or in proximity thereto, is placed a permeable cathode of iron gauze or perforated cathode, from which the alkali is washed off as electrolysis proceeds by steam or vapour, or spray or jet of liquid led on to it through a second containing chamber or tank, in which the soda is collected as solution at the bottom, but which does not contain the electrolyte, except such little as may pass from the anode compartment by filtration through the diaphragm, or which may be introduced in the form of steam or spray.

—J. C. R.

*Improvements in the Method of and Apparatus for the Electrolytical Separation of the Bases of Salts and of Compounds of the Same from Saline Solutions.* C. T. J. Vautin. Eng. Pat. 2267, February 1, 1893.

See under XI., page 256.

*Improvements in the Utilisation of Chlorides of Sodium and Potassium for the Obtainment of Useful Products.* T. Parker and A. E. Robinson, Wolverhampton. Eng. Pat. 1920, March 7, 1893.

THESE improvements relate to a method of removing or fixing caustic alkali resulting from electrolytic action from the sphere of such action, either as the caustic alkali is liberated, or from time to time before it accumulates in sufficient quantity to interfere in a material degree with the efficiency of the further electro-chemical reaction. The method or process of removal consists in combining with the alkali, some fatty acid, such as oleic, stearic, or margaric, either in the cathode compartment or separate tank to which the alkali is transferred. The compound thus formed, owing to its lower specific gravity than the electrolyte, rises to the surface and can be removed. The fatty acid may be added in the form of fat or oil, such as tallow, olive oil, cotton-seed oil, palm oil, &c. The soap

compound made may be used as soap, or treated with carbon dioxide, and the alkali converted into carbonate, undecomposed electrolyte being returned to the tanks.—J. C. R.

*An Improved Process of Manufacturing Bleaching Solutions by Electrolysis.* A. J. Boulton, London. From O. Knocher and F. Gebauer, Charlottenburg, Germany. Eng. Pat. 3578, March 17, 1893.

THESE inventors set out advantages in the electrolytic decomposition of a solution of common salt for the production of hypochlorites for bleaching purposes, from adding lime to the solution, which is not such a good conductor as hypochlorite of soda, nor its hypochlorite so readily reduced by hydrogen at the cathode; but in the present invention their claim is for a process of manufacturing a bleaching solution, consisting in subjecting a solution of mineral salt, without the employment of diaphragms, to electrolysis with a density of current of from 300 to 800 amperes per square metre of electrode surface, the extent to which the lye is made richer in active chlorine being determined by correspondingly regulating the rise of temperature, while, conversely, by maintaining a uniform rise of temperature an equally uniform percentage of active chlorine may be secured in the lye produced. A 10 per cent. solution of salt being preferred, the quantity which passes through the electrolytic tank in a given time may be said to regulate both temperature and percentage of available chlorine in the body of liquid treated, and may be made guides for practical work without chemical analysis; at least, such seems to be the idea of these inventors, who give a short table of results, and refer to Eng. Pat. 20,214, 1892 (this Journal, 1893, 933).—J. C. R.

*Improvements in the Manufacture of Nitric Acid.* M. Prentice, Woodfield, Suffolk. Eng. Pat. 6960, April 4, 1893.

THE object of the invention is to manufacture nitric acid by a continuous process. A still or vessel is provided, having an inlet for the nitrate and acid, and a siphon or other outlet for the fluid mixture, as well as an outlet for taking off the nitric acid as it distils. The still may be divided into separate compartments, these communicating with each other from below. The inventor does not limit himself to any particular construction of still.—E. S.

*Improvements connected with the Manufacture of Nitric Acid, Muric Acid, or the like, where Sulphuric Acid is employed, and of Superphosphate of Lime.* M. Prentice, Woodfield, Suffolk. Eng. Pat. 8902, May 3, 1893.

IN the manufacture of nitric or hydrochloric acid or the like, wherein sulphuric acid is used, the manufacture is facilitated by using the sulphuric acid in large excess. The available acid left in the acid sodium or other sulphate formed, is then employed in the preparation of superphosphate, by running off the acid product and mixing it with calcium phosphate.—E. S.

*Process for the Production of Alkali Cyanides.* W. Siepermann, Elberfeld, Germany. Eng. Pat. 13,754, July 15, 1893.

ALKALINE cyanides are obtained by heating alkaline carbonates, or alkaline compounds convertible into carbonates in the process, with fuel, aided by a hot blast. Pieces of wood or coke fuel may be saturated by a solution of the alkali carbonate and dried; or the materials may be mingled, the proportions being about one part of the carbonate to two or three parts of the fuel. The charge is introduced through a hopper box with double closure, to prevent escape of poisonous gases; but when a nitrate or nitrite is used instead of a carbonate, it is run in a fused state through a funnel into a pipe connecting one of the heat-regenerating chambers to the furnace, so that the hot blast projects the salt in



spray on to the fuel, to which a small addition of alkaline carbonate has been made. The reacting chamber or furnace containing the charge has a floor inclined towards the tapping hole, and a hearth extended in an opposite direction, joining a pipe which carries the gases and vapour of the cyanide into a cooler furnished with spiral wings or plates, wherein the alkaline cyanide settles as powder. The cooler is a large iron vessel mounted on flange wheels running on rails for convenience of replacement. From the cooler the gases pass into a coke-scrubber, where they are washed free from alkali compounds, and thence to one of the two regenerators to be burnt. "The residual cyano compounds contained in the gases are obtained in the form of red or yellow prussiate of potash by washing with water to which iron salts have been added."—E. S.

*Improvements in Apparatus and Furnaces for Concentrating Sulphuric Acid and other Liquids.* J. Bradbury, Manchester. Eng. Pat. 22,327, November 22, 1893.

A SERIES of evaporating vessels of lead or other suitable metal, earthenware, or glass, is set upon a metal plate, which plate, as well as the chambers containing the vessels, are heated by flues from a fireplace set at the opposite end of the furnace to that at which the acid to be concentrated enters. The vessels are at one uniform height, and discharge one into another, either by a spout or by a siphon tube reaching nearly to the bottom. If a spout be used, the overflow is into a tube bent horizontally at the lower end close to the bottom, so as to ensure thorough mixing of the lighter entering acid with that more concentrated in the vessel. Metallic shields, made in halves, and secured by bolts passing through lugs, are provided to protect the evaporating vessels from the heat; but these may be dispensed with in the cooler part of the furnace. A closed-in space above the evaporators is connected by suitable openings to a pipe conveying the vapour and fumes given off to a stack. An acid tank, heated by flues, is set over the chamber containing the series of vessels, and supplies by a tap or valve a small box, from which the acid is led to the tube delivering at the bottom of the first vessel of the series. As this vessel fills, it discharges through its spout into the tube of the next vessel, and so on through the series. But if the siphon arrangement be used, the acid tank is placed at the side of the furnace, nearly level with the evaporating vessels, and is supplied with a well, into which the longer limb of a siphon dips, the other limb entering the first vessel of the series. Another siphon tube, with limbs of equal length, connects this vessel with the next, and so throughout. When the acid in the tank (which is fed through a pipe with tap) reaches the level of the tube connecting the limbs of the siphon, the vessels are charged with acid in succession through the tubes. The vessels may be emptied from another corresponding acid tank forming the terminus, by opening a tap at the bottom of the well, and so running out the acid. The conduct of the process may be modified in various ways.—E. S.

*Improvements in Apparatus for Electrolytically Producing Soda and Chlorine.* H. Lake and Co., London. From the Union Chemical Company, New York, U.S.A. Eng. Pat. 23,436, December 5, 1893.

See under XI., page 257.

*Improved Process and Apparatus for Dissociating Soluble Salts by Electrolysis.* H. Haddon and Co., London. From H. S. Blackmore, Mount Vernon, New York, U.S.A. Eng. Pat. 23,913, December 12, 1893.

See under XI., page 257.

*Improvements in the Concentration or Vaporisation of Sulphuric and Acetic Acids, and in Apparatus therefor.* A. J. Bault, London. From S. C. Penchen and P. Clarke, Toronto, Canada. Eng. Pat. 24,739, December 23, 1893.

THE objects of the process are, to precipitate impurities from the sulphuric or acetic acid treated, and to effect the concentration or vaporisation of the acid, as may be desired, and it is stated that "these objects are accomplished by passing through the acid an electric current from acid-proof electrodes of high electrical resistance, either held stationary or arranged so that they may be revolved in the liquid," the necessary heat being produced within the liquid by the electrical resistance. The sulphuric acid to be concentrated (for instance) may be contained in a glass retort within which is a porcelain stand supporting two carbon electrodes connected to a dynamo or battery by platinum wires. The current is preferably of low voltage. If the acid is to be distilled, the retort is connected to a suitable condenser, and a stronger current in amperes is required. An apparatus is shown, in which a hollow revolving shaft carrying arms with electrodes at their extremities is fitted within a close vessel containing the acid to be treated. Insulated wires are connected to the electrodes, and to metal terminals at the ends of the shaft, details of the arrangements being shown and described. Alternatively, the shaft carrying the arms with electrodes, may be rigidly fixed.—E. S.

## VIII.—GLASS, POTTERY, AND ENAMELS.

### PATENT.

*An Improved System and Apparatus for Removing Impurities from and Refining China Clay.* W. W. R. Nicholls, W. Nicholls, and F. H. Nicholls, St. Austell. Eng. Pat. 19,699, October 19, 1893.

EACH "set" of the elutriating apparatus used for washing china clay is provided with "mica traps" operated by eccentrics on a lever disposed at right angles to the set and actuated by a handle turning in a quadrant with locking holes and pins so that the traps may be set at any desired angle. The purified clay passes into a refiner with a wire-gauze covering kept clean by revolving brushes, which are themselves cleansed by a jet of water at one part of their revolution. The brushes are driven from a water-wheel hung in the launder conveying the stream of purified clay, suspended in water, to the settling pit.

—B. B.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*The Effect of the Addition of Gypsum to Portland Cement.* Schott. Thonind. Zeit. 1893, 17, 926.

THE author has investigated Michaelis' explanation of the accelerating action of the addition of gypsum to Portland cement as being due to the formation of a double salt ( $\text{Al}_2\text{O}_3, 3\text{CaO} + 3\text{CaSO}_4$ ), and has experimented with cements made from a mixture corresponding to a formula of  $2\text{CaO}, \text{SiO}_2$ , and from a mixture of marble and kaolin. His results leave it uncertain whether the accelerating action is due to the formation of this double salt or whether to the crystallisation of calcium hydrate.

—J. G. W.

*The Characteristics of Portland Cement.* R. Fresenius and W. Fresenius. *Zeits. Anal. Chem.* **32**, 433—445.

This article is a continuation and amplification of the authors' communication published in 1884 "On the Detection of Adulterations in Portland Cement" (*Zeits. Anal. Chem.* **23**, 175; this Journal, 1884, 373), and gives the results of investigations undertaken, some three years ago, on behalf of the Society of German Portland Cement Makers. One of the objections raised to the figures given in their first communication was that they were obtained from too small a number of samples, and another was that these samples were not representative of actual commercial cements. In a second paper published in 1885, the authors replied to this criticism (*Zeits. Anal. Chem.* **24**, 66; this Journal, 1885, 349), and stated their conclusion that any further investigation of the subject was unnecessary until a more exact definition of what constituted "Portland cement" had been formulated and become recognised. In 1885, at a general meeting of cement makers, a definition or specification of what constituted "Portland cement" was unanimously adopted, and in 1888 the Society of German Portland Cement Makers was founded, and adopted the following resolutions as a basis:—

"The undersigned members of the Society of German Portland Cement Makers bind themselves to place on the market under the designation 'Portland cement,' only one kind of commodity, which shall be made by burning to a frit an intimate mixture of materials containing lime and clay as their essential constituents, and grinding the product to the fineness of meal.

"Any product made in any other way than the above, or to which any foreign matter has been added either during the burning or afterwards, shall not be recognised by them as 'Portland cement,' and the sale of such under the designation 'Portland cement' shall be held to be a deception practised on the buyer.

"This provision does not refer to small additions made to regulate the time required for the 'setting' of the Portland cement, which additions shall be permissible to the extent of 2 per cent."

The establishment of this clearer definition of "Portland cement" made it possible and desirable to redetermine the limits of the constituents found in various qualities of Portland cement, and especially was an extension of the work upon the determination of the contents of magnesia needful, because, in the interval, the signification of the contents of magnesia had become a question of first interest.

Practical experience of the durability of cement used in various public works has shown that less than 3 per cent. of magnesia was harmless, but that appreciably higher amounts than this produced very serious evils. Now, since the adopted definition excludes Portland cements containing large amounts of magnesia, those containing small quantities are not excluded by it, and therefore it became necessary to decide what amount of magnesia should be permissible in the sense of this definition. For this purpose a determination of the average amount of magnesia contained in the Portland cements manufactured at the time in Germany was undertaken.

The samples were supplied by the executive of the Society, which is also responsible for their authenticity, and were for the most part taken from cements actually in commerce, and not, as in the case of the authors' first investigations, from cements chiefly derived from the makers' works. On the former occasion, some English and French cements were examined, but in this investigation they were all samples of German makes. Both big and little works are represented, and the 25 samples examined represent a total of 5,750,000 casks of cement per annum, the total German output in 1889 being 7,950,000 casks.

The lines of the present investigation differ from those of the first, by the specific gravity in the calcined state and the contents of magnesia being now determined, whilst the determinations of the amount of acid used up on shaking with the cement, and of the capacity for absorbing carbonic acid, are omitted as being unnecessary.

The results obtained are given in the following table:—

Mark.	Specific Gravity.		Loss on Calcination.	Alkalinity of the Aqueous Solution of 0.5 grm., stated in Decinormal Acid.	Magnes. Potassium-Permanganate reduced by 1 grm.	Percentage of Magnesia.	Strength.		Fineness.		
	Uncalcined.	Calcined.					Kilos. per sqm. Section.		Residue left on Sieve of		
							Pressure.	Pull.	5,000 Meshes.	900 Meshes.	
			Per Cent.						Per Cent.	Per Cent.	
Cement 1...	3.611	3.163	3.37	7.20	0.33	1.46	212.5	18.4	27.0	6.3	Slow setting.
" 2...	3.631	3.176	3.11	3.95	0.37	1.89	160.0	15.1	22.5	3.0	Quick setting.
" 3...	3.600	3.172	1.74	4.60	0.29	0.75	169.3	17.3	25.7	4.8	
" 4...	3.604	3.150	1.22	5.90	0.23	0.63	187.5	18.4	24.2	3.5	
" 5...	3.679	3.168	1.63	4.35	0.16	1.40	232.5	21.1	25.0	5.3	
" 6...	3.126	3.185	1.61	5.86	0.23	0.80	212.5	21.3	27.5	8.0	
" 7...	3.111	3.128	1.42	5.35	0.21	0.91	185.0	18.6	24.0	3.5	
" 8...	3.683	3.118	1.57	4.33	0.40	1.96	190.0	20.9	27.2	6.0	
" 9...	3.080	3.123	1.54	4.60	0.22	0.62	216.0	21.1	22.7	4.5	
" 10...	3.130	3.150	1.15	4.15	0.38	1.705	170.0	17.1	28.7	7.5	Slow setting.
" 11...	3.612	3.183	2.96	5.25	0.80	0.80	180.0	19.5	22.7	3.0	
" 12...	3.664	3.157	2.92	3.33	1.48	1.46	185.0	19.7	18.0	2.5	
" 13...	3.679	3.165	1.61	3.00	0.93	0.35	215.0	21.1	15.7	2.5	
" 14...	3.600	3.167	2.34	4.97	1.30	1.97	215.0	19.5	25.2	7.0	
" 15...	3.645	3.140	1.86	3.65	1.00	0.74	180.0	18.4	25.8	5.5	
" 16...	3.655	3.131	2.05	3.95	1.48	3.06	160.0	17.4	20.5	4.7	
" 17...	3.126	3.194	1.73	4.10	0.95	2.49	135.0	16.1	32.0	12.5	Quick setting.
" 18...	3.692	3.182	1.88	5.49	2.19	2.83	149.0	12.7	23.7	3.5	

Mark.	Specific Gravity.		Loss on Calcination.	Alkalinity of the Aqueous Solution of 0.5 grm. stated in cc. Decinormal Acid.	Mgms. Potassium Permanganate reduced by 1 grm.	Per-centage of Magnesia.	Strength.		Fineness.	
	Uncalcined.	Calcined.					Kilos. per sqm. Section.	Pail.	Residue left on Sieve of 5,000 Meshes.	900 Meshes.
			Per Cent.						Per Cent.	Per Cent.
Cement 19..	3.103	3.219	1.19	5.25	0.26	1.19	210.0	20.4	18.0	3.5
" 20..	3.076	3.176	2.77	5.85	0.37	1.60	202.5	20.6	20.5	2.7
" 21..	3.113	3.179	0.79	3.60	0.62	0.90	207.5	20.5	24.5	2.8
" 22..	3.084	3.141	1.96	5.20	0.26	0.92	187.5	16.1	30.5	8.0
" 23..	3.126	3.184	1.73	4.95	0.31	0.92	210.0	19.6	23.2	3.7
" 24..	3.072	3.175	2.45	4.15	0.105	1.92	220.0	21.8	24.2	3.0
" 25..	3.144	3.234	1.43	5.80	0.26	2.04	182.5	18.1	22.7	3.5

Slow setting.

Quick setting.

From this table it will be seen that the following maxima and minima have been obtained.

The specific gravity of the uncalcined cement lies between 3.011 and 3.144.

The specific gravity of the calcined cement lies between 3.118 and 3.234.

The loss on calcination is found to be from 0.79 to 3.37 per cent.

The alkalinity of the aqueous solution of 0.5 grm. of cement lies between 3.00 and 7.20 cc. of  $\frac{1}{10}$  normal acid.

The potassium permanganate reduced by 1 grm. of the cement lies between 0.105 mgrm. and 2.19 mgrm.

The percentage of magnesia lies between 0.35 and 3.064.

Comparing these numbers with those obtained in 1884, some slight differences are noticeable.

To begin with the least important, the potassium permanganate reduced is less in many cases than was formerly found.

The alkalinity in several cases does not reach the lowest limit of the previous tests, but this is unimportant, as it is only when the upper limit is exceeded that this determination has significance. In these samples the maximum is lower than the maximum of the earlier tests, with the exception of sample No. 1, which exceeds the maximum considerably.

The loss on heating to redness is generally higher than in the former tests, which is no doubt explained by the fact that the samples were taken from cements that in many cases had been stored some time, and were not direct from the works, as in the earlier tests.

The most noteworthy difference is in the lower results that have this time been found in the specific gravities of the uncalcined cements. With regard to this point also it may be mentioned that experience has shown that the specific gravity of cement falls after storing.

With regard to the specific gravity of the calcined cement, this seems to be greater than the specific gravity of the uncalcined cement, according as the loss in heating increases, but no fixed relation between the three can be drawn from the results obtained.

The maximum contents of magnesia is 3 per cent., and this result agrees with the percentage declared in 1888 by the German Portland cement makers, to be quite harmless to the qualities of Portland cement.

Cement No. 1 requires special notice, as it gave the greatest alkalinity, and the greatest loss on heating, whilst its specific gravity in the uncalcined state was the lowest of all the samples examined. On comparison of this cement with another from the same firm, which only showed an alkalinity of 3.40 cc., and 2.09 per cent. of loss on heating, and also from a consideration of its high specific gravity after calcination, and of its strength, the authors are left in no doubt that the cement is not abnormal, but that these variations from the respective values found in the case of the other samples of cement are due to the length of time that

cement No. 1 had been stored. By taking the low specific gravity and the high alkalinity of this cement as limits, the authors think they have provided sufficiently against any cement which happens to have been stored a long time being mistaken for an abnormal cement.

From the results obtained in their first investigation, and from those now obtained, the authors consider the following to be limiting values characteristic of Portland cement:—

1. Specific gravity in the uncalcined state, not less than 3.00.
2. Specific gravity in the calcined state, not less than 3.12.
3. Loss on calcination, not more than 3.4 per cent.
4. Alkalinity of the aqueous solution of 0.5 grm. of cement, not more than 7.2 cc. of decinormal acid.
5. Potassium permanganate reduced by 1 grm. of cement, not more than 2.8 mgrms.
6. Magnesia in the cement, not more than 3 per cent.

The loss on calcination was determined by heating portions of 10 grms. of the cement to redness, in a covered platinum dish for at least one hour over a large gas flame. (Maste's burner). It was then kept in a desiccator, and afterwards used for determining the specific gravity in the calcined state, for which purpose Schumann's apparatus was used. The estimation of the magnesia was made by dissolving about 2 grms. of the cement in hydrochloric acid, evaporating to dryness to render silica insoluble, filtering, and then precipitating the iron and alumina from the filtrate by ammonia. This precipitate was redissolved and again precipitated by ammonia, and from the united filtrates the lime was separated by ammonium oxalate, this precipitate being also redissolved and reprecipitated. The filtrate now obtained was evaporated to dryness, the residue calcined to expel ammonia salts, and the remaining substance dissolved in dilute hydrochloric acid. Any traces of iron, alumina, and lime still remaining were then removed by further treatment with ammonia and ammonium oxalate, and the magnesia finally precipitated as ammonium-magnesium phosphate and weighed as magnesium pyrophosphate. An attempt to shorten the method by a modification doing away with some of the filtrations had to be abandoned, as the results obtained were not sufficiently exact.

Should a cement give results that fall outside any of the limits above specified, it must be more particularly examined with a view of finding if foreign matter be present, or if it be a sample of under-burnt cement. In the latter case its quality can be further judged off by the usual mechanical tests.

The authors, in conclusion, give the results of some investigations made by them in 1885, on—

1. The behaviour of strongly-burnt cements when tested by their method.
2. On the influence on their method of manganese dioxide (manganese ore) mixed with cement.

3. On the behaviour of some other substances of interest in this connection when tested by their method.

In these tests the magnesia and the specific gravity in the calcined state were not determined.

1. Experiments with strongly-burnt cements made of the same raw materials.

The results obtained are given in the following table:—

Mark.	Specific Gravity.	Loss on Heating.	Alkalinity of Aqueous Solution of 0.5 Grm. Ce. in Normal Acid.	Ce. Normal Acid consumed by 1 Grm. of Cement.	Mgms. Potassium Permanganate reduced by 1 Grm.	Mgms. of Carbonic Acid absorbed by 3 Grms.
		Per Cent.				
Not quite fritted....	3.083	3.59	7.86	20.2	0.82	2.1
A. Slightly fritted.....	3.105	0.66	3.71	20.3	2.83	0.3
Strongly fritted....	3.115	0.27	3.74	21.6	1.19	0.0
Over-burnt.....	3.05	0.05	2.75	18.32	0.74	5.8
Not quite fritted....	3.094	1.47	7.5	17.8	0.87	0.9
B. Slightly fritted.....	3.15	0.59	3.64	20.5	0.81	0.3
Strongly fritted.....	3.18	0.19	5.09	19.8	0.94	0.0
Over-burnt.....	3.05	0.28	1.57	23.18	0.91	0.0
Slightly fritted.....	2.92	5.39	0.3	18.1	0.62	31.1
C. Strongly fritted.....	3.00	2.36	4.3	21.0	0.65	15.7
Very strongly fritted	3.19	0.21	2.7	18.9	0.97	1.9

These samples were from three different works, A., B., and C. respectively. From the above table it may be seen that there is a marked difference between the under-burnt and the over-burnt cements, as compared with the "slightly fritted" and the "strongly fritted" made of the same raw materials. But there appear to be some discrepancies on comparing the products of one works with those of the others, but whether this is due to differences in the materials used, or to some different understanding of the term "fritting," the authors are unable to say.

2. The effect of mixing manganese with the cement was investigated, because it was suggested that the value of the test with potassium permanganate, which is important as an

indication of adulteration with slagmeal, would be interfered with, supposing both slagmeal and manganese ore were mixed with the cement. The results show that, although a somewhat less quantity of permanganate is reduced when manganese dioxide has been mixed with Portland cement containing slagmeal, nevertheless in order to neutralise the reducing effect of the slagmeal, the quantity of manganese ore must be so large as to be easily detected by the dark colour of the cement and the chlorine evolved on dissolving it in hydrochloric acid.

3. Three samples of Roman cement, one sample of coal clinker, and one sample of Pozzuolana were tested by the authors' process and gave the following results:—

Mark.	Specific Gravity.	Loss on Heating.	Alkalinity of Aqueous Solution of 0.5 Grm. = Ce. in Normal Acid.	Ce. Normal Acid used up by 1 Grm. of Substance.	Mgms. of Potassium Permanganate reduced by 1 Grm.	Mgms. Carbonic Acid absorbed by 3 Grms.
		Per Cent.				
Roman cement, A.....	3.08	8.44	1.70	20.55	..	1.7
" B.....	2.80	14.28	6.30	15.45	..	4.1
" C.....	3.06	6.33	1.49	16.52	..	2.8
Coal clinker.....	2.69	0.41	0.82	1.80	12.40	2.7
Pozzuolana.....	2.69	6.73	15.27	24.12	27.90	10.0

—H. S. P.

#### PATENTS.

*Manufacture of Artificial Marbles.* F. Grand' Montagne, Brussels, Belgium. Eng. Pat. 1582, January 24, 1893.

MIXTURES suitable for the production of artificial stones having a basis of powdered marble or other stone, and Keen's cement, or Portland cement, are mixed with zinc chloride and zinc oxide, or with magnesium chloride, and separate portions, tinted with as many different colours as may be required, are dashed on to the bottom of a mould so as to produce a veined pattern which after filling up the mould, constitutes the ornamental surface of the artificial marble. The block may be veined throughout by the insertion and subsequent withdrawal of silk threads or wires covered with silk soaked in appropriate colours. The product may be polished by treatment with a hardening

liquid, e.g., zinc chloride followed by polishing with a mixture of alcohol 75 per cent., oil 5 per cent., plaster 5 per cent., white gum lac 5 per cent., turpentine 5 per cent., and yellow wax 5 per cent.—B. B.

*Improvements in the Manufacture of Cement.* W. Webster, Blackheath. Eng. Pat. 3106, February 11, 1893.

This patent is a development of Eng. Pat. 11,765, 1892 (this Journal, 1893, 604), in which sewage sludge is utilised in the manufacture of cement. In addition to or in place of the sludge, the patentee claims the use of the ash of waste vegetable matter or of coal or coke, which is mixed with the clay and chalk used in the manufacture of the cement,

in proportion depending upon the composition of the clay. The quantity of ash added is from 1–5 per cent. of the whole cement mixture.—B. B.

*Improvements in the Utilisation, in the Manufacture of Portland Cement, of Precipitated Carbonate of Lime forming Waste Products from Chemical Works and in Apparatus for Employment thereon.* J. Hill, Wolverhampton. Eng. Pat. 5132, March 9, 1893.

The process is designed to mix precipitated carbonate of lime, such as that recovered in Chance's sulphur recovery process, with clay for the production of Portland cement, with the use of less water than is customary, to diminish the cost of drying. The clay is fed through a hopper into a barrel-shaped vessel in which a shaft having wedge-shaped beaters keyed to it, revolves. The disintegration of the clay is aided by blowing in steam during the process. The precipitated carbonate of lime is then added and incorporation continued until the material is homogeneous, when it is discharged through a man-hole at the bottom of the barrel-shaped vessel and is dried and burnt in the usual way. Economy of water, and therefore saving in cost of drying, are claimed for this process and apparatus.—B. B.

*Improvements in the Manufacture of Artificial Stone, Preservative Paint and other Analogous Materials.* L. Grote, Landberg-on-Lech, Germany. Eng. Pat. 17,142, September 12, 1893.

VARIOUS recipes are given containing magnesium chloride, magnesia, calcium plumbate, water and such filling materials as slagwood, sand, gravel, clay, wood, wood-pulp, wood-shavings, sawdust, paper, vegetable and animal fibres, resinous and fatty matters, are patented, the ingredients and their proportions being varied according to the character of the composition required, which may be used for building stone, insulating material, plaster, or paint.—B. B.

## X.—METALLURGY.

*Preparation of Metallic Lithium.* M. Gnutz. Comptes rend. 1893, 117, 732–733.

The author finds that the best yield of lithium is obtained when the temperature at which electrolysis occurs is low. In order to assist the fusion of lithium chloride it is mixed with an equal weight of potassium chloride, such a mixture melting at about 450° C. The positive electrode is of carbon and 8 mm. in diameter, and the negative electrode is a rod of iron 3–4 mm. in diameter, and enclosed in a glass tube 20 mm. in diameter. On passing a current of 10 ampères and of an electro-motive force of 20 volts, electrolysis rapidly proceeds and the lithium becomes deposited within the glass tube.

The metal thus prepared contains about 1–2 per cent. of potassium, and is sufficiently pure for most purposes.

—A. K. M.

*The Action of Gaseous Hydrochloric Acid and Oxygen on the Platinum Metals.* W. L. Dudley. J. Amer. Chem. Soc. 1893, 15, 272–274.

THE author observed that finely-divided platinum sponge on being washed with hydrochloric acid gave evidence of solubility. On moistening platinum black on a filter with dilute hydrochloric acid he found that the paper became yellow after a few hours, and on washing, the filtrate contained platinum chloride. On continuing the washing even with hydrochloric acid, platinum ceased to be present in the filtrate, but on standing exposed to air the attack of the platinum again became manifest. The action of air and hydrochloric acid on platinum sponge was similar to, but

less marked than, that of the same substances on platinum black. When dried HCl and air were used similar results were noted; the action of HCl and oxygen dried and moist was sensibly the same, both at ordinary temperatures and at 100° C.

It has already been shown that finely-divided palladium dissolves by the action of hydrochloric acid and oxygen or air (Fischer, Schw. 51, 132; Pogg. 71, 131).

The author experimented with iridium, rhodium, ruthenium, and osmium and obtained like results. The action on ruthenium and osmium was greatly increased on heating, especially to 250° C. or more, and the action was comparatively rapid, the chlorides subliming. The metals used in these experiments were not specially purified, but bought of good makers as "pure"—B. B.

*An Examination of the Ordinary Methods for the Estimation of Carbon in Iron.* A. Ledebur. Chem. Zeit. Rep. 17, 1893, 259.

See under XXIII., page 281.

*Estimation of Chromium in Chromium Steel and Chromium Ore.* J. Speller and S. Kalman. Chem. Zeit. 17, 1893, 1360 and 1412.

See under XXIII., page 281.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

*The Use of Electrical Heat for Concentrating Sulphuric Acid.* A. H. Bucherer. Chem. Zeit. 17, 1597.

See under VII., page 249.

*Purification of Water.* Villon. Revue chim. ind. 1893, 87.

See under XVIII. B., page 271.

*The Electro-deposition of Iridium.* W. L. Dudley. J. Amer. Chem. Soc. 1893, 15, 274–276.

ACCORDING to the author's experiments made in 1884, it is not practicable to prepare baths of iridium solutions electrolytically, strictly chemical methods being necessary. It follows that the strength of the bath cannot be maintained by the dissolution of an iridium anode, and that other means must be adopted for meeting this need. Finally he found that the strength of the bath could be kept constant by surrounding each anode (a carbon plate) with a loosely-fitting linen bag containing  $\text{Ir}(\text{OH})_3$ , the acid liberated at the anode being thus immediately neutralised. The salts suitable for use as electrolytes are sodium iridochloride, ammonium iridochloride, and the double sulphate of iridium and ammonium. Iridium, like all hard and brittle metals, tends to form blisters in depositing, and needs care in the adjustment of the current density.

The method described above was discovered independently by W. H. Wahl in 1890.—B. B.

## PATENTS.

*Improvements in Electrolytic Cells and Diaphragms.* J. Hargreaves, Farnworth-in-Widnes, and T. Bird, Cressington. Eng. Pat. 18,039, October 10, 1892.

A DIAPHRAGM or cell is formed by depositing a pulp of asbestos, vegetable or other suitable fibre, upon the inside (apparently) of a vessel or diaphragm of the required shape and made of wire gauze or perforated sheet metal. Upon

this is deposited a second stone-like layer of such material as Portland cement, or a mixture of clay and sodium silicate. The metal of the diaphragm forms the cathode, the part of the diaphragm in contact with it being rendered specially porous as above to facilitate the liberation of the hydrogen; this specially porous layer may, however, be dispensed with in certain cases.—E. T.

*Improvements in the Manufacture of Alkali and in Apparatus therefor.* J. Haigreaves, Farnworth-in-Widnes, and T. Bird, Liverpool. Eng. Pat. 18,871, October 21, 1892.

See under VII., page 250.

*Improvements in Means or Apparatus for Use in the Application of Electricity for Heating Liquids, and in Means or Apparatus for Manufacturing the Same.* R. E. B. Crompton and H. J. Dowsing, London. Eng. Pat. 258, January 5, 1893.

TUBES of copper or other material with a suitable coefficient of expansion by heat, are covered with a coating of enamel of high melting point, and mounted in a lathe in such a manner as to allow of their being heated while rotating. The wire, for which copper or a copper alloy is not suitable, is then wound on, whilst the enamel is in a viscous state, and is covered with a second coat of enamel, the process being finally completed in a furnace.

For use in geysers, &c., these tubes may be filled with sand, and bent while hot into spiral or other convenient form. A former patent, No. 17,091 of 1892, contains details as to enamel to be used, and the method of heating it.—E. T.

*An Improved Method of Manufacture of Elements or Plates for Secondary Batteries.* H. F. K. Picard and H. Thame, London. Eng. Pat. 600, January 11, 1893.

SUFFICIENT lead is poured into a hot metallic mould, "lubricated" with graphite or such material, to form a "pole rim" to which the external electrical connections are to be made. The mould is then filled up with a sub-sulphide of lead, formed by pouring molten lead slowly from a height into melted galena. On cooling, the plate is in a finely-crystalline, homogeneous state, and in metallically good connection with the "pole rim." It is intended, by the elimination of the sulphur during the after-forming process, to leave the plate in a very porous state, while the inventors claim that it is still mechanically strong.—E. T.

*Improvements in the Method of and Apparatus for the Electrolytical Separation of the Bases of Salts and of Compounds of the Same from Saline Solutions.* C. T. J. Vantin, London. Eng. Pat. 2267, February 1, 1893.

THIS invention relates to the separation by electrolysis of the bases of salts, and compounds of the same, from saline solutions, in which the base of the salt electrolysed is to be obtained as metal or amalgam, or a solution of any desired salt, a diaphragm of the usual form being dispensed with by a liquid mercury cathode supported by a reticulated mesh or sieve or perforated plate of non-conducting or insulated conducting material. Apparatus is shown and described, in which the mercury is held in a supporting frame for vertical, horizontal, and oblique or slanting use; in the last case the sieve, perforated plate, &c., being provided with upright divisions to support mercury thereon in a series of steps or terraces. Methods for making liquid-tight joints around the walls of the mercury compartments are described, to separate the electrolytic face of mercury from the back or opposite face, which may be in contact with solution different from the electrolyte, as oil or water, the former to obtain amalgams, and the latter solutions as sodium hydrate from sodium salts. The inventor says that mercury will not pass through the apertures of a reticulated mesh or sieve or a finely perforated sheet up to a coarseness of about 60 meshes to the inch, or equivalent fineness of perforations, without considerable shock or pressure, but will support a small layer or head having an active surface of

about 60 per cent. of the whole area of the sieve or mercury layer. In the horizontal forms of arrangement the escape of hydrogen or other gas freed upon the active surface of the mercury must be arranged for.—J. C. R.

*Improvements in the Manufacture and Production of Elements for Secondary Batteries.* The Lithanode and General Electric Company, Limited, and J. T. Nihlett, London. Eng. Pat. 4691, March 3, 1893.

TO render lithanode plates more porous and so allow of greater discharge rates, an inert or comparatively inert material is added to the ingredients of the plates—litharge and ammonium sulphate, usually. This material, magnesium sulphate or other suitable salt, is afterwards dissolved out, leaving the plate more porous.—E. T.

*Improvements in the Utilisation of Chlorides of Sodium and Potassium for the Obtainment of Useful Products.* T. Parker and A. E. Robinson, Wolverhampton. Eng. Pat. 4920, March 7, 1893.

See under VII., page 250.

*An Improved Process of Manufacturing Bleaching Solutions by Electrolysis.* A. J. Boulton, London. From O. Knoefer and F. Gebauer, Charlottenburg, Germany. Eng. Pat. 5578, March 17, 1893.

See under VII., page 250.

*Improvements in Storage Batteries.* W. P. Thompson, Liverpool and Manchester. From M. Hartung, Berlin, Germany. Eng. Pat. 5844, March 18, 1893.

A GRID is constructed of intersecting bars of triangular section. In one set of parallel bars the base of the triangle is turned towards one side of the plate; in the other set of bars the base of the triangle is turned towards the other side of the plate. The pellet of active material is thus locked in position.

The mode of construction of the grids is not described.  
—E. T.

*Improvements in Materials for Diaphragms for Use in Electrolytic Processes, applicable also for Filtering and other Purposes.* E. T. Parker, Wolverhampton. Eng. Pat. 6605, March 29, 1893.

DIAPHRAGMS, according to this inventor, hitherto used in electrolytic processes, are subject to rapid disintegration, or great variation of electric resistance, and his object is to obviate these objections by the use of fluoride of calcium, or fluoride of aluminium, or "eryolite," in or as diaphragms for use in electrolytic processes, or for filtering or other purposes in chemical operations, where glass and the silicates would be attacked and acted upon; the fluorides being made into the form of wool, after the manner of slag wool, or woven or otherwise incorporated or made into a coherent article used as cloth, or as a lining for perforated vessels.—J. C. R.

*Improvements in Secondary Electric Batteries.* W. Petschel, Berlin, Germany. Eng. Pat. 19,567, October 23, 1893.

IN A U-frame of lead or suitable material is packed a series of strips of thin lead tape. By corrugations or any suitable means these strips can have slight spaces between them in which the liquid can circulate, and which allow of more perfect formation.—E. T.

*Improvements in Disinfecting, and Apparatus for that Purpose.* E. Hermite, C. F. Cooper, and E. J. Patterson, all of London. Eng. Pat. 22,279, November 21, 1893.

See under XVIII. C., page 271.

*New Improved Electro-positive Galvanic Battery Electrodes.* C. A. J. H. Schroeder and H. E. R. Schroeder, London. Eng. Pat. 22,956, November 29, 1893.

INSTEAD of ordinary zinc electrodes are used containing also lead and aluminium in varying proportions, the zinc, however, being always in relatively large, and the aluminium in relatively small proportion. Plates as hard and dense as possible are rolled out, amalgamated, and pressed together to form compound plates.

Others are described containing, in addition to the above ingredients, "any suitable bariun compound," manganese, iron, and mercury. The material is always to be subjected to great pressure to make it very dense and reduce local action.—E. T.

*Improvements in Apparatus for Electrolytically Producing Soda and Chlorine.* H. Lake and Co., London. From the Union Chemical Company, New York, U.S.A. Eng. Pat. 23,436, December 5, 1893.

APPARATUS is described and shown in this invention for decomposing solutions of chloride of sodium for obtaining chlorine as gas, and soda as solution. The containing vessel, of iron, acts as a cathode, and collects the soda; the anode is of gas-retort carbon, to which electrical connection is made by carbon pins driven into holes drilled in the ends of the carbon, and, to avoid corrosion, the carbon pins are impregnated with paraffin under pressure, or the terminal ends may be protected by an envelope or coating of glass or other insulating substance. The anode stands upon an insulated base resting on ganze at the bottom of the tank, with the pieces of carbon vertical, connection to the outside being made by wires, attached to the carbon pins, carried through an insulated pipe; and above them, dipping below the level of the solution, is a dome or bell of stoneware to collect the chlorine gas, which may be connected with an exhauster, and by which fresh brine is introduced.—J. C. R.

*Improved Process and Apparatus for Dissociating Soluble Salts by Electrolysis.* H. Haddon and Co., London. From H. S. Blackmore, Mount Vernon, N.Y., U.S.A. Eng. Pat. 23,913, December 12, 1893.

THE apparatus described in this invention has three chambers or tanks, two containing water, and another, between them, having a lower level and less depth of electrolyte, a solution of sodium chloride, separated from the water chambers, into which the electrodes dip, by porous, or "dialytic" diaphragms, to oppose the passage of any portion of the electrolyte through the diaphragms into the (water) tanks, which is effected by their higher level. It may be understood that the electrodes dip into weaker solutions of electrolyte than that contained in the middle compartment, which would feed them, water only being mentioned as run into these compartments.—J. C. R.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*Mineral Oils on Ships in the Tropics.* C. A. Lobry de Bruyn. Chem. Zeit. 17, 1412.

STEAMSHIP engineers find it necessary to use vegetable or animal oils in place of mineral oils as lubricators in the tropics. At the suggestion of the author, American cylinder oil, a thick mineral oil, has been tried as a lubricator on a number of steamships. The best kinds of

this oil have the consistency of vaseline, and, in fact, according to their constitution, are nothing but unbleached vaseline.

Extensive experiments extending over almost a year, on about 20 men of war in the Dutch East Indies, have proved that a mixture of this cylinder oil with mineral oils forms a splendid lubricator. These mixed oils have a great advantage to the engineer. By varying the proportions for mixing, he can get an oil of any viscosity he may require to suit the different machinery. These mixtures are much cheaper than fatty oils, even when the latter are mixed with mineral oils.—H. M.

*Composition of Rape-Seed Oil.* G. Ponzio. J. prakt. Chem. 48, 187—188.

REIMER and Will (this Journal 1887, 218 and 732) stated that rape-seed oil contains, besides erucic and rapinic acids, a small quantity of behenic acid (melting point 75° C.). The author, by using their method of extraction, obtained from 1 kilo. of rape-seed oil 0.4 gram. of an acid melting at 75° C. He recommends, however, the following process:—1 kilo. of the oil is saponified on the water-bath with 150 grms. of caustic potash in 300 grms. of alcohol, water is added, and the acids precipitated by sulphuric acid. The mixed acids are then dissolved in alcohol and treated with an alcoholic solution of 50 grms. of zinc acetate added in small portions at a time, with constant stirring. A precipitate of zinc salts is formed, which is suspended in water and decomposed with dilute hydrochloric acid on the water-bath. The liberated acids are dissolved in a little warm alcohol, the solution being then cooled in ice. An acid separates which, on being twice recrystallised from alcohol, melts at 75° C. It is arachic, and not behenic acid, as was proved by the melting point and ultimate analysis, as well as by the properties of its amide and esters. It constitutes about 1 per cent. of the acids in the oil.—J. W.

*Cotton Stearin and Maize Oil.* F. Hart. Chem. Zeit. 17, 1522.

THE author has redetermined the physical and chemical constants of these two fats. The results are given in the following tables; those obtained for maize oil are contrasted with Spuller's numbers.

### COTTON STEARIN.

Colour.....	Pale yellow.
Consistency.....	Butter-like.
Specific gravity at 19° C. ....	0.947.
Melting point.....	30° 31° C.
Melting point of the solid fatty acids.....	45.5°—47.5° C.
Free fatty acids.....	Absent.
Helmer value.....	96.3.
Saponification value.....	194.6.
Iodine absorption.....	93.6.
Unsaponifiable matter.....	0.50 per cent.
Thermal reaction with concentrated $\text{SO}_3\text{H}_2$ .....	18° C.
Colour reaction with concentrated $\text{SO}_3\text{H}_2$ .....	Dark purple red.
Colour reaction with Beechi's reagent.....	Dark black.
Colour reaction with Wellmann's reagent.....	Dark green, on addition of ammonia base.

Addition of colza oil, however, prevents the reaction with Beechi's reagent.



## MAIZE OIL.

	Spaller.	Hart.
Colour .....	..	Dark brown.
Consistency .....	..	Liquid.
Specific gravity at 15° C. ....	..	0.9283.
Melting point of solid fatty acids. ....	..	25° C.
Free fatty acids in terms of oleic acid. ....	Absent	0.75 per cent.
Holmer value. ....	94.7	95.7.
Saponifiable value of the oil. ....	188.1	189.2
"    "    "    fatty acids. ....	..	198.4.
Iodine absorption of the fat ..	119.4	119.9
"    "    "    fatty acids. ....	..	125.0.
Unsat. saponifiable matter. ....	1.55 per cent.	1.55 per cent.
Thermal reaction with concentrated $\text{SO}_3\text{H}_2$ . ....	56° C.	60.5° C.
Colour reaction with Bechler's reagent. ....	..	But little blackened.
Colour reaction with Wedmann's reagent. ....	..	Dark green, on addition of ammonia blue.
Colour reaction with concentrated $\text{SO}_3\text{H}_2$ . ....	..	Beautiful dark red.

The unsaponifiable matter in maize oil consists to the greatest part of phytosterol, as evidenced by the melting point and the colour reaction with concentrated sulphuric acid and chloroform.—J. L.

*Effect of Electrical Treatment in Improving the Quality of Oils.* L. A. Levat. Comptes. rend. 1893, 117, 734.

OLIVE oil of inferior quality, of high colour, and acid taste was placed over water at the negative electrode of a voltmeter, which was set in action by a small Siemens machine. The oil became improved in colour, almost sweet in taste, and possessed a pleasant, very agreeable odour. In the case of lubricating oils, containing as much as 5 per cent. of free acid, the acidity was very much diminished.—A. K. M.

*Kapok Oil.* R. Henriques. Chem. Zeit. 17, 1283.

Kapok oil is a vegetable fat expressed from the seeds of *Erisodendron anfractuosum* (or *Bombax pentandrum*), a plant belonging to the *Malvaceae*, and related to the cotton plant (this Journal, 1893, 923). The seeds of the Kapok tree also resemble, in many respects, cotton seeds. The oil recovered from them is brought into the European market by the Dutch and is used in soap making; in its home (East India, West Indies) the oil is also employed for culinary purposes.

A sample of this fat supplied by a Dutch firm furnished, on examination, the following physical and chemical characteristics and constants.

PHYSICAL AND CHEMICAL CHARACTERISTICS AND CONSTANTS.

Colour .....	Greenish-yellow.
Taste and odour .....	Not unpleasant.
Consistency .....	Viscous.
Transparency .....	On keeping, becoming clear with separation of stearin.
Thermal reaction with concentrated $\text{SO}_3\text{H}_2$ . ....	95° (higher than for cotton-seed oil).
Colour reaction with concentrated nitric acid. ....	Similar to that obtained with cotton-seed oil, but appearing more slowly and more greenish.
Specific gravity at 15° C. ....	0.9199.
"    "    "    of fatty acids ..	0.9162.

PHYSICAL AND CHEMICAL CHARACTERISTICS AND CONSTANTS—continued.

Melting point of fatty acids. ....	29° C.
Solidifying point of fatty acids ..	23—24°.
Saponification value of oil .....	181.
"    "    "    fatty acids. ....	191.
Mean molecular weight of fatty acids. ....	293.
Iodine value of oil .....	116.
"    "    "    fatty acids. ....	108.
Holmer value .....	94.9.

—J. L.

*The Oxygen Absorption, Acid and Iodine Numbers of Various Drying Oils.* L. Lettenmayer. Chem. Zeit. 17, 1506.

As drying oils may be counted linseed, lallémantia, hemp seed, nut, poppy seed, sunflower, and madia oil. These all contain varying amounts of oleic, linoleic, linolenic, and isolinolenic acids, which being all unsaturated compounds readily absorb oxygen or iodine, so that their power of taking up the latter becomes a test of their value to painters. Oxygen absorption, however, only takes place when they are spread out in thin layers; in bulk they become rancid, acids of lower carbon-content being formed together with carbonic anhydride and glycerin; and this rancidity also occurs when pigments have been mixed up with them. Experiments were carried out on the drying power of thin films of the following oils which were all from the first pressing of the several seeds:—(1) East Indian and (2) Baltic linseed, (3) crude and (4) bleached nut, (5) poppy seed and (6) hemp seed oils. During the first few days of exposure all except (4) lost in weight, the loss being greatest in the case of (3), less in the others in the following order: (6), (5), (1 and 2). The linseed oils were some time before they began to gain in weight, but finally the absorption was very rapid, and they yielded films covered with reticulations, while those from (5) and (3 and 1) were smooth. For this reason poppy and nut oils are the best for oil painters. (4) gained 0.3 per cent. in weight in one day and 0.4 per cent. in a week, while the others gained 0.9 per cent. in 10—11 days, and the increase continued in the case of (3 and 4), (6), and (5) for 50 days, and after 150 they began to lose again. Lallémantia (*lallémantia ibérica*) is the quickest drying oil of all, then in order come (1), (3), (5), (6), and (1 and 2).

Freshly pressed oils give the highest iodine numbers, and if they are kept in closed vessels filled up to the neck, these do not decrease, age having no influence, but if exposed to the air, the iodine absorptions become lower. Linseed oils vary considerably in their power of absorbing iodine, while the others have a nearly constant value. The acid numbers are liable to considerable alteration when the oils are exposed or when the vessels are frequently opened, but the bleaching process carried out with hydrogen peroxide has no noticeable action on them. Painters should always employ oils of the first pressing, and for this reason it is advisable that artists' colourmen should prepare them themselves.—F. H. L.

*The Analysis of Lubricating Oils containing Blown Rape Oil and Blown Cotton-Seed Oil.* T. B. Stillman. J. Amer. Chem. Soc. 1893, 15, 265.

See under XXIII., page 286.

*Soaps most Useful for Scouring Woollen Goods.* J. Lewkowitsch. J. Soc. Dyers and Colorists, 1894, 42—53.

THE author reviews in the introduction the natural fats used in the production of textile soaps and the properties of the soaps obtained from each individual fat. He considered tallow, palm oil, cotton-seed oil, linseed oil, olive oil, cocoa-nut oil, and palm-nut oil.

*Tallow* requires weak leys for saponification and little salt to throw it out. It yields a white hard soap; too hard, in fact, to be used by itself, inasmuch as it would damage the fibre in scouring or washing. Therefore, tallow is only used admixed with other fats.

*Palm oil soap* is similar to tallow in many respects; it differs, however, in that it is easily saponifiable owing to the large amount of free fatty acids it usually contains. Like tallow, palm oil is mostly used in admixture with other fats.

*Cotton-seed oil* requires very weak leys for saponification and is difficult to salt out. The soap made from it is soft, and has the disagreeable property of possessing a very unpleasant smell, owing, no doubt, to the fact that cotton-seed oil is a semi-drying oil, the fatty acids of which undergo some oxidation process. Besides, colouring matters and resinifying bodies contained in the oil, produce yellow stains in the finished soap. Cotton-seed oil is preferably used for making soft soaps.

*Linsed oil* finds its application in the production of soft soaps; it is only used for inferior kinds of hard soaps.

*Olive oil* is the best possible material for textile soaps. Like tallow it requires but little salt to throw it up; the curd is, however, less hard, resembling a curd made from equal parts of tallow and lard. Owing to its chief property of remaining liquid and fluid, even in very concentrated solutions, olive oil soap is the textile soap "par excellence."

*Cocoa-nut oil*, in contradistinction from the previously mentioned fats, is not saponified easily by weak leys, requiring as it does strong leys. Saponification is so readily effected by the latter, that it suffices to stir the oil well into caustic lye and allow the mixture to stand, when the combination will take place in course of time. Cocoa-nut oil further differs from the foregoing fats in that its soap requires very large quantities of salt for separation, so large, indeed, that it would be altogether disadvantageous to prepare pure cocoa-nut oil soaps by the "salting out process." This soap becoming easily rancid, the oil is only used in admixture with other fats.

*Palm-nut oil* is in most respects similar to cocoa-nut oil. It requires strong leys for saponification and large quantities of salt to "salt it out."

The last two fats form, in conjunction with tallow, an excellent material for the best "curd" soap made for the woollen trade.

*Bone fat, kitchen fat, ship fat, and curriers' grease* are also largely used for making scouring soaps, the quality of which would largely depend, *ceteris paribus*, on the quality of the raw material employed. Thus, bone fat recovered by the benzine process would be a most undesirable ingredient for textile soaps.

A very important raw material for making soaps for the woollen trade is *oleic acid*. Being a free acid it is easily saponifiable and being a stronger acid than carbonic acid, it can be made into soap by simply boiling it up with sodium carbonate. It is a most suitable material for textile soaps and is largely used for that purpose. The "olefine" soaps, as they are called, if prepared carefully will give the same effect as an olive oil soap.

Of the soap stock substitutes, the author condemns "recovered grease" altogether, although it is largely used. The "crude grease" from which the material is distilled is, to begin with, after contaminated with mineral oils, which pass over into the distillate, along with some other hydrocarbons formed by destructive distillation and with certain quantities of unsaponifiable matter (this *Journal*, 1892, 142). The author further deprecates the employment of resin for textile soaps, the resinate being liable to give lime resinate in the milling process, which is very difficult to remove in the ordinary way, thus causing endless trouble in the subsequent dyeing operation.

*Filling materials* should be altogether absent from soaps for the trade. *Sodium silicate*, although extensively used for lower class scouring soaps, will tend to weaken the fibre, perhaps even destroy it by acting mechanically, thus breaking the tender fibres of the wool. For in the washing operation the salt will be precipitated within, and

on the fibre as calcium silicate and silicic acid. All other filling materials are absolutely condemned by the author.

As to the action of soap, its general application for washing and scouring purposes is undoubtedly due to its property of easily penetrating within the fibre, and coming into intimate contact with the fibre, of easily removing the fat by emulsifying it and consequently giving the water a chance of washing it off the fibre. The old view, due to Chevreul, that soap was decomposed by cold water into free caustic alkali and an acid salt, which latter is deposited on the fibre, preventing the dirt from settling down again on it, attributed the scouring, washing, and emulsifying property of soap to the dilute solution of the caustic alkali. This view, however, was an erroneous one, since a very dilute solution of caustic alkali could not replace soap, and for the further reason that a hot and clear soap solution (when no hydrolytic action has taken place to all appearances) cleanses far better than a cold one.

A better explanation, based upon experiment, had been suggested by *Rotundi*, who had shown that soap is split up into two salts, viz., a basic salt, which remained in solution, and into an acid salt, which settled down on cooling. The alkaline salt, at a higher temperature, being able to dissolve the acid salt, a clear solution is obtained directly by dissolving soap in warm water. The detergent property of soap may still be considered as due to the alkaline salt only, whereas the acid salt may simply fulfil the function of depositing itself on to the fibre and preventing the dirt from settling down again on it.

Passing on to the employment of soap in the woollen trade the author considered the three stages at which it was used, viz.: firstly, for scouring the raw wool; secondly, for the scouring of the woven fabric; and thirdly, in the milling and fulling process, the two last operations being considered together as being so nearly related to one another, that the two expressions were used promiscuously, although certainly the machines used for milling and fulling were different.

In answer to the question as to what soap should be used at any of these three stages was: any soap that is neutral, free from alkali, not containing any unsaponified fat, free from resin, mineral oil and other unsaponifiable substances, and lastly, devoid of silica and other filling material.

A soap fulfilling all these conditions was a well-made olive oil soap, the excellent qualities of which were well recognised by the trade. The only objection to its extensive use, if objection it was, was its high price.

In the *scouring of the raw wool* any soap, containing free caustic alkali would act most injuriously on the wool, destroying its surface by pitting the scales and taking away the lustre. This deleterious action of free alkali would rapidly increase with a rise in the temperature of the wash water. Although for lower classes of wool soaps containing sodium carbonate or even a dilute solution of sodium carbonate might be permissible, still, for the very finest wool a neutral soap, preferably a neutral potash soap, should be used. The highly praised "secret powders" were nothing better than frauds, and should, therefore, be guarded against carefully.

The object of *scouring the woven fabric* being to remove the lubricant employed in spinning the wool, the quality of soap would naturally depend on the quality of the goods as having previously determined the quality of the lubricant.

For the finest and best goods, which were spun with olive oil or best "olefine," a neutral soap would be required. For worsteds, which were, as a rule, not milled, potash soaps should be used.

The reason why preference is and should be given to potash soap is found by the author in the fluidity and easy solubility of potash soap, which enabled it to easily penetrate within the fibre, and consequently to do its work quickly, whereupon it could be washed out just as quickly. This explanation was illustrated and supported by the demonstration of several solutions of hard and soft soaps made from exactly the same stock of fat with varying

quantities of water. The solution of soft soap,—1:7.5—was shown to possess far greater fluidity than the solution of hard soap—1:100.

For lower qualities of goods, alkaline soaps were permissible, and, in the opinion of practical men, even necessary. For the lowest qualities of mixed mungo and cotton goods the lowest qualities of soaps were in vogue, their "strong action" being considered indispensable. The author was of opinion that good soaps would be preferable, as the scourer could add himself any alkali he considered necessary.

Granting that an alkaline soap may be required, the author tried to explain its action in the following way:—For lower classes of goods inferior lubricating oils, easily oxidised and readily resinifying, were generally used, and a strong action was required to remove them. It had to be assumed, according to Rotondi's experiments, that soap in solution was decomposed into an acid and alkaline salt. By using free alkali along with the soap, the acid salt, which would be practically useless as a scouring agent, was rendered alkaline again, and converted into a neutral or even alkaline soap. If a neutral soap resulted, it would be split up like a neutral soap, but if it had been converted into an alkaline soap it would act just in the same way as the alkaline salt got from soap. The acid salt would consequently dissolve easily, and what is more important, it would be washed out easily, carrying with it the emulsified lubricant.

In the scouring of *dyed* goods a neutral soap should be used, as the colour might be destroyed or bleed out.

In the *milling* operation the cloth underwent felting and shrinking, and in the opinion of some writers a slightly alkaline reaction of the soap tended to raise the scales of the woollen fibres, thus allowing them to slip more easily into one another, and producing more quickly the desired effect. In the author's opinion, however, soap required for milling the woven fabric should be a good neutral soap, free from any excess of alkali, and free from any unsaponified fat or unsaponifiable matter.

In support of his views was cited the fact that for the finest goods as a rule an absolutely neutral soap was used, some manufacturers even adding ammonium carbonate to the cloth in the stocks so as to neutralise any free alkali.

By means of several analyses of good household soaps and by reference to the quality of the best toilet soaps, the author showed that neutral soaps can be supplied to the trade, if the proper price was paid. He exhibited further several "milling" and "scouring" soaps, and discussed their value as indicated by analysis. A ready qualitative test for alkalinity of soaps was shown in the application of phenolphthalein solution to a freshly cut surface of the soap.

The author strongly advised the woollen manufacturers to prepare their own soap, and pointed out as the most suitable material oleic acid. Although no experienced soap-maker would find any difficulty in making the oleine soap with either caustic alkali or carbonated alkali, the woollen manufacturer had better use the former, as in the latter case boiling over and consequently loss of material might ensue.

In conclusion the author laid stress on the necessity of removing lime from the wash-water, its presence causing waste of soap, precipitation of lime soap on the fibres, and subsequent production of stains on the cloth. Several lime soaps, prepared from completely and partially saponified fat were demonstrated, proving by their clamminess and greasiness the difficulties the removal of such substances from the fibres must entail.

In the lengthy discussion following the reading of the paper, the injurious action of silicated soaps was recognised and absence of resin considered necessary by most speakers. The difference of action of potash and soda soap was ascribed by Mr. Leach to the presence of glycerin in the former, which was said to impart to the wool the "feel" that readily distinguished potash-scoured wool from soda-scoured wool in the hands of an expert. To this opinion, however, the author took objection, as, in his opinion, the glycerin was present in a too extremely dilute solution as to exert any influence. This question, however, he suggested,

should be settled by experimenting with a neutral soda soap with addition of the corresponding quantity of glycerin. Mr. Hey condemned the use of hard soaps as requiring higher temperature and more mechanical action; he further advised the use of some water-soluble lubricant. As the author pointed out, such lubricant was used abroad in the shape of castor-oil soap or of a neutral salt of sulphoricinoleic acid. Finally, the author declared that the removal of mineral oil offered no difficulty, and pointed to some experiments of his own demonstrating that a neutral soap was far better able to emulsify mineral oil than one containing free alkali.—J. L.

*A Study on the Methods of Estimating Glycerol.* E. Suhr. Archiv. f. Hygiene, **14**, 305.

See under XXIII., page 287.

#### PATENTS.

*Process for the Distillation of Substances having a High Boiling Point.* Benno Jaffé and Darmstaedter, Charlottenburg, Berlin. Eng. Pat. 1468, January 23, 1893.

See under I., page 239.

*Improved Manufacture or Production of Soap.* J. Peacock, Croydon, Surrey, and W. B. Colls, Barnes, Surrey. Eng. Pat. 5621, March 15, 1893.

The following composition is stated to be "very beneficial in improving the appearance of the skin of the user." 92 lb. of the dried shavings of curd soap, or like soap, which will allow of its being milled; 1½ oz. of boric acid, made into a saturated solution with water; and about 8 lb. of yolks of eggs are thoroughly milled or incorporated; the mixture is then made into cakes in the ordinary or any suitable way.—C. R. A. W.

*Improved Detergent Compound or Preparation for the Washing or Scouring of Textile Fabrics or other Articles.* W. Brothers, Rawtenstall, Lancashire. Eng. Pat. 6123, March 22, 1893.

Solid ammonium hydrogen carbonate,  $\text{NH}_4\text{HCO}_3$ , is powdered and dissolved in boiling water. Or the ammonium hydrogen carbonate may be previously mixed with carbonates of the fixed alkalis or with granulated or other soap, a suitable proportion being equal quantities of these materials, or 75 per cent. of the one and 25 of the other. These compounds may also be used in the bleaching or dyeing industry, or in any other industry where it is desired to neutralise excess of acid.—C. R. A. W.

*Improvements in Treating Wool Greases and Separating their Constituents.* R. Hutchison, Cowlares, N.B., 6993, April 5, 1893.

The patentee applies the term "lanichol" (abbreviation for lanicholesterine) to the material obtained by the following process, regarding it as different from "lanolin" (or "lanoline"). Impurities of albinous, gummy, or similar character are first separated by boiling nine parts of wool grease with as much resin of soda (made by boiling together rosin and twice its weight of soda crystals) as contains one part of rosin; brine is added, and the whole boiled up with steam, enough salt being present to prevent the whole of the grease from going into solution; or salt may be added during boiling. The undissolved grease boiled up repeatedly with brine to wash out impurities, and finally with water containing alkali in solution; free fatty acids and soaps dissolved in the grease are thus washed out; on standing, "lanichol" rises to the surface, whilst good soap stock is obtained on treating the soapy wash waters with salt.—C. R. A. W.



say 80 decagrams. of the cleansing agent in 20 litres of water: finally the articles are rinsed twice in cold water. Coloured stuffs must not be boiled—C. R. A. W.

*Improvements in, and in Apparatus for, the Manufacture of Margarine and Alimentary Fat.* T. Cordewener and A. de Kunwald, France. Eng. Pat. 24,461, December 19, 1893.

See under XVIII. A., page 270.

### XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

#### (A.)—PIGMENTS, PAINTS.

*Strontium Urinate.* T. Niederländer. Chem. Zeit. 17, 1893, 1507.

This new pigment is prepared in three distinct shades, lemon yellow, dark yellow, and orange. It is very permanent, but it has little covering power, and its chief use therefore is for blending. By prolonged exposure to strong light it becomes duller with a slight greyish tendency. The pale shades are precipitated from solutions of uranium nitrate by strontium hydrate, the former being kept in excess, and while temperature and concentration are without influence on the shade, it is found better to work hot. The darker pigments are precipitated cold from a 10 per cent. uranium solution, a considerable excess of strontium hydrate solution being employed, and the whole boiled for some time. By heating the dark yellow colour to 200°–250° C., it is gradually changed into the orange. Barium compounds may be used, and form better and more permanent pigments.—F. H. L.

*Cadmium Yellow: its Preparation and Properties.* T. Niederländer. Chem. Zeit. 17, 1893, 1505. (Compare Buchner, this Journal, 1887, 665.)

CADMIUM sulphide (*Jaune brillante*) is prepared commercially in shades varying from a bright yellow to a red, but is invariably of the same chemical constitution, containing the theoretical amounts of metal and sulphur. The paler shades are precipitated from 10 per cent. solutions of the sulphate or chloride of cadmium by sulphuretted hydrogen, the alkaline sulphides yielding pigments which from their instability are useless to painters. The stream of gas must be stopped when half the cadmium has been thrown down, and the precipitating vessel must be so arranged as to bring the sulphide into contact with the sulphuretted hydrogen as little as possible. For dark yellows, the whole of the cadmium is thrown down, the liquid being kept warm, and the precipitate constantly stirred up. To prepare cadmium orange, sulphuretted hydrogen is led into a boiling 2 per cent. solution of chloride, to which 5 per cent. of hydrochloric acid (1:3) has been added, the success of the operation depending largely on the efficiency of the agitating apparatus.

The author does not believe in Buchner's theory (*loc. cit.*) that the darker shades of cadmium sulphide are due to molecular condensation, but considers that the variation is a purely physical one, the presence of the free acid in the hot liquid when preparing the orange shade, causing the precipitate to fall more slowly and consequently in coarser particles, and that it is to the finely-divided state in which the pale yellow sulphides exist, that their lack of permanency is due.

As met with in commerce, many of the cadmium pigments are very pure, but, on the other hand, the paler shades from one well-known works contain 21.5 per cent. of zinc, which gives them a greenish cast. Cadmium yellow, dry,

exposed to light bleaches quickly, but when ground in oil will keep its shade for nine months. Some lemon yellows of a beautiful colour are prepared from the hydrate, carbonate, or oxalate of cadmium by decomposition with sulphuretted hydrogen, but these contain but little sulphide and are very unstable.—F. H. L.

*The Importance of the Study of the Chemistry of Oil-painting.* T. Petruschewsky. Chem. Zeit. 17, 1505.

In a paper read before the German Society for the Advancement of Oil-painting held in Munich, the author points out that still very little is known as to the cause of the fading of pigments, and that, as the fame of a painter is largely dependent on the permanency of his pictures, this matter should be thoroughly investigated. He advises that suitable resin varnishes should be employed instead of those containing oil only, that the finished pictures should always be varnished on the back, and that the paintings should not be exposed to direct sunlight.—F. H. L.

*The Properties of Essential Oils, and their Employment in Painting Compared with that of Mineral Oils.* L. Lettenmeyer. Chem. Zeit. 17, 1506.

See under XX., page 273.

#### PATENTS.

*Improvements in the Preparation of Oxide of Zinc Pigments.* A. P. Laurie, London. Eng. Pat. 4870, March 7, 1893.

To improve the quality of paints having zinc oxide as their basis, the inventor grinds them with a mixture of oil and free fatty acid, either produced in the linseed oil by the action of superheated steam, or added thereto during the grinding. For house painting the acids should form 5 per cent. of the varnish, for artists' use about 15 per cent., and either linoleic or oleic acids may be employed.—F. H. L.

*Improvements in the Manufacture of Red Oxide Paints.* M. N. D'Andria, Strerford, Lancaster. Eng. Pat. 5349, March 13, 1893.

THE very high temperature which it is necessary to employ to remove all the sulphuric acid contained in ferrous sulphate has a very deleterious influence on the shade, and the other physical properties of the resulting pigment. To overcome this, the inventor calcines a mixture of about equal parts of ferrous and magnesium sulphates, finding that the decomposition takes place at a lower temperature, that the colour of the oxide is brighter, and that it is formed in a finer state of division. The magnesium sulphate is removed by washing, and the gases evolved during the furnacing may be led into a vitriol chamber as described Eng. Pat. 4343, 1893; this Journal, 1894, 163.—F. H. L.

#### (B.)—RESINS, VARNISHES.

*The Importance of the Study of the Chemistry of Oil-painting.* J. Petruschewsky. Chem. Zeit. 17, 1505.

See under XIII. A., page 262.

#### PATENTS.

*Improvements in Pyroxyline Compounds, Solutions, and Solvents.* L. Paget, New York, U.S.A. Eng. Pat. 22,137, November 18, 1893.

THE inventor has found that by the action of ozone he has been able considerably to increase the number of liquids practically available as solvents of nitrocellulose.

The ozone is prepared by the electrical treatment of oxygen; it must be dry, and, at a temperature not exceeding 120° F., is blown through the oil, which is kept cold. If the latter is viscid, it may preferably be sprayed into a chamber filled with the ozonised atmosphere. Oils and other liquids which are amenable to this treatment may have been previously useless for one of two reasons—first, that they were not solvents of nitrocellulose; and second, that although solvents, they had no drying power. The inventor has tried the following liquids, and found that to the first class belong:—Oil of turpentine, camphor, methyl alcohol, acetone, acetal, ethyl amyl ether, and fusel oil (to a certain extent); while oil of caraway seed gains strongly-marked drying powers. In fact liquid solvents are not necessary in all cases, as by placing the soluble gun-cotton in a chamber and blowing in vapour of camphor, ozone, and an alcohol spray, the pyroxylin is rapidly converted into a solid mass. As an example of an ozonised liquid, the inventor finds the following to be a very good metal lacquer:—Amyl aceto-acetate, 20 galls.; ozonised fusel oil, 5 galls.; wood alcohol, 5 galls.; "benzine," 15 galls.; and pyroxylin, 15 to 20 lb.—F. H. L.

*Notes on Asphaltum.* T. Niederländer. Chem. Zeit. 17, 1893, 1507.

ASPHALTUM itself being rather a treacherous material for painters to use, the author has experimented on the action of various solvents, to see whether its noxious ingredients can be removed. He finds, however, that the residues obtained after treatment with various liquids are useless for artistic purposes, as they do not possess the characteristic lustre (*lasur-tone*) which makes this material so valuable. Alcohol extracts 4·5 per cent., but the solution is useless, as it will not dry. Petroleum ether dissolves 40 per cent., and the extract has a beautiful colour, dries well, and may be used either alone or mixed with linseed oil. The ethereal and acetone extracts are valueless. The alcoholic residue, if melted with one-third of powdered amber, may be used in oil, and possesses as fine a colour as the original substance. On the whole, the author considers that the use of asphaltum should be avoided as much as possible in painting.—F. H. L.

#### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

*The Valuation of Glue-stuffs.* F. Gautier. Zeits. Anal. Chem. 32, 413.

See under XXIII., page 283.

#### XV.—MANURES, Etc.

*Comparative Value of Various Phosphatic Manures.* N. von Dehn. Bied. Centralbl. Agrikul. 1893, 590.

THE author draws the following conclusions from his results:—1. Phosphorite and Thomas slag cannot compete with superphosphate. 2. The effect of phosphorite is directly proportional to the amount of phosphoric acid which it contains. 3. Increased manuring with phosphorites increases the production; in the case of low grade phosphorites, however, the increase is not sufficiently great to render them preferable to high-grade phosphorites. 4. The assimilability of Thomas slag always surpasses that of 25 per cent. phosphorite, but the vegetables produced are tolerably equal, which may perhaps be ascribed to local conditions.—A. R. L.

*The Analytical Determination of Probable Available "Mineral" Plant Food in Soils.* Bernard Dyer. Proc. Chem. Soc. 1894, 134, 36.

See under XXIII., page 288.

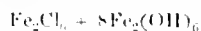
*An Automatic Extractor.* W. D. Horne. J. Amer. Chem. Soc. 1893, 15, 270.

See under XXIII., page 278.

#### XVI.—SUGAR, STARCH, GUM, Etc.

*Purification of Beetroot Juice with Hydrated Basic Ferric Chloride.* Spunt and Schachttrupp. Neue Zeits. Rubenzucker Ind. 31, 108.

By the ordinary method of defecating the juice with lime and subsequent saturation with carbonic acid or sulphurous acid, it is known that many albuminous substances coagulable at a high temperature, as well as other organic non-sugar compounds are either incompletely precipitated or partially redissolved by the excess of lime. The patentees propose to use the hydrated basic ferric chloride—



or other higher basic chloride in the following manner:—The diffusion juice as it comes from the battery of diffusers is mixed at the ordinary temperature with sufficient milk of lime until its alkalinity is 0·08 per cent., and to 60 litres of juice 0·5 litre of the hydrated basic ferric chloride added, together with a further quantity of milk of lime, corresponding with 1 per cent. (on the roots) of caustic lime. The juice is now heated to 80° C., and saturated with carbonic acid to an alkalinity of 0·1 per cent. The boiled juice is filtered, pressed, and treated a second time, at first with 0·25 litre of hydrated ferric chloride per 100 litres of juice, and then with lime (0·25 per cent. caustic lime, on the roots). It is then saturated with carbonic acid or sulphurous acid to an alkalinity of 0·05 per cent., and further treated in the usual manner. Laboratory tests proved that an increase in the purity quotient of 75 to 82 was attained with crude juices, and of 85 to 90 with thin juices. The juice thus purified was free from iron. The patentees intend to extend their process to the purification of molasses.—A. R. L.

*Defecation of Beetroot Juice with Ammonia.* F. Hanuš. Zeits. Zuckerind. Bohm. 1894, 18, 161—166.

THE author's experiments relate to the defecation of the diffusion juice with ammonia, prior to saturation. Ammonia, although a more expensive material than lime, has several advantages over the latter, thus:—Certain organic non-sugar constituents, which comprise cell membrane besides other substances present in the cell contents, are not dissolved; the precipitate which results by the use of ammonia is of a crystalline nature and quickly subsides, so that the liquid can be decanted from it. The last-mentioned precipitate contains organic matters, iron alumina, lime, and much ammonium magnesium phosphate. The juice when neutralised with ammonia may be heated prior to saturation without fear of inversion taking place, and in presence of an excess of ammonia it may be kept at the ordinary temperature for several weeks without undergoing alteration. About 50 cc. of ammonia (ordinary strength) was stirred into 19 litres of diffusion juice. The author directs attention to the fact that the ammonia evolved during the saturation and evaporation of the juice is at present lost, and he suggests its possible application in this process.

—A. R. L.

*Use of Asbestos for the Filtration of Sugar Juices and Syrups.* P. A. Maignen. Bull. de l'Assoc. des chim. 10, 881.

The author recommends asbestos, either in the form of cloth or powder, as a mechanical filtering medium. Its advantages are—(1) that complete clarification is attained; and (2) that sediments of lime, &c., can be removed by treating the asbestos with hydrochloric acid.—A. R. L.

*Augmentation of the Fermentable Capacity of Molasses.* F. Greger. Sac. Indigène, 1893, 42, 651.

See under XVII., page 268.

*Action of Bases on Glucoses.* L. Jessen. Oesterr. Ungar. Zeit., Zuck. Ind. und Landwirths. 661. Neue Zeit. Rübenzuck. Ind. 31, 177, 179: 185—188.

In a previous paper (Neue Zeit. 21, 3) the author showed that the acids produced by boiling invert sugar with lime or caustic potash had an acidity (neutralising power) corresponding with "1.5 mols. of alkali or lime" "calculated on two molecules of the glucose." [Equivalents, not molecules, are possibly here meant.] He now finds that caustic baryta behaves in a precisely analogous manner. A study of the action of alkaline carbonates on dextrose, levulose, and invert sugar has in the main confirmed Herzfeld's observations (Zeit. Ver. Rübenzuck. Ind. 1890, 263), and the evolution of carbonic acid gas which the last-named chemist has observed is to be ascribed to the acids resulting from the glucoses. When 0.5 grm. of either dextrose, levulose, or invert sugar is boiled with 1 grm. of pure lime (free from carbonate) and 100 grms. of water, the amounts of carbonic acid formed are found to be respectively equal to 0.0016 grm. = 0.32 per cent.; 0.016 grm. = 3.2 per cent.; and 0.0078 grm. = 1.56 per cent.

The remaining portion of the paper deals with the behaviour of the decomposition products of invert sugar by alkalis on titration, in presence of various indicators.

—A. R. L.

*Phoma Beta, the Beetroot-destroying Organism.* Hollrung. Neue Zeits. Rübenz. Ind. 1893, 31, 165.

At the meeting of the "Zweigvereines für Rübenzucker Industrie" at Halle, the author communicated the following:—Frank has pronounced the disease produced by this organism a new one, which is not the case, for the organism was first described in 1846; Kühn studied it in 1852. The evil occasioned by the organism in France during 1851 is computed at 400,000 ctrs. of sugar. The disease is generally known as the *core-rot*, since it is characterised by the rotting, blackening, and withering of the inner rudimentary buds. The organism makes its appearance in the formation of small cavities in the buds, which in time open,

and spores result which then develop. The causes are at present unknown. Frank states that manuring with lime promotes the growth of the organism; this is, however, not so, as it has been observed in beetroot plantations which were not manured with lime. The author believes the cause of the appearance of this organism to be the stoppage in growth of the beetroots; thus he observed a stoppage in growth during the drought, which favoured the development of the disease; the organism was never found in moist localities.—A. R. L.

*Estimation of the Quantity of Juice contained in Beet-roots.* A. Vivien. Bull. de l'Assoc. des Chim. 10, 833.

The above estimation is made by the following three processes:—1. The direct estimation of the sugar in the roots, and of the same in the juice. 2. The estimation of the water in the roots and in the juice. 3. The extraction of a known weight of roots and the determination of the weight of in-soluble substances. The third method is the most accurate:—100 grms. of raspings are extracted with water at first at a low temperature, and afterwards with the same solvent at 80° C.; the extracts are decanted on to a tared filter, and the weight of insoluble substance directly ascertained.—A. R. L.

*Reports on Modified Starches prepared by Siemens and Halske, Berlin.* Bull. Soc. Ind. Mulhouse, October—November 1893, 363.

M. SCHAEFFER reports that these modified starches are prepared principally by the instrumentality of ozone generated by electricity acting on the ordinary potato-starch (fecula) of commerce. The brands named "Soluble Starch" and "Refined Starch" are perfectly white and have a greater thickening power than potato-starch of the best quality. Thickenings made with them undergo no change after keeping five days, whilst those of ordinary starch become thin after lying 24 hours. They would probably prove of value in the finishing of calicoes.

M. Schenrer reports that of the various brands the "Raw Gum," "Universal Gum No. 1," and "Universal Gum No. 2" are the ones most likely to be useful in printing and finishing, being almost entirely soluble in boiling water, whilst the Universal Gums dissolve even in cold water. They have less colour than roasted wheat-starch and locoeome (British gums), which they to some extent resemble.

Used for thickening colours, 3 to 4 lb. per gallon is sufficient. The Universal Gums Nos. 1 and 2 do not stiffen the cloth even with acetate of chrome mordants, and the shades got are as deep as and clearer than with roasted starch. The "Raw Gum" gives colours as clear as the preceding, but deeper in tone, and it stiffens the cloth a little.

The following table shows the physical and chemical properties of the six samples examined:—

Brand.	Appearance.	Coloration with Iodine.	Action on Fehling Solution.	Action of Cold Water.	Action of Boiling Water.	Appearance of Solution at once.	Appearance of Solution after 24 Hours.	Appearance of Solution after 96 Hours.	Colour of Solution.	Remarks.
Raw starch	Like inferior potato-starch, greyish.	Pure blue	None	Insoluble	Very thick and starchy with 20 per cent.	Greyish, dirty, and slightly viscous.	Loses some of its thickness.	Loses some of its thickness.	..	This is the raw material of all the brands.
Refined starch.	Like very fine potato-starch.	Pure blue	None	Insoluble	Very thick and starchy, viscous (if hot).	Very white, very viscous.	Without change.	Without change.	..	Slight smell of hypochlorite.
Soluble starch.	Like ordinary potato-starch, slightly amber-tinted.	Pure blue	None	Insoluble	Thick and starchy, less viscous.	Consistency resembling that of wheat-starch.	Thickens somewhat.	Without change.	Very slightly amber-coloured, almost white.	..



Brand.	Appearance.	Coloration with Iodine.	Action on Fehling's Solution.	Action of Cold Water.	Action of Boiling Water.	Appearance of Solution at once.	Appearance of Solution after 24 Hours.	Appearance of Solution after 36 Hours.	Odor of Solution.	Remarks.
Raw gum	Like British gum, very clear.	Blue, with a touch of violet.	Very strongly reducing.	Turbid solution with 2 per cent. substance.	Dissolves completely with 20 per cent. substance.	Gummy and transparent with 20 per cent. substance.	Slight insoluble deposit.	Deposit increased.	As above.	Does not ferment on addition of yeast.
Universal Gum No. 1.	Like gummiine, transparent and yellowish.	Violet	Strongly reducing.	Solution almost complete.	As above	Gummy and thin with 20 per cent. substance.	Slight deposit.	Hardly changed.	Like egg-white solution.	Contains a trace of chloride of calcium. Does not ferment.
Universal Gum No. 2.	As the last	Violet	As above	Solution less complete than the above.	Solution somewhat less transparent than the above.	Like the above.	Slight greyish deposit.	Hardly changed.	As above.	As above.

—W. E. K.

## PATENTS.

*Improvements relating to the Manufacture of Soluble Starch.* H. H. Lake, London. From W. Angele, Berlin, Germany. Eng. Pat. 5617, March 15, 1893.

SOLUBLE starch has heretofore been made by Lintner's method, which consists in subjecting it to the action of 7·5 per cent. hydrochloric acid at the ordinary temperature for several days, or to that of 15 per cent. sulphuric acid at a moderate temperature. The process which the patentee claims is exemplified as follows:—Dry starch is intimately mixed with sufficient dilute hydrochloric acid so that the mixture contains about 0·5—1 part of anhydrous acid to 100 parts of dry starch. This quantity of acid is diluted with as much water as is required to make a stiff paste of such consistency that it may be stirred. For example, 1,000 kilos. of dry starch are mixed with 1,000 litres of water, to which mixture 20—40 kilos. of 25 per cent. hydrochloric acid is added. The mixture is placed in a drying room on hurdles, where it is allowed to dry at a temperature of about 20°—40° C. The drying is continued until a sample completely dissolves, without gelatinisation, in hot water; the time required for this is generally about 18 hours. The required time depends in a certain degree on the perfection of the drying plant, and may be considerably reduced by first extracting a portion of the water by the aid of a centrifugal machine. In the latter case, however, the mixture must originally contain 5—10 kilos. of acid for every 100 kilos. of starch, so that, after spinning, it may contain the requisite quantity (as above) of acid. The action of the centrifugal machine causes a very intimate penetration of the acid into the starch. The acid is then got rid of by washing with cold water and with dilute solutions of alkaline carbonates, after which the product is spun in a centrifugal machine and dried in the usual manner. The process may also be carried out with hydrochloric, nitric, sulphuric, phosphoric, oxalic, lactic, acetic, and other liquid or volatile acids.—A. R. L.

*Improvements in the Continuous Refining or Bleaching of Sugar, and in Machines employed for this Purpose.* L. E. A. Prangey. Eng. Pat. 7695, April 15, 1893.

THE improvements relate to machinery for carrying out the process described in another specification (see this Journal, 1893, 702). Several layers of massecuite are deposited one above another, and successively subjected to pressure. The combination of an endless apron conveyor with a massecuite reservoir containing partitions at different levels, dividing it into hoppers which debouch at different levels above the apron, or (in the case of treating sugar in powder which is more or less dry) with a series of V-shaped hollow transverse partitions at different levels within the reservoir.

Furthermore, of pressure rollers in front of the hoppers, supporting rollers below the apron and under the pressure rollers.—A. R. L.

*Improvements in the Manufacture of Sugar.* H. H. Lake. From P. H. Van der Weyde and O. Lugo, New York, U.S.A. Eng. Pat. 136, January 2, 1894.

THE invention consists in electrolysing a sugar solution contained in a shallow tank, and heated to about 200° F. by electrodes of aluminium and carbon connected in the usual way to the poles of an electric generator, preferably a constant current dynamo machine. The E.M.F. need not be more than 4—5 volts, and the quantity is proportional to the surface of the electrodes in contact with the solution. The anode is thus attacked, producing "nascent aluminium oxyhydrates," which are said to effect the defecation and decolorising of the liquor. Although the "oxyhydrates of aluminium" are insoluble in sugar solution, they combine with certain impurities in crude sugar liquor and pass into solution as soluble compounds. To completely precipitate the latter, therefore, the patentees add a soluble phosphate—preferably in the form of bone ash, bone black, or spent bone black treated with sulphuric acid.—A. R. L.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*Lac-lined Casks.* Bohmischer Bierbrauer, 1894, No. 2.

A BREWER attributes disastrous results obtained in a brewery to the employment of lac-lined casks. The lac had become porous, and teemed with wild yeasts and other organisms. He strongly advises the employment of pitch rather than lac.—A. K. M.

*The Production of Saccharose (Cane Sugar) during the Germination of Barley.* L. Lindet. Comptes rend. 1893, 117, 668—670.

THE author has examined samples of barley during various stages of germination, and finds that the cane sugar gradually increases whilst the starch diminishes, and concludes that the former is produced at the expense of the latter.—A. K. M.

4. *Study of the Use of Apple U.S. for Cider-making.*  
A. D. Comptes rend. 1893, 117, 735—767.

THE author has studied the variety *Amre* of Sarville during the various phases of its development from the blossom to the decomposition of the fruit. He divides the investigation over four periods, thus:—(1) Period of forma-

tion and development, June to September; (2) Period of maturity on the tree, October to mid-November; (3) Period of ripening in store house, November to February; (4) Further period, March to July. The results of his analyses are tabulated as follows:—

	Minimum.	Maximum.	Variation.
Sap.....	746 grs. s. fruit rott n. June, end of experiments.	8221 grms. July, end of experiments.	Varies but slightly.
Diastase.....	1504 grms. June, end of experiments.	1489 grms. July, end of experiments.	The variation in the sugars is very appreciable, that of the saccharose being greatest.
Saccharose.....	10742 grms. November, during storage period.	11360 grms. during storage period.	" "
Tannin.....	0.139 grm. fruit rotten, end of experiments.	1570 grms. November.....	" "
Pectin, albumenoids, &c.....	0.141 grm. November.....	1560 grms. July, end of experiments.	This ill-defined principle is very variable.
Pectin, albumenoids, &c.....	0.139 grm. August.....	2320 grms. July, end of experiments.	Very variable.
Tartronicity as malic acid.....	0.085 grm. for most months.....	0.151 grm. fruit rotten June.....	Slightly variable.
Vegetable tissue.....	5.050 grms. December.....	13.07 grms. June, end of experiments.	Rather variable.
Tadash.....	0.451 grm. January.....	1.287 grms. June, end of experiments.	Very variable.

—A. K. M.

*Influence of Carbon Dioxide on the Growth and Life of Fermentation-Organisms.* Jahresb. d. Königl. Lebranst. f. Obst- u. Weinbau, zu Gießenheim.

It is found that both *Saccharomyces ellipsoides* and *S. apiculatus* are considerably retarded in their growth by carbon dioxide, and the latter species to a greater degree than the true wine yeast (*S. ellipsoides*). The mould-fungi *Penicillium glaucum*, *Mucor racemosus*, and *Dematium pullulans*, on the other hand, are not merely retarded in their growth, but soon perish in an atmosphere of carbonic acid gas, whilst *Mycoderma*, the acetic ferment and *Torula* species can, under the same conditions, live for months, although unable to bud.

By means of a rapid fermentation, therefore, wine can be protected from the action of the moulds and *S. apiculatus*, but in order to protect it from *Mycoderma*, *Bacterium aceti*, and *Torula* it is necessary that as much as possible of its carbonic acid be retained in the wine during the whole period of its storage. It is not, however, advisable to employ artificial carbon dioxide to increase the stability of wine, as the latter then acquires a peculiar taste, and, on coming into contact with the air, also an objectionable brown colour.

—A. K. M.

*The Action of Calcium Sulphite and of Potassium Bisulphite on Alcoholic Fermentation.* F. Ravizza. Le Staz. Sperim. Agr. ital. 24, 1893, 593.

From experiments made with artificial must, prepared by adding invert sugar solution—inverted by means of tartaric

acid—to the juice of white grapes, the author finds that small quantities of calcium sulphite are without effect on the fermentation, whilst quantities sufficiently large to produce a retarding effect, would prejudice the character of the wine. Potassium bisulphite has a more strongly retarding, and hence regulative effect.—J. G. W.

*Studies on Yeast.* II. Elien. Cent. für Bakt. und Parasit. 1893, 53; Zeits. für Spiritusind. 17, 1894, 11.

THE amount of carbon dioxide evolved when yeast acts on sugar solutions under certain conditions varies with the variety of yeast used, and may be made use of to distinguish these varieties. The author employs the determination of the amount produced of this gas to calculate out two constants for the yeast, viz., the fermentative power and the fermentative energy; the fermentative power being defined as the total amount of sugar a given amount of yeast is able to ferment, while the fermentative energy is the amount fermented by a given quantity of yeast in a given time.

The carbon dioxide was determined by the volumetric method, taking, however, the precaution to expel the gas contained in the fermented liquid at the close of the experiment, as the author finds, contrary to Hayduck, that the amount retained in solution at any time while fermentation is proceeding may vary.

The following are some of the results obtained by the author:—

Variety of Yeast.	Quantity of Yeast.	Time of Fermentation.	Sugar.	Water.	Temperature.	CO <sub>2</sub> evolved.
	Grms.	Hours.	Grms.	Ce.	° C.	Ce.
Bottom yeast I.....	2.5	2	10	100	35	328
" " I.....	2.5	2	10	100	30	330
" " II.....	5.0	2	10	100	30	660
Top yeast in pure.....	5.0	2	10	100	18	274

The presence of phosphates largely increases the fermentative energy, but different yeasts are influenced to a different extent. The fermentative energies of different varieties of yeast show still more variation when determined at varying temperatures.

The fermentative energy of a yeast is no guide to its value for baking purposes, and the author finds that, contrary to the opinion of many, the presence of bacteria is not essential to a good baking yeast. He emphasises the importance of the fact that some yeasts have a very great

fermentative energy at comparatively high temperatures, and that among these are varieties which are undesirable for brewing purposes, but which may be most suitable for the bakery (this Journal, 1888, 636).—J. G. W.

*The Influence of Certain Chemical Conditions on the Activity of Brewers' Yeast.* J. Effront. Comptes rend. 1893, 117, 559—561.

THE experiments described lead the author to the following conclusions:—1. That when yeast is cultivated in wort containing per litre, 200—300 mgrms. of ammonium fluoride, its power of reproduction is sensibly weakened, but that the different species are affected in varying degrees. 2. That a wort containing 200—300 mgrms. of fluoride per litre, can be employed for the cultivation of all beer-yeasts without distinction of race, and with the same success, if they have previously become accustomed to the presence of fluoride. 3. That yeasts which have been cultivated in the presence of gradually increasing quantities of fluoride acquire a greatly increased fermentative activity—ten times greater than before treatment—and exhibit properties which physiologists have hitherto regarded as belonging exclusively to certain races.—A. K. M.

*The Kilning of Malt in Relation to the Colour of the Malt-Germ.* Cerny. Oesterr. Brauer- und Hopfen-Zeitung, 1894, No. 1.

EXPERIMENTS conducted by the author show that the germs of an undergrown and of a normally grown malt were darker than in the case of an overgrown malt, although the conditions of kilning were identical. The colours of the worts, however, were the reverse, the malt with the long plumule giving the darker wort. Consequently, to produce equal results as regards colour, the undergrown malts require more kilning than overgrown malts. In explanation of the above results, the author further expresses the opinion that the colouring matter of malt is formed in the radicle, and on further growth becomes subsequently translocated to the plumule.—A. K. M.

*Some Products of Starch Transformation.* W. L. Hiepe. The Country Brewers' Gazette, 1893, No. 431; 1894, Nos. 433, 434, 435.

*Experiments with Isomaltose.*—Isomaltose was prepared by Lüntner's method, and its fermentability was tested with various yeasts and with the following results:—

Type of Yeast.	Percentage of Sugar Fermented.
S. Cerevisiae, Saaz (Jørgensen 255) .....	24.1
" Froberg (Jørgensen 60) .....	57.1
" High fermentation type .....	54.3
S. Pastorianus I. ....	56.3
" II. ....	47.3
" III. ....	43.1
S. Ellipsoidens I. ....	43.0
" II. ....	58.3
S. Exiguus .....	27.2
S. Apiculatus .....	None.

The exact conditions as regards time and temperature are not stated.

*Examination of Commercial Amyloins.*—The author has examined the two products known as A and B, and his results lead him to the following conclusions:—(1.) The commercial amyloins are partly fermentable by nearly all species of yeast. (2.) Diastase produces an increase of

reducing power and decrease of rotatory power, together with an increase of solids. (3.) After the action of diastase, or by diastase and yeast together, they are practically completely fermentable. (4.) The phenyl hydrazine test shows the presence of glucose, maltose, and isomaltose. (5.) The fractional precipitation gives no homogeneous precipitates between dextrin and isomaltose. (6.) They consist, therefore, of a mixture of glucose, maltose, isomaltose, and dextrin; the latter being entirely hydrated by diastase and having a distinct reducing power.

The results of analysis of amyloin A indicated the following composition:—

	Per Cent.
Dextrose .....	2.85
Maltose .....	
Isomaltose .....	
Dextrin .....	36.44
Other matters .....	2.11
	100.00

The author has conducted a large number of experiments on the fermentability of the various constituents of the "amyloins" by different species of yeast. The following results taken from an elaborate table refer to the percentage of isomaltose fermented by the different yeasts:

	Per Cent.
Sach. Cerevisiae (Jørgensen 120) .....	53.5
" " " 60 .....	49.9
" " " 84 .....	54.0
" " " 255 .....	67.0
" " " 304 .....	45.8
" " (High fermentation type) .....	51.5
" Pastorianus I. ....	57.9
" " II. ....	45.5
" " III. ....	33.9
" ellipsoidens I. ....	31.4
" " II. ....	49.2
" exiguus .....	8.7
" apiculatus .....	3.2

The figures representing the dextrin and which are given in the complete table are remarkably constant and indicate that this carbohydrate is absolutely unfermentable and that the wild yeasts possess no diastatic action.

The "amyloin A" has also been submitted to an elaborate process of fractional precipitation by alcohol and the author finds it to contain dextrose, maltose, isomaltose and dextrin, whilst no substance having the properties of a malto-dextrin could be detected.—A. K. M.

*An Apparatus for Pasteurising Beer.* E. Windeck. Petit journal du Brasseur, 1893, No. 13.

THIS apparatus consists of a cylinder capable of withstanding a pressure of ten atmospheres, and containing a coil the ends of which pass through the ends of the cylinder. The coil can be rotated at the rate of about 30 revolutions in the minute thereby bringing the beer into a moderate degree of motion. Hot water is first passed through the coil so as to raise the temperature of the beer as quickly as possible to the desired pasteurising temperature; this is followed by spring water to effect a rapid and uniform cooling and finally by cold water from the ice-machine in order to attain a really low temperature. Beer treated in this manner does not lose its carbonic acid gas and does not acquire the pasteurising taste.—A. K. M.

*The Evolution of Carbon Dioxide and Hydrogen Sulphide in Boiling Beer, Hopped and Unhopped Wort and Hops.* Ehon. Rec. des Trav. Chim. des Pays-Bas, 1893, 12, 255.

THE author has found that small quantities of carbon dioxide and hydrogen sulphide are evolved when beer and hopped or unhopped wort are boiled. The evolution of a volatile sulphur compound on boiling hops with water had been previously observed by Prior. The author has made experiments to determine the effect of these decompositions on the accuracy of determinations of the carbonic acid in

beer. He obtained beer free from carbonic acid by heating it to 39° under diminished pressure and passing a current of air through it for two hours. On subsequently boiling the beer for various lengths of time, it was found that the evolution of carbonic acid was greatest at the beginning of the boiling process, but the amount is not sufficient to seriously affect the determination of the carbonic acid in beer, if the boiling be not carried on too long.

Experiments with wort gave similar results. 200 cc. of a wort containing 11.5 per cent. extract yielded 5.3 mgrs. of carbonic acid when boiled for an hour and a half.—A. K. M.

#### *Production of Coconut- and Rice Beer.* A. Calmette. La Bère, No. 1, 1894.

THE author discusses the manufacture of beer in hot countries and treats especially of the conditions prevailing in Java, Fouquin, and Saigon. After describing the primitive methods adopted and the unsatisfactory results obtained, he points out the difficulty of obtaining worts containing a sufficient quantity of saccharine matter, the employment of barley-malt being out of the question owing to its high price. On the other hand, the employment of koji or Chinese yeast for saccharifying the rice is unsatisfactory, owing to the difficulty with which the wort is clarified and on account of the musty taste which the product acquires.

The author finds that a good beer may be obtained from coconut milk which contains 9 to 13 per cent. of fermentable sugar. He adds cane-sugar, boils for an hour and a half with hops (80 grms. to the hectolitre), filters and ferments with pale ale yeast. A beer of excellent quality is obtained, containing 3.4 per cent. alcohol and 3.1 per cent. extract.—A. K. M.

#### *Augmentation of the Fermentable Capacity of Molasses.* F. Greger. *Suer. Indigène*, 1893, 42, 651—652.

THIS process has been patented in Germany and France. Maereker and Meale have recognised that molasses which ferment with difficulty contain volatile acids which exert a toxic action on the yeast, and they proposed to boil the molasses for a certain time with an excess of sulphuric acid, in order to expel these volatile organic acids. Czeczetha has constructed an apparatus for this purpose. The patentees' experiments show, however, that it is necessary to heat the molasses with a known excess of sulphuric acid at 60°—75° C., and subsequently to filter the thus sterilised liquid. By this filtration, which is carried out with either the concentrated or diluted molasses, the substances harmful to the yeast are eliminated, and the process secures the following advantages:—A better fermentation, purer alcohol, increased yield, and a great economy in yeast. The yield of alcohol is increased when molasses, which ferment readily by the ordinary mode of procedure, are treated by this process.—A. R. L.

#### *Estimation of Succinic Acid formed in Fermentations.* A. Rau. *Archiv. f. Hygiene*, 14, 225.

See under XXIII., page 237.

#### *The Determination of Malic Acid in Wines, &c.* C. Micko. *Zeits. d. Allgemeinen österr. Apotheker.* Ver. 30, 289.

See under XXIII., page 286.

#### *The Fermentation of Dextrose, Rhamnose, and Mannitol by Levulactic Ferment.* George Tate. *Proc. Chem. Soc.* 1893 [128], 203.

In studying the micro-organisms that attack ripe pears, the author has had occasion to isolate an organism that under

aerobic conditions brings about levulactic fermentations of the hexoses, dextrose, mannose and galactose and also of mannitol, but an inactive lactic fermentation of the methyl-pentose rhamnose (isodulcitol).

The products of the fermentation of dextrose, mannitol and rhamnose have been quantitatively determined: 9 molecules of dextrose are found to yield two molecules of alcohol, one of succinic acid, seven to eight of levulactic acid, the smaller molecular proportions of formic and acetic acids.

Mannitol yields the same products, but in different proportions, there being a greater yield of alcohol and slightly greater yield of levulactic acid.

Neither alcohol nor formic acid was found among the products of the fermentation of rhamnose; nine molecules of this sugar afforded approximately five molecules of acetic acid and four molecules of optically inactive lactic acid.

The action of the organism as a levulactic ferment of dextrose, was found to be unchanged after cultivating the parent cells in fluids containing rhamnose, inactive acid being formed.

The organism which is referred to as a levulactic ferment from its action upon the typical sugar dextrose belongs to the class of asco-bacteria, and can develop so as to produce two forms of growth, widely differing in macroscopic appearance—one in which rods and cocci predominate, a second in which the organism propagates as an asco-bacterium.

### PATENTS.

#### *An Improvement relating to the Process of Malting Grain.* A. Blake, Newport, Mon. Eng. Pat. 223, January 5, 1893.

LIME water, or lime and water, is used instead of ordinary water for steeping and sprinkling purposes.—J. G. W.

#### *Improvements in the Manufacture of Yeast.* F. M. Polsky, Paris. Eng. Pat. 1887, January 27, 1893.

RICE flour is mixed with double its weight of water at 70°—75° C., containing 6 grms. of calcium acid phosphate per kilo. of rice, and allowed to stand. Green barley malt, or a mixture of green barley and wheat malts, is then added, and the mixture again allowed to stand. Water at a temperature of 90°—95° C. is next added, until the temperature of the mixture reaches 62°—65°, and the resulting liquid is transferred to a vessel divided into several compartments. After cooling to not lower than 30°—32° C., yeast is now added, and subsequently and successively solutions of tartaric acid and potassium bicarbonate in several portions. Another lot of wort is then added, and finally ammonium carbonate or chloride. Fermentation is allowed to continue for six or eight hours, and the yeast removed. It is claimed that conducting the fermentation in a vessel divided into several compartments allows a large quantity of material to be dealt with, but yields better results than fermenting a large bulk at once.

—J. G. W.

#### *Improvements in the Treatment of Rice or other Grain for the Production therefrom of a Material suitable for Use in Brewing, or for other Purposes.* F. E. V. Baines, London. Eng. Pat. 4981, March 7, 1893.

RICE or other grain is digested with water containing lactic or sulphuric acid for half an hour at a temperature equivalent to 50 lb. steam pressure, in the proportions of one part of grain to seven parts of water containing 0.5 per cent. of lactic acid or 0.05 per cent. of strong sulphuric acid (sp. gr. 1.840). The digestion is stopped as soon as the product gives a reddish-brown colouration with iodine. The resulting solution is neutralised with milk of lime, filtered, concentrated, and applied to the surface of a steam-heated cylinder, upon which it is kept in a layer of equal thickness by means of cooled rollers. The dried product is then scraped off by means of spring steel scrapers. It is claimed that complete conversion of the

starch of the grain into dextrin is effected by this method and that the product can be reduced to powder, and will remain dry under ordinary atmospheric conditions.

—J. G. W.

*Improvements relating to the Clarification of Beer.* H. H. Lake, London. From K. Müller, Kehl, Baden. Eng. Pat. 5870, March 18, 1893.

Thin strips of aluminium of any convenient size are placed in the store casks, instead of chips of wood, to keep the sediment at the bottom.—J. G. W.

*Improvements in Apparatus for Cooling and Evaporating Liquids such as are used in Breweries and Distilleries, and for such like Purposes.* H. J. Worssam, London. Eng. Pat. 6316, March 24, 1893.

The apparatus described in this specification only differs from the vertical refrigerator in general use in breweries, in that there are two supply standards at each end, the pair on one side of the refrigerator being connected by the usual thin flattened tubes, as is the pair on the other side. The two pairs of standards and their attached tubes can be taken apart for cleansing purposes, and are so arranged that their tubes occupy alternate positions, so that when placed together they form a single, continuous, vertical series of tubes.—J. G. W.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY AND WATER PURIFICATION, DISINFECTANTS.

### (A).—CHEMISTRY OF FOODS.

*The Temperature of Bread during Baking.* A. Girard. Comptes rend. 1893, 117, 584.

The author states that 101° C. is the normal temperature of bread during baking and not merely the temperature sometimes attained, as was inadvertently stated in a recent note. (See Balland, this Journal, 1894, 55.)—A. K. M.

*The Influence of Mineral Antiseptics on the Lactic Fermentation.* A. Chassevant and C. Richet. Comptes rend. 1893, 117, 673–675.

In this paper the authors distinguish between the amount of an antiseptic required to check the multiplication of the ferment—to which action they apply the term *antigenetic*—and that required to arrest its functional activity—*antibiotic*. They have experimented with the salts of a number of metals, and their results are given in the following table:—

	Antigenetic.	Antibiotic.	Ratio, taking the First Column of Figures as Unity.
Magnesium .....	0.5	1.5	3
Lithium .....	0.25	0.5	2
Calcium .....	0.15	0.4	2.5
Strontium .....	0.125	0.25	2

	Antigenetic.	Antibiotic.	Ratio, taking the First Column of Figures as Unity.
Barium .....	0.125	0.5	2
Aluminium .....	0.025	0.05	1.4
Manganese .....	0.004	0.005	1.3
Iron .....	0.101	0.005	1.2
Lead .....	0.006	0.004	1.7
Zinc .....	0.0025	0.0035	1.4
Copper .....	0.0015	0.0015	1
Calcium .....	0.0085	0.0021	2.5
Platinum .....	0.00025	0.00075	3
Mercury .....	0.00185	0.00185	1
Nickel .....	0.000125	0.000200	1.6
Gold .....	0.000080	0.000125	2
Cobalt .....	0.00015	0.00025	1

—A. K. M.

*Milk, Skim Milk, and Whey: a Study of their Comparative Composition and Specific Gravity.* C. B. Cochran. J. Amer. Chem. Soc. 1893, 15, 247–251.

RICHMOND calculates that each gm. of albuminoids in 100 cc. of milk raises the sp. gr. 2.57 (*sic*), or that the density of albuminoid in milk is 1.346. Dupré shows that 1 per cent. of casein raises the specific gravity of milk 2.55, or that the density of casein in solution is 1.31; Hehner gives 1.3106 for the last-mentioned value.

The author has determined the total solids, fat, solids not fat, and specific gravity of a number of samples of skim milk, and has coagulated each sample with acetic acid or rennet at the temperature of a water bath. The total solids and specific gravity of the clear whey were ascertained and the difference between the total solids of the whey and the solids not fat of the skim milk was taken to represent the casein removed. The difference between the specific gravity of the skim milk and the whey, divided by the percentage of casein removed, represents the loss of specific gravity due to removal of 1 per cent. of casein. "The density of the casein in solution is found by the formula  $D-D^1 = \frac{1-x}{100x}$ ;  $D$  = specific gravity of whey;  $D^1$  = specific gravity of skim milk, minus all fat;  $x$  = effect of 1 per cent. of curd on density."

A table is given which embodies the results obtained by this method; the average calculated from them shows that each percentage of casein removed lowers the specific gravity of the solution 2.72, and that the density of the casein in solution is 1.376.

The solids coagulated by rennet and freed from fat were found to contain 1.08 per cent. of ash. When coagulation is properly performed under similar conditions, the specific gravity and total solids of whey of pure milk will vary within narrow limits. The specific gravity averages 1.027 and does not exceed 1.0285; the total solids vary between 6.5 and 6.9 per cent. If the whey be reheated, a further coagulation will take place, and the specific gravity be deduced to 1.028–1.0266. The specific gravity of the whey from a milk which had been allowed to decompose for 20 days was 1.0255. If the number representing the specific gravity of the whey above 1.000 be divided by 4.25, the quotient approximately represents the percentage of total solids in the whey.

The composition of whey does not materially differ whether it be derived from the original milk, the cream, or skim milk. Thus a knowledge of its composition may be

of value in determining a alteration, especially when the milk is in a churned or sour condition. The following table

serves to show the variation of the composition of the whey in samples of whole, skimmed, and watered milk :—

No.	Specific Gravity.	Total Solids.	Solids, not Fat.	Specific Gravity of Whey.	Total Solids of Whey.	
1	1.0307	11.32	8.78	1.0280	..	A partially creamed sample of milk.
2	1.0315	12.05	8.55	1.0270	..	Poor milk.
3	1.0273	10.99	7.50	1.0230	..	Watered milk.
4	1.0294	12.05	6.15	1.0213	..	Watered cream.
5	1.0.18	11.68	9.49	1.0255	5.89	Whey separated after milk had stood three weeks.
6	1.0310	12.05	8.45	1.0280	6.53	
7	1.0312	12.45	8.55	1.0270	6.35	
8	1.0320	12.90	8.80	1.0275	6.47	
9	1.0320	13.22	9.02	1.0273	6.42	
10	1.0324	12.98	8.83	1.0283	6.66	
11	1.0320	12.49	8.90	1.0270	6.35	
12	1.0278	8.81	8.71	1.0234	5.50	Watered skim milk.

—A. G. B.

*The Detection of Eosin in Coloured Tomatoes.* K. F. McElroy and W. D. Bigelow. J. Amer. Chem. Soc. 1893, 4, 191.

See under XXIII., page 284.

#### PATENTS.

*Improvements in the Preparation of Extracts of Tea and Coffee and in Utilising a By-product of the Process.* E. Soustalt, Churchfields. Eng. Pat. 6596, March 29, 1893.

THE inventor claims as chief improvement the removal of the tannin from tea or coffee extracts by addition of milk, and separates the insoluble tannin compound as a compact mass from the associated liquid by sterilising in a closed vessel. The milk, which may be skimmed, is added in suitable proportion to the extract, preferably during its concentration. The bulk of the tannate of casein readily separates in clots, which are afterwards made into a confection with sugar, or otherwise utilised. The proportion of milk is about 8 oz. to the extract from 1 lb. of tea, but the inventor does not confine himself to this proportion, as it naturally varies with the amount of tannin which it is intended to remove. When treating coffee extracts a considerable reduction is made in the proportion of milk.

—L. de K.

*An Improved Preparation of Malted Pulse Food.* M. Watson, London. Eng. Pat. 10,689, May 31, 1893.

THE invention claims the manufacture of nutritious digestible or partly digested foods from cooked leguminous seeds with malt, or a diastatic product from the same. The food may be further improved by the addition of oily or fatty leguminous matter. Pepsin, sugar, starchy matters, flavourings, essences, or alkali bicarbonates may also be added, but they form no part of the invention. Egyptian or Indian lentils are decorticated, soaked for 12 hours in water, and roasted until the unpleasant taste is removed and an appetising flavour is obtained. The product is then reduced to a flour and mixed with 5—35 per cent. of malted barley or oats. If it is desired to convert most of the starch, the compound may be treated with warm water or infusion of malt, and after some time be dried. Finally, half a per cent. of either sodium or potassium bicarbonate may be added, but this addition is not always desirable. To raise the proportion of albuminoids, ground roasted beans may be

added, but if it be desired to increase the quantity of oily matter only, ground roasted peannts should be used.

—L. de K.

*Improvements in and in Apparatus for the Manufacture of Margarine and Alimentary Fat.* J. Cordeweener and A. de Kunwald, France. Eng. Pat. 24,461, December 19, 1893.

THE patentees have constructed special appliances for the purpose of carrying out the process resulting from the combination of the following operations in the manufacture of margarine and alimentary fats:—(a) the physical union by cold of the constituent elements of the dry nonhydrated fatty matter; (b) the hydration, unification, bleaching, and odorification in the cold of the molecularly united fatty elements. In order to physically unite the non-emulsified elements, oleine, margarine, and stearine, constituting the fatty matters of various origins, these matters in a melted condition are placed in a container, from which they flow by gravitation through a pipe bent upwards at the base, and terminating in a rose inside a vessel containing liquid at a low temperature. The elements of the melted fatty matter, oleine, margarine, and stearine, being thus rapidly chilled, unite molecularly and float upwards to the surface of the refrigerating liquid. The congelation may be obtained either by means of cooled metallic surfaces, or by means of liquids or gaseous fluids. The congealed fatty matter thus obtained becomes, on standing, a translucent product of nacrous aspect. In order to hydrate and bleach this translucent product it is warmed to 20°—22° C. by showering on it liquid at that temperature, and is then transferred to a kind of churn with a perforated dasher. The semi-solid mass, which at the outset was yellow and translucent, is thus caused to pass through the perforations of the dasher, and becomes thereby transformed, without any appreciable elevation of temperature, into a whitish matter of creamy appearance. At this moment cream or milk is added for the purpose of incorporating in the mass the flavouring and odorous elements, which, by the action of the dasher, become intimately infused into the pores of the fatty matter. At this stage the product, if allowed to rest, would become brittle, to obviate which continued kneading is requisite; this, however, may be avoided by running it through a pipe into another churn with perforated dasher containing enough cold water to reduce its temperature to 12°—14° C. when the dasher is worked vigorously. The bottom of this churn is movable, so that the mass of factitious butter can be allowed to drop out into a truck with perforated bottom,

in which it drains without becoming brittle. Finally, it is kneaded to expel superfluous water, and then is said to possess all the visibly apparent qualities of natural butter.

—C. R. A. W.

### (B.)—SANITARY CHEMISTRY AND WATER PURIFICATION.

*Purification of Water.* Villon. *Revue Chim. Industrielle*, 1893, 87.

THE removal of calcium and magnesium sulphates and carbonates from potable and industrial waters, is generally effected by the addition thereto of caustic potash, sodium carbonate, or barium chloride. This method is, however, not only insufficient, because ammonium sulphate remains in the water, but it necessitates an exact estimation of the reagents and the use of costly apparatus.

The following electrolytic process is less costly and needs no surveillance, because the amount of the necessary reagents produced by the electric current is always equivalent to the compounds to be precipitated; no trace of the reagents remains in the water. The percentage of incrusting substances—the magnesium and calcium salts mentioned above—in the water, is determined in the usual manner, and the equivalent quantity of sodium nitrate is added, the water being thereafter submitted to the action of an electric current. Lead electrodes are employed. Caustic soda separates at the negative pole and nitric acid at the positive pole, which latter must be renewed from time to time. The last-named acid attacks the electrode, forming lead nitrate. Calcium and magnesium carbonates and sulphates are precipitated by the caustic soda, sodium sulphate and carbonate being formed; the sodium carbonate then precipitates the lead nitrate.

About 250 litres of water can be purified in an hour; and when 20 cubic metres of water are dealt with daily, the cost is not more than 5 centimes per cubic metre. The precipitates settle quickly, thus enabling the clear water to be drawn off.—A. R. L.

### PATENTS.

*Improvements in Filtering Sewage.* B. Rushford, Aldershot, and J. Edwards, Aldershot. Eng. Pat. 5689, March 16, 1893.

THE inventors claim as chief improvement a filtering-bed layer consisting of fine slag and small coal-dust screenings in equal proportions unwashed, with a small quantity of sulphate of iron.

The crude sewage is let into precipitating tanks and there treated, preferably with Gibb's alumina. The sewage water is then passed through layers of large gravel, broken gravel, shingle, coarse sand, fine sand, and then through the above filtering bed. Finally it passes through fine sharp sand.

The easing of the filter may be made of any impervious material. The filters may be constructed in series. A square foot of surface is capable of filtering about 2,500 gallons in 24 hours.—L. de K.

*Improvements in Apparatus for Softening Water.* C. E. Gittins. Eng. Pat. 8900, May 3, 1893.

A CLOSED vessel containing lime is fed with hard water, by a branch pipe from the rising main, and is thoroughly mixed with the lime by means of a stirrer, driven by a water motor, which latter is preferably actuated by the incoming water. The softened water afterwards flows away into the store cistern. Cut-off cocks are fitted on the inflow and outflow pipes to disconnect it for repairs, and suitable filling and cleaning orifices are provided on the mixing vessel.

—E. G. C.

*Improvements in or relating to Machinery or Apparatus for Treating, Deodorising, Drying, and Utilising, Excreta, Nightsoil, Town's Refuse, and the like.* S. Smithson. Eng. Pat. 23,082, December 1, 1893.

THE inventor claims as chief improvement the sorting, drying, and deodorising of town refuse by means of machinery. The refuse (excreta, nightsoil, &c.) is deposited in the hopper of a screen and passed through a screen, whereby the finer and useful portions are separated from the useless, which are then destroyed in a destructor. The useful portion is conveyed to and dried in a revolving cylinder, and caused to travel in an opposite direction to that from whence the heat proceeds. After drying, the material is passed through a second and finer screen to collect those portions which are useful for, say manurial purposes.—L. de K.

### (C.)—DISINFECTANTS.

#### PATENTS.

*Improvements in the Manufacture of Antiseptic and Disinfectant Materials for Sanitary Purposes.* C. R. Alder Wright, London. Eng. Pat. 1950, March 7, 1893.

THE inventor's claims are: I. An improved process for preparing monochloro- or bromonaphthalene fairly free from the higher halogen derivatives. II. The use of these compounds as antiseptic or germicide materials. III. The manufacture of disinfecting fluids by dissolving or suspending the said compounds in a solution of rosin soap, or in an emulsion of rosin oil in soap. IV. The manufacture of sanitary soaps by incorporating the naphthalene derivatives with ordinary soap. V. The manufacture of disinfecting powders by mixing the compounds with a suitable absorbent, such as sawdust, plaster of Paris, &c.

Monochlorinated naphthalene is prepared by suspending naphthalene in water contained in a suitable vessel provided with a cooling jacket and an efficient agitator. Chlorine, about 60 parts for every 100 parts of the hydrocarbon, is passed through, and the unaltered naphthalene is removed from the semi-solid mass by means of a current of steam. The monochlorine compound is left as an oily liquid, which may be directly used. The brominated compound is prepared in an analogous manner. Mixed compounds may also be produced.—L. de K.

*Improvements in Vessels for Containing Liquefied Sulphur Dioxide for Use as a Disinfectant and Vermin Destroyer, and for other Medical and Sanitary Purposes.* E. Soy, London. Eng. Pat. 12,276, June 23, 1893.

THE inventor claims to have constructed a vessel made of glass or other suitable material, which has a contracted orifice, through which it is filled with liquefied sulphur dioxide. The end is finally sealed in the flame of a blow-pipe.

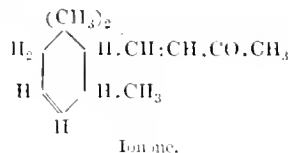
The vessels are kept in a suitable box to prevent accidental breakage. When required for disinfecting a room, the box is opened and the tubes are struck with a hammer so as to break the glass. The room must then be at once left and the door sealed. The liquefied acid will rapidly resume its gaseous form and penetrate into every crevice of the floor, walls, and ceiling.—L. de K.

*Improvements in Disinfecting, and Apparatus for that Purpose.* E. Hermite, London, C. F. Cooper, London, and E. J. Patterson, London. Eng. Pat. 22,279, November 21, 1893.

THE inventors claim as chief improvement a method of disinfecting by conducting "electrolysed chloride solution" by pipes from a station to closets, urinals, and drains. The apparatus, a combination of a dynamo machine, electrolyser, and conductors, with suitable pumps, cisterns, pipes, cocks, &c., is minutely described and drawn in the original specification.



The process is carried out by charging reservoirs with sea water, or, if this is not available, with common water containing 10 kilos. of salt and 5 kilos. of commercial magnesium chloride per 1,000 litres. The liquid is now electrolysed until it contains about 2 grms. of free chlorine per litre, when it is diluted with six or seven times its bulk of water. The amount of chlorine being so small, the use of the liquid as a disinfectant is not attended with any unpleasant chlorine smell, whilst it destroys the unpleasant and noxious odour and effluvia of sewage matter.—L. de K.



Iou me.

—A. K. M.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Note on the Liberation of Chlorine during the Heating of a Mixture of Potassium Chlorate and Manganese Peroxide.* Herbert McLeod, F.R.S. Proc. Chem. Soc. 1894, [133], 29—30.

In 1889 the author suggested an explanation of the catalytic action which takes place when an "oxygen mixture" is heated, an essential feature in the explanation being the evolution of a small quantity of chlorine; a few months later O. Brunck published in the *Berichte* a statement that the gas evolved together with oxygen is not chlorine but ozone (this Journal, 1894, 170).

The author has made several experiments to endeavour to solve the question. One test only was made for the presence of ozone in the gas, but it indicated that under the conditions of the experiment, ozone was not produced. In course of discussion Prof. Duostan remarked that the odour of the gas evolved from the mixture was more like that of an oxide of chlorine than chlorine itself. He inquired whether Prof. McLeod had ascertained whether a mixture of potassium chloride and manganic oxide furnished chlorine when heated in mercury vapour. If so, then not only the production of chlorine, but also of permanganate and alkali, from the chlorate, was susceptible of another explanation.

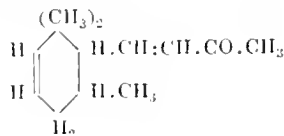
Prof. McLeod stated that he had not hitherto closely investigated this question.

*The Odoriferous Principle of the Violet.* F. Tiemann and P. Krüger. Comptes rend. 1893, 117, 548—552.

THE best known sources of the aroma of the violet are the fresh flowers of the violet and the dried orris root. The latter was chosen as being the source from which the odoriferous principle could be most readily obtained. The orris root is exhausted with ether and the ethereal extract is distilled in a current of steam. The product contains: (1) myristic acid, oleic acid, and other fatty acids; (2) the methylic ethers of the above acids; (3) traces of oleic aldehyde; (4) small quantities of neutral substances; and (5) *irone*. The last substance is the odoriferous principle of orris root, and is a ketone of the formula  $\text{C}_{13}\text{H}_{20}\text{O}$ ; it is an oil which is readily soluble in alcohol, ether, and chloroform. By the action of hydriodic acid on *irone*, a hydrocarbon *irone*,  $\text{C}_{13}\text{H}_{18}$ , has been obtained.

In an attempt to synthesise *irone*, the authors obtained an isomeric substance by the condensation of citral with acetone. This substance is named *pseudo-ionone*, and on treatment with dilute acids is converted into *ionone*,  $\text{C}_{13}\text{H}_{20}\text{O}$ , which likewise possesses and odour resembling that of violets. From this they have obtained a hydrocarbon *ionone*,  $\text{C}_{13}\text{H}_{18}$ , and some other derivatives.

The authors assign the following formulae to the two isomeric ketones:—



Iron.

*Irisin, the Glucoside of the Iris Root.* G. de Laire and F. Tiemann. J. Amer. Chem. Soc. 1893, 15, 351—355.

THE alcoholic extract of the powdered, dried rhizomes of *Iris florentina* is stirred with 2 litres of lukewarm water and 1 litre of a mixture of acetone and chloroform of 0.950 sp. gr.; the upper acetone-chloroform layer contains the substances of the alcoholic extract insoluble or only slightly soluble in water. The two layers are separated, the white flakes collected on a filter, washed with a little hot water, and dried at 100°. The white powder thus obtained is washed with ether and light petroleum, and crystallised from boiling dilute alcohol.

*Irisin*,  $\text{C}_{22}\text{H}_{34}\text{O}_{13}$ , crystallises in white needles, which become yellow in moist air and melt at 208°. It is moderately soluble in acetone, but hot alcohol is the best solvent for it. By hydrolysis it yields *D*-glucose and *Irogenin*,  $\text{C}_{20}\text{H}_{30}\text{O}_4$ , which crystallises in rhombohedra, melts at 186°, and dissolves in most solvents, particularly when warmed with them; it behaves as a phenol and is coloured violet by ferric chloride. *Dibenzoyl*- and *diacetyl*-derivatives were obtained.—A. G. B.

*Bulgarian Oil of Roses.* W. Markownikoff and A. Reformatsky. J. prakt. Chem. 48, 293—314.

THE authors have investigated three specimens of genuine oil of roses obtained through the Bulgarian Government. The chief constituents of oil of roses are elaeoptene and stearoptene, but the present paper deals principally with the former, to which is due the peculiar perfume of the oil. On freezing, the elaeoptene separates out from the oil in the crystalline form, whilst the stearoptene remains in the mother-liquor, from which it may be precipitated by the addition of alcohol. After repeated freezing (at  $-55^\circ\text{C}$ .) and fractional distillation, elaeoptene is obtained as a yellowish oil, boiling at  $222^\circ\text{—}222.5^\circ\text{C}$ . It rapidly oxidises on exposure to the air. The analysis agrees with the formula  $\text{C}_{10}\text{H}_{16}\text{O}$ . On treating with acetic anhydride it yields an acetate, and on oxidation with potassium permanganate a glycerin is formed. The molecular refraction of elaeoptene shows that it contains only one double bond, and in confirmation of this it was found that only two atoms of bromine add themselves on. The authors conclude that the chief constituent of elaeoptene is an alcohol having the above formula, for which they propose the name roseol. It belongs to the allyl alcohol series and is isomeric with the two known members of this series, allyldipropylcarbiol, b.p.  $192^\circ\text{C}$ ., and allyldipseudopropylcarbiol, b.p.  $169^\circ\text{—}171^\circ\text{C}$ . From its higher boiling point roseol is probably the normal alcohol. Oil of roses is thus closely related to some other ethereal oils. For example, Gladstone and Wright have shown that oil of citronella (*Andropogon nardus*) contains a substance,  $\text{C}_{10}\text{H}_{16}\text{O}$ , which Dodge proved was an aldehyde and that on reduction it gave an alcohol,  $\text{C}_{10}\text{H}_{20}\text{O}$ , boiling at  $225^\circ\text{—}230^\circ\text{C}$ , and possessing the smell of roses. According to Semmler's researches (Ber. 24, 201), oil of verbena (*Andropogon citratus*) contains an aldehyde identical with that obtained from oil of citronella. Indian oil of geranium (*Andropogon Schoenanthus*) also appears to contain a similar aldehyde. It seems quite possible that the valuable elaeoptene in oil of roses may be obtained from the much cheaper substances, oil of verbena and oil of citronella, and perhaps also by the reduction of geraniol. The rest of the paper is of a polemical nature, in which the authors declare that Eckhart's results (Ber. 24, 958 and 4205; this Journal, 1892, 265), which are not in agreement with their own, were likely obtained by experimenting on impure material, or through some adulteration in the oil of roses.—J. S.

*The Properties of Essential Oils, and their Employment in Painting Compared with that of Mineral Oils.* L. Lettenmayer. Chem. Zeit. 17, 1893, 1506.

In painting, essential oils are used for two purposes, either as simple diluents for oil pigments or as substances assisting in the drying of the other oils. If required for the first purpose only, oils free from or poor in terpenes, such as heavy camphor oil, clove, cajuput, or perhaps lavender, must be employed, or else oils containing terpenes which have been resinified. As driers, fresh turpentine or the light oil of camphor are the most suitable, as only those are available which yield a perfectly clear and transparent residue. The best resin solvents are turpentine, oil of rosemary, and the light camphor product, and these also, either alone or mixed with fresh nut oil, evaporate most quickly and completely, leaving only a small residue. Rectified oil of amber and wax oil lose by evaporation 50 per cent. in 24 hours, but leave after 75 days 20 per cent. and 40 per cent. of non-volatile matter respectively. The oils of cajuput and spike evaporate more slowly, leaving a small residue, while oil of lavender is the slowest. Heavy camphor oil and that from copaiba are useless, the latter gaining in weight on exposure. By means of petroleum, either the burning oil or a specially prepared product, the drying of the colours can be regulated to a nicety, but the residue, being soft and sticky and liable to turn brown in the air, renders the pictures liable to injury, and its use cannot be recommended, especially as it has the additional disadvantage of precipitating resins from their solutions.

The author advises the use either of rectified turpentine, light oil of camphor, or oil of rosemary when quick-setting paints are wanted, oil of lavender or spike being employed, either alone or mixed with the others, when slower drying products are preferred. In every case care must be taken that the oils are genuine and free from adulteration.

—F. H. L.

*The Preparation of Basic Organic Bismuth Salts.* A. Voswinkel. Pharm. Zeit. 1893, 33, 594.

*Basic bismuth gallate* is prepared by dissolving 100 grms. of bismuth chloride in a solution of 1,800 grms. of sodium chloride. To the filtrate, 400 grms. of gallic acid are added and the whole boiled for 20 minutes, the evaporated water being made up by the addition of more. The liquid is then poured into a large quantity of water so that an excess of gallic acid remains in solution. The precipitate, having the formula  $(\text{OH})_3\text{C}_6\text{H}_2\text{COO}.\text{Bi}(\text{OH})_2$ , is washed and dried.

*Basic bismuth pyrogallate* is prepared by dissolving 150 grms. of pyrogallol in a solution of 650 grms. of common salt, and adding a solution of 316 grms. of bismuth chloride dissolved in 1,000 grms. of common salt. The whole is heated on the water-bath for half an hour and poured into 20 times the amount of water. The formula of the compound is  $\text{OH}.\text{C}_6\text{H}_3.\text{OOBi}.\text{OH}$ . (This Journal, 1894, 171.)

—F. H. L.

*The Quantitative Estimation of Acetone and Salicylic Acid in "Sal Acetol."* H. Eckenroth and K. Kock. Also *ibid.* H. Helbing and F. W. Passmore. Pharm. Zeit. 1893, 38, 621.

See under XXIII., page 288.

*Contributions to the Knowledge of Albuminoses.*

H. Schrötter. Monatsh. f. Chem. 1893, 14, 612.

WHEN albuminous matters are decomposed by means of ferments, two classes of products are formed, distinguished as *peptones* and *albuminoses*. These are separable by saturating the liquid with ammonium sulphate, when the latter are precipitated, whilst the former remain in solution. By fractionally precipitating albuminose solutions with common salt, various more or less different substances are obtained, e.g., dys-, hetero-, proto-, and dentero albuminose. From the commercial manufactured article known as "*Peplone Witte*," the author has separated a variety of albuminose, soluble in alcohol, not distinctly crystallisable

therefrom, but exhibiting a microcrystalline structure, and forming a tolerably definite hydrochloride. Determinations of the molecular weight by Raoult's method give values lying between 587 and 711. On treatment with benzoyl chloride two benzoate ethers were formed, one containing sulphur, the other sulphur-free. C. R. A. W.

*Cresol as an Antiseptic.* Lacroix - Hunkialéyendian. Journ. Pharm. Chim. 28, 31.

Cresol is only slightly soluble in water, but its solubility in an aqueous solution of potassium cresol renders its employment as an antiseptic possible. This solution is named "*Solveol*." The cresol of coal-tar consists of a mixture of three isomerides, *o*, *m*, and *p* cresols, and this mixture has a greater action on micro-organisms than has either of the pure isomerides. The author has investigated the action of "*Solveol*" on putrescible matter, and considers it of great value in surgical work, having a strong antiseptic action, not affecting the hands and instruments, having a less unpleasant smell than phenol, and, in addition, giving a permanent, clear, neutral, aqueous solution, which does not solidify on cooling.—J. G. W.

*Isomers and Analogues of Aniligne.* G. N. Vis. J. prakt. Chem. 48, 24—30.

*Aniligne*, or *o*-ethoxy-*ana*-benzoyl-amidoquinoline, is formed by the reduction of ethoxynitroquinoline and treatment of the amido compound with caustic soda and benzoyl chloride. It is insoluble in water, but soluble in hot alcohol, from which it crystallises in small white crystals. It is used as an antineurotic. The corresponding methoxyl compound is prepared in a similar way, and possesses similar properties except that its salts are mostly easily soluble. It melts at 268—269° C.

*o*-Methoxy-*ana*-amidoquinoline is produced from the nitro compound by reduction with tin, stannous chloride, or iron, and hydrochloric acid. It crystallises from alcohol in yellow needles, m.p. 155°—156° C., and from water with 1 mol.  $\text{H}_2\text{O}$ , m.p. 76°. The acetyl compound is obtained by treatment of the free base with glacial acetic acid. From hot water it may be obtained in needles or in octahedral crystals, m.p. 179°.

*p*-Ethoxy-*ana*-nitro-quinoline, from *p*-ethoxy-quinoline by nitration, melts at 117° and is practically insoluble in water. The corresponding amido compound is soluble in hot water, yielding crystals with 1 mol.  $\text{H}_2\text{O}$ , m.p. 76°, the anhydrous base melting at 115°—116°. Acetylation gives a mono-acetyl derivative, which crystallises from hot water in white scales, m.p. 163°; the salts are soluble in water. The corresponding benzoyl compound melts at 144°, and is fairly soluble in alcohol, its salts being all soluble in water.

—J. W.

*On the Xanthine Bodies.* P. Balke. J. Prakt. Chem. 47, 537.

See under XXIII., page 284.

*Reactions and Constitution of Morphine.* G. N. Vis. J. Prakt. Chem. 47, 1893, 584.

See under XXIII., page 284.

*The Aerial Oxidation of Terpenes and Essential Oils.* C. T. Kingzett. Proc. Chem. Soc. 1894 [135], 51—52.

AFTER referring to his previous work on the subject, the author describes the results of an examination of a number of oily and semi-resinous products obtained on oxidising various turpentines by passing air either into the oil or into a mixture of the oil and water.

It is found that when any terpene is oxidised by air in the presence of water, or when the oxidised oil is shaken up with water, the aqueous solution always contains hydrogen

peroxide and a substance that may be obtained by evaporating off the water, this latter at the ordinary temperature of the atmosphere having the consistency of soft pitch, and being very bitter to the taste. Analyses are quoted of a considerable number of samples prepared from various oils, showing that the composition of the substance is approximately represented by the formula  $C_{10}H_{14}O_2$ . It appears to yield some cineol when distilled with dilute sulphuric acid. The oils which were found to yield this substance, and whether by oxidation alone or in presence of water, were Russian turpentine, American turpentine, camphor oil, several varieties of Eucalyptus oil, and an oil called "*Palustre*" obtained by the destructive distillation, at low temperatures, of *pinus taeda* and *pinus palustris* of the South Atlantic coast of the United States. (This Journal, 1890, 1005—1008.)

When Russian turpentine is oxidised in the presence of water and the aqueous solution is distilled, a small quantity of a light-yellow coloured oil passes over, which darkens on standing, and has a characteristic smell something like that of thymol. About 23 grms. of this oil were obtained by distilling 25 litres of the fluid. On analysis it gave numbers approximating to those corresponding with the formula  $C_{10}H_{14}O_2$ ; but on distilling this oil, after drying it over calcium chloride, only a portion passed over between 180 and 205°, leaving a viscous residue.

When oxidised Russian turpentine is evaporated down on the water-bath until all the volatile matter has apparently been driven off, and the residue is dried at 100°, a dark brown translucent mass is obtained, which, when cold, is quite hard and brittle; an analysis of this substance shows that it has a composition represented roughly by the formula  $C_{10}H_{14}O_2$ . Other oils afford somewhat similar results.

The author believes that in the limited oxidation (by air) of essential oils the terpene grouping is not broken down, as he has ascertained, by special investigation, that the acetic acid which sometimes accompanies the other products when the process is conducted in the presence of water, is not observed to be present if only pure or refined oils are employed, and he has not met with any product which can be assumed to contain less than 10 atoms of carbon in the molecule. See also this Journal, 1886, 7; 1888, 549; and 1890, 3.

## PATENTS.

*Improvements in the Manufacture of Salols.* H. Byk, Berlin, Germany. Eng. Pat. 23,419, December 20, 1892.

The patentee claims an improved process for obtaining salols (phenyl or naphthyl salicylates).

1. Salicylic acid (100 kilos.) is mixed with phosphoric anhydride (55 kilos.) in a closed iron boiler provided with a stirrer; the reaction is started by raising the mixture to a moderate temperature under a gentle pressure of carbonic acid gas. The source of heat is removed when the thermometer registers a temperature of 65° C., as otherwise the heat evolved in the reaction causes a rise of temperature above 100°, which acts deleteriously. Half an hour subsequently to this, the mass is heated at 90° C. for three hours, when the reaction is complete. Anhydrous phenol or naphthol is now added, whereby a rise in temperature of 15 to 20° C. is produced; the mixture is then stirred for an hour without further application of heat. The product is allowed to cool in earthenware vessels, where it separates into a deep red lower layer containing pyrophosphoric acid, and an upper pale-coloured paste consisting of the crystalline salol. When phosphoric anhydride alone is used, the pyro-phosphoric acid syrup separates with difficulty. "By the addition of 12 kilos. of syrupy phosphoric acid of sp. gr. 1.7 to 1.60 kilos. of salicylic acid, after preparing the boiler with a dressing of phenol, a good separation of the phosphoric acid from the salol paste is obtainable."

2. Meta-phosphoric acid (2 kilos.) is fused with salicylic acid (100 kilos.) and phenol (70 kilos.), a current of carbonic acid gas being passed through the mixture meanwhile. The boiler is then closed and the mass stirred for half an hour, after which the product is removed with hot water.

3. Salicylic acid and syrupy phosphoric acid of specific gravity 1.7 (100 kilos. of each) are heated with phenol (70 kilos.) in an open vessel for 6—7 hours; the mixture is kept stirred, and a current of carbonic acid gas is passed through it.

4. A quantity of a salicylate, equivalent to 100 kilos. of salicylic acid, is heated with 80 kilos. of phosphoric anhydride, 90 kilos. of meta-phosphoric acid, or 130 kilos. of syrupy phosphoric acid.

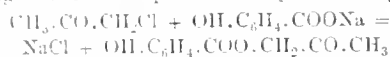
5. 100 kilos. of a mixture of equivalent quantities of sodium salicylate,  $C_6H_4(ONa)COONa$ , and phenol, is heated with 30 kilos. of phosphoric anhydride or 40 kilos. of syrupy phosphoric acid. Meta-phosphoric acid is not to be recommended on account of its high fusing point. The raw salol, separated from the aqueous stratum, is first washed with hot dilute soda, and then twice with hot water, to remove unaltered salicylate; the phenol is got rid of by blowing steam into the product; the crude salol is crys-tallised from alcohol.—A. R. L.

## *Manufacture of Salicylic Acid Ester of "Acetol."*

P. Fritsch, Ludwigshafen am Rhein, Germany. Eng. Pat. 3361, February 22, 1893.

MANY warnings have been raised, especially by Kohert and Hesselbach, against the use of salol for medicinal purposes, on the ground that phenol is set free from the compound in the human body and poisoning results therefrom. The patentee claims the manufacture the *salicylic acid ester of acetol*, i.e., the production of an ether of salicylic acid in which the latter is combined with a non-poisonous substance.

Sodium salicylate (180 parts) is suspended in alcohol (200 parts), and monochloroacetone (92.5 parts) added to the mixture, which is then heated in a reflux apparatus until the odour of monochloroacetone has disappeared. The following reaction takes place almost quantitatively:—



The reaction likewise occurs when the components are heated in aqueous or aqueous-alcoholic solution, and also, but with diminished yield, when the components are heated together without solvents. The new ester crystallises from alcohol in long woolly needles, melts at 71° C., is insoluble in cold water, and only sparingly soluble in hot water. A dilute solution of ammonia or of soda hydrolyses it with great facility.—A. R. L.

## *Manufacture of Compounds of p-Amidophenol Ethers and Aromatic Oxalaldehydes.* O. Imray, London. From the Society of Chemical Industry of Basel, Switzerland. Eng. Pat. 4191, February 25, 1893.

THE claims set forth in this specification comprise the manufacture of compounds of *p*-amidophenol ethers and aromatic oxalaldehydes, by two methods described below, as substances having in some cases valuable therapeutic properties; the latter is especially true of the compounds obtained by the action of salicylaldehyde on *p*-phenetidine or *p*-anisidine.

1. When a solution containing 23 parts of salicylaldehyde in 100 parts of alcohol is mixed with one of *p*-phenetidine containing 26 parts in 100 parts of alcohol, these being molecular proportions, the following reaction occurs:—



Heat is evolved, and at the end of two hours, there separates from the mixture a magma of yellow needles; these when recrystallised from alcohol melt at 90°—91° C. When, instead of *p*-phenetidine, *p*-anisidine is used, a similar compound is obtained which crystallises in yellow leaves and melts at 80°—82°.

2. These two compounds are also obtained when *o*-hydroxy-*p*-amidophenol, melting at 135° (see Ber. 1892,

25, 2754), is ethylated or methylated by boiling a solution of it in ethyl or methyl alcohol with the requisite amount of caustic alkali and ethyl bromide or methyl bromide.

—A. R. L.

*A Method of treating Tobacco or Tobacco Products to Prevent or Neutralise the Injurious Action of Nicotine contained therein.* H. H. Lake, London. From H. Gerold, Halle, and H. O. Wendt, Bremen, Germany. Eng. Pat. 4774, March 4, 1893.

Tobacco leaves are impregnated with a solution containing tannic acid and the essential oil of *origanum vulgare*, or with an infusion of oak bark and *herba origani vulgaris*, the proportion of the solution added being regulated according to the richness of the tobacco in nicotine. As soon as the leaves have absorbed the infusion they are lightly pressed and then dried with moderate heat. This treatment prevents the injurious dissolution and distillation of the nicotine.—C. A. K.

*Improvements in the Manufacture of Guaiacol.* J. Y. Thompson, London. From F. von Heyden, Nachfolger, Radebeul, near Dresden, Germany. Eng. Pat. 5284, March 11, 1893.

Impure guaiacol, or wood-tar, or materials containing guaiacol, are cooled by a freezing mixture, when pure guaiacol separates out, the impurities remaining liquid. The product thus separated is quite pure and is separated by pressing, centrifugal action, or other suitable means. The impure guaiacol can also be diluted, before cooling, by a neutral solvent, such as petroleum ether, and the pure product subsequently distilled, if necessary.—C. A. K.

*Improvements in the Manufacture of Piperazine and its Salts.* H. E. Newton, London. From the Farben-fabriken vormals F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 5320, March 11, 1893.

PIPERAZINE is produced by boiling dinitrosodiphenyl-piperazine, which is obtained by the action of sodium nitrite upon diphenylpiperazine, with hydrochloric, sulphuric, or phosphoric acid. The use of sulphuric acid is claimed in a previous patent (Eng. Pat. 4497, March 7, 1892; this Journal, 1893, 463). The resulting solution is neutralised with alkali, filtered, an excess of alkali added, and the piperazine distilled over with steam. The piperazine or its salts are then obtained from the distillate in a suitable manner.—C. A. K.

*Process of Producing Esters from Creosote, Guaiacol, or Creosol.* C. D. Abel, London. From F. von Heyden, Nachfolger, Radebeul, near Dresden, Germany. Eng. Pat. 5856, March 18, 1893.

THE invention relates to the production of esters from creosote, guaiacol, or creosol with fatty acids, especially oleic and stearic acid, but also with other fatty acids, such as caprylic, capric, lauric, myristic, palmitic, arachidic, ricinoleic, linoleic, erucic, caproic, sebacic, &c. The esters are produced by the action of the phenols (or their salts) upon the fatty acids (or their salts) in the presence of dehydrating agents, such as the chlorides of phosphorus, or sulphur, or acid alkaline sulphates; or the acid chlorides of the fatty acids can be first prepared and these treated with the phenols. The resulting esters are liquids, insoluble in water, soluble in alcohol and ether; they are all devoid of the smell and taste of the phenols from which they are derived, and have no burning or caustic action on the mucous membranes. It is intended that they should replace the free phenols for medicinal purposes.—C. A. K.

*Manufacture of Production of Salols.* C. Kolbe, Radebeul, near Dresden, Germany. Eng. Pat. 6749, March 18, 1894.

THE patentee claims an extension of his previous patents for the manufacture of salols (Eng. Pat. 8018, June 16, 1886; 10,260, July 22, 1887; 13,169, September 12, 1888; 17,221, October 28, 1890. This Journal, 1886, 577; 1887, 561; 1888, 587; 1889, 817; and 1892, 58) to substituted phenols, the dehydrating agents employed being the same as previously claimed, with the addition of carbonyl chloride and of thionyl chloride. The substituted phenols employed are 1,2, 1,3, and 1,4-chloro-, bromo-, or iodo-phenol, 1,2,3 and 1,2,4-dibromo-, dichloro-, and diiodo-phenol, and trichloro-, trichloro-, and triiodo-phenol. The new compounds are colourless bodies insoluble in water, soluble in alcohol, which form salts with alkalis, and are separated on boiling with alkali. They possess advantages for therapeutic purposes over the ordinary salols, being free from smell and having only a slight taste, if any. The para-chlorophenol compound has the formula  $C_6H_4(OH)(CO_2OC_2H_5)$ .

—C. A. K.

*A Method for Producing Artificial Citric Acid.* A. L. C. Welmer, Hanover, Germany. Eng. Pat. 5620, November 2, 1893.

THE patentee claims the manufacture and method of producing artificial citric acid by the fermentation of carbohydrates, glycerol, and analogous substances, under the influence of certain fungi (organisms), particularly of two hyphomycetes discovered by himself and named *Citrifungus citricus*. The spores of these organisms, which are generally present in the air, develop with special facility into sugar-like substances in presence of small quantities of acid. Two species have been isolated in a state of purity. The organisms produce an intense citric acid fermentation in sugar solution; their separation is not necessary for industrial purposes.

To prepare these organisms, sugar solution containing 2—5 per cent. of citric acid is allowed to remain for a few days; the growth formed on this solution is introduced into sterilised sugar, where it develops. The "pure culture" prepared in this way is then introduced into large vessels containing a sugar solution in which is dissolved a small quantity of ammonium nitrate, dipotassium phosphate, and magnesium sulphate; the liquid is then allowed to remain at the ordinary temperature for 8—14 days. At the expiration of this time the liquid contains a certain amount of citric acid, so that it can be directly employed for human juice. The acid can also be isolated, and preferably in the form of a salt. For the latter purpose the liquid is neutralised with lime or carbonate of lime, or the latter or other convenient salts are added previously to the culture liquid, so that calcium citrate is precipitated as soon as the acid is formed.—A. R. L.

*An Improved Preparation of Ferrous Salts.* F. W. W. Clark, London. Eng. Pat. 22,931, November 29, 1893.

THE ferrous salt, either alone or mixed with other drugs which are prescribed with ferrous salts, is mixed with glyco-gelatin, a preparation of gelatin, glycerol, and water. Ferrous salts thus mixed are not readily oxidised when in contact with air.—C. A. K.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

### PATENTS.

*An Improvement in the Manufacture of Photographic Plates.* O. Magerstedt, Berlin. Eng. Pat. 5932, March 20, 1893.

This invention has for its object to obviate the defect shown by negatives which have been exposed on subjects possessing much contrast, known as halation. The plate is coated with gelatin containing a dye in solution, of which the following are the most suitable:—The red colours of the rosaniline series and their sulphonic acids, those of the rosolic acid series, the reds and yellows of the aurine process, and the safranines, phosphines, uranines, &c. When this solution is dry the sensitive emulsion is applied as usual, and the dye will be completely removed from the negative either by the developer or the fixing bath, an acid one being preferably employed.—F. H. L.

*An Improved Method of Photographic Printing in Bromide of Silver.* E. Norden, Hatcham, S.E. Eng. Pat. 6491, March 27, 1893.

PAPER or other material is coated with a mixture of gelatin and a soluble bromide—with or without the addition of a small proportion of chloride. It is dried and floated in a solution of silver nitrate, dried again, and treated with a 20 per cent. solution of potassium oxalate. It forms a "printing-out" paper, or one not requiring development.—F. H. L.

*Hygroscopic Paint or Coating Material for keeping Packing Cases Dry.* C. D. Abel, London. From the Westfälisch-Anhaltischen Sprengstoff Actien Gesellschaft. Eng. Pat. 7571, April 13, 1893.

To avoid the expense of the metal-lined cases with soldered joints usually employed for packing hygroscopic goods which have to travel by sea, the inventors make use of an ordinary porous packing-case and coat it on the inside, either all over or only in one place, with a mixture of 30—40 parts of calcium chloride, 30—35 parts of glue, and 10—15 parts of an absorbent powder, such as infusorial earth. It is stated that the calcium chloride will absorb its six equivalents of water, and yet be retained in place by the glue and the infusorial earth. The inventors also claim the use of other deliquescent salts, other absorbents, and other binding materials.—F. H. L.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*The Gelatin Explosives.* P. G. Salford. J. Amer. Chem. Soc. 1893, 15, 552—560.

The chief "gelatin" explosives are blasting gelatin, gelatin dynamite, and gelignite. They all consist of the variety of nitro-cellulose known as collodion-cotton (*i.e.*, the pentanitrated tetra-nitrates, the hexa-nitrate-cellulose being known as gun-cotton) dissolved in nitro-glycerol, and made up with various proportions of wood pulp, and some nitrate or other material of a similar nature.

Blasting gelatin has a specific gravity of 1.5—1.55, and consists of 92—93 per cent. of nitro-glycerol and 7—8 per cent. of nitro-cotton, of which the following is a typical analysis:—

Soluble nitro-cotton .....	59.118
Gun-cotton .....	0.612
Non-nitrated cotton .....	0.210
Nitrogen .....	11.64
Ash .....	0.25

"Collodion-cotton" is soluble in ether-alcohol, nitro-glycerol, and acetone; "gun-cotton" only in the last-named liquid. It follows that nitro-cotton used in the manufacture of gelatin compounds should be free from gun-cotton, and it is also important that the nitro-cotton should stand the Government heat test. It must be finely powdered and rubbed through a brass sieve by hand in the dry state. Its percentage of nitrogen should be over 11 per cent. Before incorporation with the nitro-glycerol, the nitro-cotton, which is generally transported wet in zinc-lined wooden boxes, must be dried, the progress of drying being determined by testing a sample at intervals by drying it in the water-oven. The product should contain not more than 0.5 per cent. of moisture, and in sifting and handling should be exposed to the air as little as possible, as it may take up some 2 per cent. of hygroscopic moisture. The drying house is usually a wooden building fitted with a framework to contain drawers of wood, the bottoms of which are made of brass or copper wire netting. The cotton is spread out on these to a depth of 2 in., and a warm current of air passed over it. If the building be itself heated by hot-water pipes, the pipes should be boxed in, as much dust from the nitro-cotton settles on all parts of the building. A temperature of 40° C. in the drying house is quite hot enough.

Gelatin dynamite and gelignite are manufactured in the same way as blasting gelatin. The following are typical analyses of the three compounds:—

	Blasting Gelatin.	Gelatin Dynamite.	Gelignite.
Nitro-glycerol .....	32.94	71.128	60.514
Nitro-cellulose .....	7.66	7.632	4.888
Wood pulp .....	..	4.259	7.178
Potassium nitrate .....	..	16.720	27.420

The preparation of blasting gelatin is effected by mixing dry nitro-cotton with pure dry nitro-glycerol in a lead-lined trough by stirring at a temperature of 40°—45° C. for half an hour until gelatinisation takes place. The jelly is mixed in a kneading machine (made of gun-metal) such as is used for bread. The preparation of the mixture of nitro-cotton and nitro-glycerol for the manufacture of gelatin dynamite and gelignite takes place in the same manner, the other ingredients (wood pulp and nitre) being afterwards incorporated. The application of heat in the manufacture of jelly from collodion-cotton and nitro-glycerol is necessary unless some solvent other than nitro-glycerol, *e.g.*, acetone, acetic ether, methyl, or ethyl alcohol, be employed. These solvents not only dissolve the nitro-cellulose in the cold, but also render the resulting explosive less sensitive to concussion, reduce its rapidity of explosion, and lower the freezing-point. The product, however obtained, is moulded in an apparatus like a sausage machine, the issuing rod of explosive being cut into lengths suitable for cartridges by means of a wooden wedge. The cartridges are required to pass the Government liquefaction test, which is carried out by cutting a cylinder of which the height is equal to the diameter, pinning it to a flat paper surface, and exposing it for 144 hours to a temperature of 85°—90° F., at the end of which time the cylinder should not have diminished in height by more than  $\frac{1}{8}$  in. and the cut edges should still be sharp. There should be no stain of nitro-glycerol upon the paper. The exudation test, which is also compulsory, consists in freezing and thawing the explosive three times, after which no exudation should be perceptible.

According to Berthelot, blasting gelatin containing 91.6 per cent. of nitro-glycerol and 8.4 per cent. of nitro-cellulose gives by explosion,  $177\text{CO}_2 + 143\text{H}_2\text{O} + 81\text{N}_2$ . The same authority gives 1,535 calories as the heat liberated by the explosion of 1 kilo. of blasting gelatin, and 8,950 litres as the corrected volume of gases liberated. Taking the explosive value of kieselguhr dynamite as unity, that of blasting gelatin is 1.4, and that of nitro-glycerol 1.45.

—B. B.

**Metallic Derivatives of Acetylene. Mercuric Acetylide.**  
M. Travers and R. T. Plimpton. *Proc. Chem. Soc.* 1894 [133], 32—33.

HERTELOR (*Ann. Chim. Phys.* [4], 9, 386), by passing acetylene through a solution of mercuric iodide in potassium iodide made alkaline with ammonia, obtained an explosive mercuric acetylide, of which, however, he seems to have made no analysis. The authors have prepared this substance in various ways, and studied its composition and properties. They have prepared it by the action of acetylene—(1) on freshly precipitated mercuric oxide; (2) on solutions of mercuric cyanide mixed with ammonia, or better, with ammonia and cupric sulphate or zinc chloride; (3) on solutions of mercuric acetate or sulphate with ammonia, when a part only of the mercury is precipitated; (4) on mercuric oxide dissolved by the aid of ammonia and ammonium carbonate. The latter method is the most convenient. The heavy white powder which separates is well washed and dried at 100°. Analysis indicated the formula  $3C_2HgH_2O$ . The substance could not be obtained free from water even after long drying at 100°. Mercuric acetylide belongs to the same class of bodies as the silver and copper acetylides, and it is explosive. It resembles the allylene derivatives obtained by Katscheroff from solutions of mercuric chloride and acetate (*Ber.* 17, 13). Mercuric acetylide detonates violently when suddenly heated or struck sharply; it can, however, be handled with safety even when dry.

## XXIII.—ANALYTICAL CHEMISTRY.

### APPARATUS, ETC.

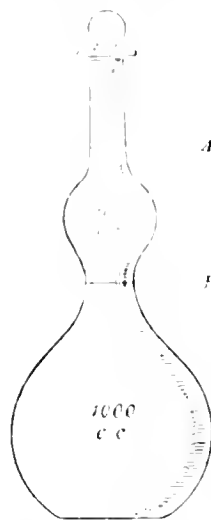
**A Modification of the Litre Flask.** W. B. Giles.  
*Chem. News*, 69, 99—100.

In the preparation of solutions intended for use in volumetric analysis, it is of primary importance that the exact titre—or strength—be established before they are applied to the estimation of undetermined factors. In many cases this is effected by taking an exact weight of the reagent and dissolving it so that the solution may amount to exactly 1 litre in volume at the standard temperature. When this method is applied to such materials as potassium bichromate, silver nitrate, and similar reagents which are readily obtained in a state of almost chemical purity, it answers fairly well, although, to an accurate worker, it is preferable even in such cases to determine their actual reacting power by direct experiment. When, however, the nature of the reagent employed renders it difficult or impossible to obtain an exact quantity by direct weighing, a correction of the resulting standard solution becomes an absolute necessity, as is well known.

In working with such bodies as, e.g., sulphuric acid, thiocyanate of ammonium, or uranium nitrate, if normal solutions are desired, they must in the first instance be prepared too strong; and, after their exact working strength has been established by experiment, they must be reduced by the addition of more fluid to the normal strength. With the ordinary litre flask this involves some difficulties. Assuming that the solution has been made up to the mark, a certain portion must be withdrawn to determine the titre, leaving an odd number of cc. in the flask to be corrected after trial, even if a portion is poured into another smaller graduated measure, and some calculation is needed to determine the amount of fluid to be added.

The author has devised the modification shown in the figure. The measuring flask has an additional bulb blown upon its neck. Up to the ring marked B it holds exactly 1,000 cc. Above this is the bulb with another mark, A. Between these two marks the contents are 100 cc. To make any normal solution with these flasks, one-tenth more of the reagent is weighed out than would be used

with an ordinary flask, and the vessel is filled to mark A, always taking care that the resulting solution shall be *rather too strong*. After thorough mixing, enough is poured into a clean vessel to bring the fluid to mark B; thus there is obtained 100 cc. of solution for ascertaining



the exact titre, and exactly 1 litre is left in the measuring vessel for correction. In this way the calculation is much simplified, and the additional space afforded by the bulb facilitates the ready mixing of the fluid.

Some 2,000-cc. flasks made on the same principle have also proved useful. In this case the bulb holds 200 cc.

**Sampling by Quartation.** P. W. Shimer and S. K. Reifsnyder. *J. Amer. Chem. Soc.* 1893, 15, 260—265.

EXPERIMENTS as to the possibility of perfect sampling of a mixture of dissimilar substances by the methods commonly in use were made by mixing 500 grms. of lead shot, just large enough to be retained by a 10-mesh sieve, 500 grms. of magnetic iron ore of a size between a 10- and 20-mesh sieve, and 500 grms. of crushed glass, that had passed a 20-mesh sieve, and from which the finest powder had been removed by washing.

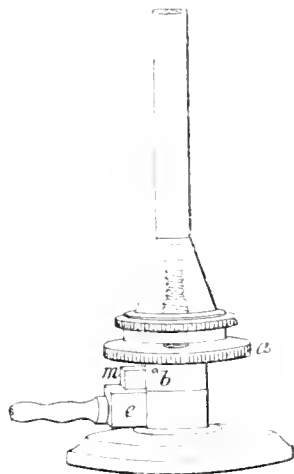
It was first demonstrated that sifting this mixture sufficed to recover 33½ per cent. (the amount used in making the mixture) of each ingredient. The constituents were again mixed, repeatedly quartated, and each diminishing fraction mechanically analysed by sifting, with the result that the sampling became increasingly inaccurate as the quartation was continued. Sampling moist was found to be slightly better than sampling dry, and flattening out the sample by pressure with a board, preferable to spreading it out by means of a spatula. Similar results were obtained with a crushed iron ore, which had been divided into four sizes by sifting. The method of halving the ore by allowing a scoopful to fall over a thin partition was tested, and found to be inferior to the more usual process of quartation. The authors arrive at the following conclusions:—

- (1.) It is more accurate to sample moist than dry.
- (2.) It is better to mix dry, than moisten and mix again before quartation.
- (3.) It is better to flatten the sample by pressure than to spread it with a spatula.—B. B.

**An Improved Laboratory Burner.** N. Teclu. *J. prakt. Chem.* 47, 535—536.

THE author has improved upon his former design for a laboratory burner so that the regulation of the air and gas occurs simultaneously. The accompanying figure represents his improved burner.

The plate *a*, for regulating the supply of air, moves vertically on a screw cut on the outside of the gas supply tube. Connected with this plate is a ring *b*, which rotates on the cylindrical foot of the burner. The ring is provided with an additional block *m*, which is cylindrical in the lower part and fits air-tight and obliquely into the tube which supplies the gas. There is a horizontal hole bored through the cylindrical part of the block *m*, and according to the position of the plate *a*, this hole will either wholly or partially

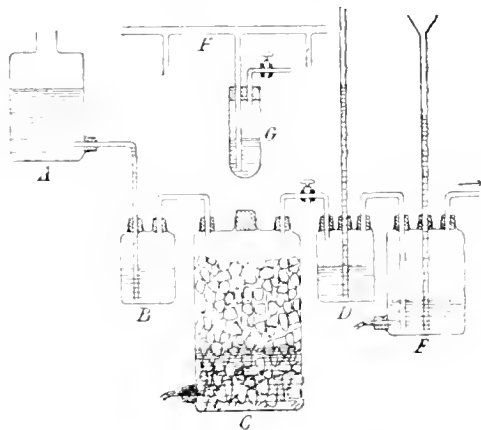


coincide with the opening in the gas-supply tube, so that the gas may have free or only a limited access to the burner.

This arrangement enables the gas to be burnt with the hottest possible flame of any size within the given limits. The character of the flame itself may be varied by means of the screw *c*, which when turned in one direction approaches, or in the other direction recedes, from the plate *a*, and regulates the supply of air.—J. S.

*Sulphuretted Hydrogen Apparatus.* F. W. Küster.  
J. prakt. Chem. 48, 595—598.

A, B, and C, in the figure are three bottles of 5, 3, and 10 litres capacity respectively, C being filled with ferrous sulphide in as large pieces as possible, whilst A and B are charged with a mixture of 2 vols. of crude hydrochloric acid and 1 vol. of water. The tube connecting A and B is drawn out to a fine jet at both ends, and its length determines the pressure obtainable from the apparatus. D



contains water, and serves both as a washing and safety bottle. E is charged with distilled water which becomes saturated with sulphuretted hydrogen, so that a supply of

sulphuretted hydrogen water can always be got from the tap at the bottom of the bottle. When gas is being drawn from the apparatus, the acid flows from A into B and thence, in drops or in a fine stream, into C. By the time the acid has trickled down to the bottom of the bottle C, it is completely exhausted, and the ferrous chloride solution may be drawn off by means of the tap when a large quantity has collected. When the tap between C and D is closed, the acid in B is partially forced back into A and the action ceases. The only attention the apparatus requires is when A requires filling with fresh acid, and C freeing from ferrous chloride. The small cut shows the method of distribution of the gas. One apparatus has been 10 years in use in the Marburg laboratory, without repair.—J. W.

*An Automatic Extractor for Phosphates.* W. D. Horne.  
J. Amer. Chem. Soc. 1893, 15, 270—272.

This apparatus is designed for the extraction of soluble phosphoric acid in the analysis of commercial fertilisers. The conventional plan of washing 2 grms. of the sample with repeated portions of 10 cc. of water until 250 cc. have run through is tedious to carry out by hand.

The apparatus consists of an inverted wide-mouthed bottle of 250 cc. capacity closed by a perforated cork carrying two glass tubes, one of which projects downwards 3—4 cm. and tapers to a fairly fine point. The other is bent upwards to the top of the bottle. By means of this device water under a constant head flows out of the shorter of the two tubes, air entering by the longer. The rate of flow can be adjusted to the capacity and permeability of the filter supplied by the bottle, by partially plugging the air inlet. The lower part of the apparatus consists of a syphon of variable length of limb, so that (1) the continuous flow from the bottle is converted into an intermittent discharge; and (2), the quantity of each discharge is suited to the capacity of the filter to be washed. The variable portion of the syphon may conveniently consist of a piece of rubber tube with an obliquely cut end to ensure its emptying at each discharge. The author states that it has been experimentally shown that the results obtained by this method of mechanical washing are identical with those arrived at when washing by hand is adopted.—B. B.

*New Tube for Polarimetric Observations.* F. Hann, Zeits. Zuckerind. Böhm. 18, 14.

The tube described has its inner portion widened at both extremities, the object of this being that if by chance air bubbles remain in the solution, they are not seen when the tube is placed in the apparatus, and do not, therefore, interfere with the observation.—A. R. L.

**INORGANIC CHEMISTRY.—**  
**QUANTITATIVE.**

*The Characteristics of Portland Cement.* R. Fresenius and W. Fresenius. Zeits. Anal. Chem. 32, 433—445.

See under IX., page 252.

*The Chemical and Physical Examination of Portland Cement.* T. B. Stillman. J. Amer. Chem. Soc. 1893, 15, 181—190.

The author attributes the fact that American cement is held in less esteem than that of German manufacture to the differences of the methods of testing in use for the two products. He gives the usual limits for the composition of Portland cement, and mentions that the main cementitious constituent is tricalcium silicate,  $3\text{CaSiO}_2$ . The following table shows the scheme of analysis adopted by the author for the analysis of Portland cement.





With regard to the mechanical tests, those of chief importance are the determination of fineness, the ascertainment of soundness, and the testing of tensile strength. The standard sieve in use in America appears to be that having 100 meshes per linear inch, made of No. 10 wire, Stubbs's wire gauge. The test for soundness is that usual in this country, namely, the observation of the behaviour of thin pans of neat cement kept under water and examined for cracks or other symptoms of internal strains. The tensile strength is determined on a mixture of one part by weight of cement to three of sand, and the mould is filled without ramming. Five briquettes are used for each test and only those breaking across their smallest section are included in the return of the test.

The testing machines in use in America are represented by two types. One is that known as the Fairbanks and is operated by running shot into the bucket at the end of the upper lever of the apparatus until the specimen breaks, when a valve automatically cuts off the supply of shot from the hopper above the bucket.

The bucket with its contents of shot is weighed on the machine itself by removing it from its position at the end of the upper lever, replacing it by a counterpoise, and suspending it below the large balance weight at the left of the instrument and counterbalancing it by the use of the steel-yard graduated on the upper lever.

The second testing machine is a single lever instrument known as the Riehle and is actuated by a handwheel, not being automatic like the Fairbanks. The upper lever is merely an indicator by means of which small movements of the beam may be detected.—B. B.

#### *The Electrolytic Separation of the Metals of the Second Group.* S. C. Schmecker. J. Amer. Chem. Soc. 1893, 4, 195—206.

THE author has endeavoured to find a general method for the separation of the metals forming acid oxides from those forming basic oxides of the second group. The lines worked upon were generally the use of solutions containing alkaline tartrates, and the device of keeping the arsenic (when that element was dealt with) in its higher state of oxidation.

*Copper from Tin.*—0.1087 gm. of tin was dissolved in hydrochloric acid, oxidised with bromine, evaporated to dryness, taken up with caustic soda, neutralised with tartaric acid and treated with 4 grms. of tartaric acid and then with 10 cc. of ammonia (sp. gr. 0.932). Ten cc. of a solution of copper sulphate containing 0.0999 gm. of copper were added, the whole transferred to a platinum dish, diluted to 175 cc. and electrolysed for 17 hours by a current yielding 0.4 cc. of electrolytic gas per minute. The copper found weighed 0.1001 gm.

*Copper from Arsenic, Tin, and Antimony.*—A solution was prepared containing 8 grms. of tartaric acid and 30 cc. of ammonia, and to this portions of solutions of arsenic antimony and tin, all in their highest state of oxidation and containing approximately 0.1 gm. of the metal apiece, were added. The quantity of copper added to this mixed solution was 0.1016 gm. and on diluting to 175 cc. and electrolysing for five hours with a current generating 0.8 cc. of gas per minute, the quantity of copper found was 0.1019 gm.

*Cadmium from Antimony.*—Similar solutions were used and the condition of experiment were also similar to those mentioned above. The quantity of cadmium taken was 0.0916 gm. and that found 0.0920 gm. The precipitated metal was in a somewhat spongy condition and needed care in washing.

*Cadmium from Tin.*—The method and results were similar in all respects to those of the previous separation. The use of a weak current (0.2 cc. of electrolytic gas per minute) favoured the obtaining of the cadmium in a compact state. The separation of cadmium from arsenic and from a mixed solution of arsenic antimony and tin, also succeeded, the prescription used previously being observed.

*Bismuth from Arsenic, Tin and Antimony.*—A solution of bismuth nitrate containing 0.0518 grms. of the metal was used in each experiment, and the use of tartaric acid and ammonia, as in former cases, resorted to. The separations were good and the bismuth was deposited in a compact form when a weak current was employed. Owing to the comparative ease with which the bismuth oxidised, rapid and careful washing was necessary and the use of alcohol after the first washing with water was requisite.

*Mercury from Tin, Antimony and Arsenic.*—Some difficulty was experienced in keeping the mercury in solution in the presence of much free ammonia, but it was overcome by adding the mercuric chloride used in the experiments to the tartaric acid solution before the addition of the ammonia, or, if the reverse order was chosen, by having the solution dilute and adding the mercuric solution with constant stirring. In separating mercury from any one of the three metals a deposit that is easy to wash is obtained, but when all three are present a portion of the mercury is finely divided, and has to be washed with great caution without decanting off the wash water closely. This makes the evaporation of the residual water by the heat of the hand very tedious, but it cannot well be avoided.—B. B.

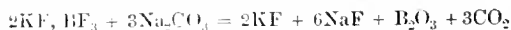
#### *Investigation as to the Value of Borax as the Basis of Acidimetry.* T. Salzer. Zeits. anal. Chem. 32, 529—537. (Compare this Journal, 1893, 466.)

TIN difficulties which attended the use of borax solutions in acidimetry (see Mohr's *Lehrbuch der Titrimethode*, vol. 2, p. 102, 1st ed.) have now been overcome, and the author has made a series of test experiments, which show that the most suitable basis of volumetric analysis is a decinormal solution of borax. The chief advantages in using this standard solution are, that it is easily prepared pure, and that it is not so liable to absorb carbon dioxide from the air. Oxalic acid is preferably used as the standard acid.

The author formerly recommended the use of methyl orange as indicator when titrating with borax solutions. On closer investigation, however, he finds that methyl orange is advantageously employed only when estimating the strong mineral acids; in other cases it is much better to use litmus. Citric acid seems to form a compound with boric acid, and is therefore best estimated in the usual way with caustic potash and phenolphthalein.—J. S.

#### *The Volumetric Determination of Potassium Borofluoride.* F. Stolba. Chem. Zeit. Rep. 17, 1893, 250.

FOURNIER on the well-known reaction between alkaline carbonates and borofluorides at a red heat:—



the author has worked out a process for the acidimetric estimation of these compounds. The dried borofluoride is weighed out into a deep platinum crucible, and a known weight, at least four times that of the former, of sodium bicarbonate added, and the whole well mixed up. The crucible is heated gently until the carbonic acid has passed off, then ignited until the mass is in a state of complete fusion. It is then put into a large platinum basin, water added, boiled, and titrated with normal acid added a few drops at a time, using litmus or brazil-wood as an indicator. If the acid be added in large quantities at once, the original borofluoride will be regenerated. The amount of acid required for the sodium bicarbonate being known, the difference between that and the quantity required on titration gives the amount of borofluoride, each cc. being equivalent to 0.042037 gm. of the potassium salt. Results quoted by the author are satisfactory.—F. H. L.

#### *The Technical Estimation of Lead.* A. H. Low. J. Amer. Chem. Soc. 1893, 15, 548—552.

THE ordinary dry assay for lead is frequently erroneous, and a quick wet method for technical use would generally be preferable. Alexander's molybdate method is the shortest hitherto proposed, but the presence of lime causes

inaccuracies. The ferrocyanide process devised by the author is as quick as the molybdate method if the quantity of lime is small enough to be neglected, but a reliable method should be capable of dealing with all classes of ores, and the two following schemes of analysis satisfy this condition fairly well.

(1.) *Permanganate method.*—One gram. of ore is treated with 10 cc. of aqua regia in a 250 cc. flask, and after the attack is complete 10 cc. of strong sulphuric acid are added and heating continued over a naked flame until fumes of sulphuric acid are evolved. The contents of the flask is cooled, diluted with 50 cc. of water, heated to boiling, filtered, washed with dilute sulphuric acid (1:10), and rinsed into a beaker with 50 cc. of boiling ammonium chloride solution made by diluting a cold saturated solution with an equal bulk of water. The lead sulphate dissolves, and its solution is decanted into the original flask. The residue in the beaker, which may contain lead, is boiled with a few cc. of strong caustic soda, and to the solution dilute sulphuric acid is added, precipitating the lead as sulphate, which is then rinsed into the beaker containing the main part of the lead. The solution of the whole of the lead sulphate in the ammonium chloride at this stage of the process is not obligatory. Three pieces of sheet aluminium, each  $\frac{1}{16}$  in. thick by  $\frac{3}{4}$  in. wide and  $1\frac{1}{4}$  in. long, are placed in the solution, which is heated to boiling for five minutes. To ensure precipitation of the whole of the lead as metal the bulk of the solution should be kept small. The flask is filled with tap water, and the lead washed by decantation and dissolved by treatment with 5 cc. of a mixture of 1 part of strong nitric acid and 2 of distilled water. The solution is separated from the residual aluminium, made slightly alkaline with strong caustic soda solution, using phenolphthalein as an indicator, treated with 10 cc. of a cold saturated solution of oxalic acid, cooled, and filtered. The lead oxalate is titrated with potassium permanganate in presence of sulphuric acid. The permanganate solution is standardised by the use of iron, and the figure thus obtained, multiplied by the empirical factor, 1.888, which is best checked by each operator by carrying out the process on 0.3–0.4 gram. of pure lead reduced from the acetate. None of the ordinary constituents of lead ores interferes with the results of this method, which are said to be accurate to 0.1–0.2 per cent.

(2.) *Ferrocyanide method.*—The ore is attacked, converted into sulphates, and filtered as described under the permanganate method. The bulk of the lead sulphate is washed with 50 cc. of cold water into the original flask, and 10 cc. of cold saturated solution of ammonium carbonate added thereto. The mixture is heated to boiling, cooled, filtered through the same filter, the precipitate (of carbonate) washed, boiled with 5 cc. of glacial acetic acid, and 25 cc. of distilled water, the solution diluted with 100 cc. of cold distilled water, and titrated with potassium ferrocyanide, uranium acetate being used as an indicator. The titration is best effected by separating a portion after solution from the main quantity, and adding it after the end point has been passed in titrating the main portion, finishing with more caution. A second reserve portion may be used in the same way. A deduction of two drops from the reading of the burette is necessary to allow for the ferrocyanide needed for the end reaction. The ferrocyanide solution contains about 10 grms. per litre, and is standardised on 0.3–0.4 gram. of lead reduced from the acetate and carried through the whole process. To provide against the influence of bismuth and antimony, the basic sulphates of which might interfere, 10 cc. of dilute sulphuric acid (1:10) and 2 grms. of Rochelle salt may be added to the original solution of the ore after evaporation with sulphuric acid. 40 cc. of water, instead of 50 cc. as mentioned in the prescription, are then added, and the process continued as already described. The only influence of lime is that due to the solvent action of calcium acetate on uranium ferrocyanide, but ores with as much as 30 per cent. CaO may be thus assayed, provided complete reaction between the calcium sulphate and ammonium carbonate be secured.

—B. B.

*An Examination of the Ordinary Methods for the Estimation of Carbon in Iron.* A. Ledebur, *Chem. Zeit. Rep.* 17, 1893, 259.

As the result of a long continued investigation into the merits of the different processes for the estimation of carbon in iron, the author concludes that Sarason's method is the best, the iron, when free from graphite, being previously dissolved in copper sulphate, while if free carbon is present this preliminary treatment is unnecessary. On fitting up a new apparatus, care must be taken that the gas issuing from the potash tubes is as dry as when entering them, and it is well to boil the decomposing flasks, and also the sulphuric acid thoroughly with chromic acid before taking them into use. For the combustion of the hydrocarbons, either a capillary platinum tube or a glass tube filled with copper oxide may be employed, preference being given to the latter when oxidising grey cast irons directly by means of chromic acid. To avoid the danger of the production of sulphurous acid, a combustion tube filled with pumice and lead chromate may be inserted in the apparatus. As a check, the chlorine method, or for wrought iron Eggertz' colour test, may be used. None of the volumetric methods yet devised can be recommended.

For the estimation of the graphite, 1 gram. of the iron is dissolved in 25 cc. of nitric acid (sp. gr. 1.18) kept cold, in an Erlenmeyer flask, the mixture is then boiled gently on the sand bath for an hour, filtered through an ignited asbestos filter, washed and dried at 120–130° C. (100° C. is not high enough). The graphite is then burnt by igniting it first in a stream of air, and afterwards, for at least an hour and a quarter, in a current of oxygen.—F. H. L.

*Estimation of Chromium in Chromium Steel and Chromium Iron Ore.* J. Spüller and S. Korman, *Chem. Zeit.* 17, 1893, 1360–1412.

This paper is in continuation of the investigations already published (this Journal, 1893, 867), on the volumetric estimation of chromium in ferrochrome. The same principle has been applied to the estimation of chromium in chromium steel. The fusion of chrome iron ore with a mixture of caustic soda and sodium peroxide has also been more carefully studied.

The chromium steel is dissolved in dilute sulphuric acid, evaporated to dryness and fused with caustic soda and sodium peroxide. The whole of the chromium is thus converted into sodium chromate. The mass is digested with water, and after removing any sodium manganate or ferrate with peroxide, and decomposing excess of peroxide with carbonic acid, the solution is made up to a certain measure, filtered, and the chromium estimated volumetrically by "Schwarz" method.

Two samples of chromium steel were analysed. The chromium was estimated, both volumetrically, by the author's method, and gravimetrically, by J. Blau's method. The results were as follows:—

	Cr. Volumetric.	Cr. Gravimetric.
	Per Cent.	Per Cent.
No. 1 .....	1.35–1.40	1.43
" 2 .....	1.08–1.10	1.10

The volumetric estimation takes only about 3½ hours, and the presence of Wolfram does not affect the working. Attempts were made to fuse the steel direct with caustic soda and peroxide, but the results were not satisfactory owing to the difficulty in bringing the steel to a very fine powder.

Further trials in the analysis of chrome iron ore have satisfied the authors as to the efficacy of their process.

By using pure caustic soda and sodium peroxide, the method can be applied to the detecting and estimating chromium in different iron ores.—H. M.

*Separation of the Stannous Chloride Method for Titration of Iron.* R. W. Mahon. Amer. Chem. J. **15**, 360; Jour. Amer. Chem. Soc. 1893, 396.

This modification depends upon the fact that when stannous chloride solution is added to a boiling solution of ferric chloride, strongly acid with hydrochloric acid, and containing also some mercuric and platinum chlorides, the ferric iron is first reduced to the ferrous state, and then the next drop of stannous chloride in excess causes the formation of a cloud of mercurous chloride, which becomes darkened and at once rendered perceptible by finely divided mercury and platinum, which are further precipitated. This mixture of mercuric chloride and platinum chloride can therefore be used as an indicator. It can be made of such strength that one litre contains 0.05 gram of metallic platinum and 34.0 grms. of mercuric chloride, and 15 cc. of this solution is a suitable quantity to use in a test.—H. S. P.

*Estimation of Barium in the presence of Calcium and Magnesium.* F. W. Mar. Amer. Jour. of Science, **43**, 321; Zeits. Anal. Chem. **32**, 470—171.

This separation depends upon the insolubility of barium chloride and the solubility of the calcium and magnesium chlorides in concentrated hydrochloric acid. One part of barium chloride according to the author, is soluble in 20,000 parts of concentrated hydrochloric acid at the ordinary temperature. The addition of ether decreases the solubility to 1 part in 120,000.

The chlorides of the above name alkaline earth metals are dissolved in as little boiling water as possible, 24 cc. concentrated hydrochloric acid added drop by drop, and, after cooling, 5 cc. of absolute ether are added, and the whole is well stirred. The precipitate is allowed to settle for a few minutes and it is then transferred to an asbestos filter in a Gooch crucible and washed with concentrated hydrochloric acid containing about 10 per cent. of ether. The precipitate should not be allowed to stand for more than a minute or two before being filtered, as on standing a small loss is found. The washed barium chloride is first dried at a low temperature and afterwards at from 150—200° C. and weighed. The method gives good results with mixtures of barium and calcium chlorides in all proportions, but when more than 3 grms. of magnesium chloride in 20 cc. of the solution are present a reprecipitation of the barium chloride is recommended.—H. S. P.

*Separation of Strontium from Calcium.* P. E. Browning. Amer. Jour. of Science, **43**, 50; Zeits. Anal. Chem. **32**, 468—469.

This method depends upon the solubility of calcium nitrate and insolubility of strontium nitrate in boiling amyl alcohol. The aqueous solution of mixed strontium and calcium nitrates is evaporated to dryness in a beaker of about 100 cc. capacity and the residue taken up with the least possible quantity of water; 30 cc. of amyl alcohol are then added and boiled for a few minutes, until the water has been completely expelled and the alcohol has attained its normal boiling point (125—130° C.). The liquid is then decanted and filtered through an asbestos filter in a Gooch crucible and the residual strontium nitrate is washed with small quantities of boiled amyl alcohol. The amyl alcohol for washing is delivered from a small wash bottle, to the mouth tube of which a small calcium chloride tube is attached containing a plug of cotton wool to prevent any of the calcium chloride falling into the alcohol. The residue is then warmed gently to expel all the alcohol, and the strontium nitrate again dissolved in a few drops of water, a drop or two of nitric acid added and again evaporated to dryness. It is then re-dissolved in a few drops of water, and again precipitated by addition of 30 cc. of amyl alcohol and treated as before, this second precipitation being necessary to remove traces of calcium that go down in the first precipitate. The precipitate of strontium nitrate is now collected on the filter, washed with amyl alcohol, dried at 150° C. and weighed. Strontium nitrate is not completely soluble in amyl alcohol, and the amount of correction

to be made for its solubility, depends of course upon the quantity of alcohol remaining after boiling the precipitate in it. As however only a few minutes boiling is needed to expel the water, the author always had about 25 cc. of alcohol remaining. This quantity was found to retain 0.001 gram of strontium in solution. For every 30 cc. of alcohol used therefore an addition of 0.001 gram of strontium must be made to the weight of precipitate found, and also from the weight of calcium sulphate found in the solution after separation of the strontium, 0.0035 gram must be subtracted for each 30 cc. of alcohol used.

The calcium in the two filtrates may be determined by precipitation with sulphuric acid and ethyl alcohol, or the filtrates may be evaporated to a small volume, and transferred to a weighed platinum dish and evaporated to dryness and ignited. Sulphuric acid is then added, the evaporation to dryness and ignition repeated and the calcium weighed as sulphate. The latter method gives the more exact results.—H. S. P.

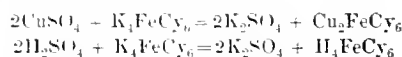
*Separation of Barium and Calcium.* P. E. Browning. Amer. Jour. of Science, **43**, 314; Zeits. Anal. Chem. **32**, 469—470.

This method depends upon the same principle as the same author's method for separating strontium and calcium (see preceding abstract), viz., the insolubility of barium nitrate in boiling amyl alcohol, whilst calcium nitrate is soluble in the same medium. The same process is carried out as already described in separating strontium and calcium, except that in the case of barium only one precipitation is needed and no correction requires to be made.—H. S. P.

*Estimation of Free Acid in Solutions of Per-Salts of the Heavy Metals.* F. Hoffmann. Chem. Zeit. **17**, 1318.

As an accurate method for estimating the free acid in copper sulphate solutions, in place of the unsatisfactory results of the ordinary methods, the author presents the following:—

By adding to a solution of copper sulphate containing free acid, yellow prussiate in excess of that required to precipitate the copper, the following reactions take place:—



So that an excess of ferrocyanide has no influence on the amount of free acid. But the solutions used must be so dilute that the ferrocyanic acid produced will not decompose.

A small quantity of copper solution, containing about 2—3 grms.  $\text{CuSO}_4$ , is precipitated by a small excess of yellow prussiate, made up to a certain volume, and allowed to settle. The clear solution is poured off, and titrated with standard alkali.

In presence of ferric salts, potassium sulphocyanide may be used to show the presence of free prussiate. On adding  $\text{KCNS}$  to the solution, iron sulphocyanide is formed, which dissolves in ether with a red colour. The copper is precipitated first by the prussiate, then the iron; so that as soon as the red colour of the ethereal solution disappears, all the copper and iron will have been precipitated.

The above method can be used for the per-salts of all the other heavy metals. In the case of lead, silver, zinc, &c., where the precipitates with prussiate are colourless, it is not necessary for the precipitate to settle, nor is an excess of prussiate harmful.

The author has used the above method for several years, and finds that it gives very accurate results in solutions of per-salts.

The effect of small quantities of proto-salts on the results is very slight, and may for technical purposes be overlooked.—H. M.

*Critical Studies of Methods for the Estimation of Sulphur.*

*II. L. Marchlewski. Zeits. Anal. Chem.* 1893, **32**, 463—411.

In continuation of a previous article (*Zeits. Anal. Chem.* 1892, **31**, 1—15; this Journal, 1893, 375—376), in which various direct and indirect methods of estimating sulphur, existing as sulphide were examined and criticised, the author now gives the results of his investigation of the gravimetric methods of Gröger (modified by Treadwell), Sauer (modified by Zulkowski), and the volumetric methods of Fohr and Klobukow. In the case of the two former methods, he used a pyrites as standard, which, according to an estimation made by Lunge's process, contained 53.28 per cent. of sulphur, and which, as pure  $\text{FeS}_2$  contains 53.33 per cent. of sulphur, may, therefore, be considered to be almost pure  $\text{FeS}_2$ . In the case of Fohr's method, he used the same Witkowitz slag-cement, containing 1.257 per cent. of sulphur, as he used in experimenting upon the methods noticed in his first article (*loc. cit.*).

*Method of Gröger* (*Ber. österreich. Chem. Gesellsch.* 1881, 2), modified by *Treadwell* (*Ber.* 24, 1937; this Journal, 1891, 951).—This method depends upon the conversion of the sulphides of iron existing in pyrites, into monosulphide, by heating the finely powdered substance with iron powder to redness, in an atmosphere of carbon dioxide. The monosulphide formed is then decomposed in a flask by hydrochloric acid, and the sulphuretted hydrogen that is evolved is conducted into a standardised solution of iodine, whereby a portion of the iodine is converted into hydriodic acid and sulphur is precipitated. The excess of iodine is then determined by titration, from which the amount of sulphur in the sample can be calculated.

The author finds that the results obtained when working with pyrites free from barium or lead sulphate are in close agreement with those given by Lunge's method (this Journal, 1889, 966; 1893, 292). But, as pointed out by Treadwell, the method is not applicable for the technical estimation of pyrites, because the insoluble sulphates (of barium, lead, &c.) existing in the pyrites are more or less acted upon, and reduced by ignition with iron powder. For the correct technical estimation of sulphur in pyrites, Lunge's method, or Treadwell's latest method (*Ber.* 1892, 2377; this Journal, 1893, 64), should be used.

The author also experimented to see if the sulphur in lead sulphate could be correctly estimated by the Gröger process, but the results obtained were not concordant, and were too low.

*Sauer's Method* (*Zeits. Anal. Chem.* 12, 32, 178), modified by *Zulkowski* (*Ber. oesterr. Chem. Gesellsch.* 1881; Dingler, 241).—Sauer's method is based upon the combustion of the sulphide in a stream of oxygen and the estimation of the sulphur oxides evolved. Various modifications of the process have been made (by Mixer, Zulkowski, Tschirikow, Jaansch, &c.), which chiefly differ from each other and the original in the nature of the absorbent used to retain the oxidised products of the combustion. Zulkowski employs a solution of caustic potash (free from sulphate) and potassium hypobromite, made by adding, drop by drop and whilst cooling, 100 grms. of bromine to an aqueous solution of 180 grms. of potassium hydrate. The solution is then made up to one litre, and 30 cc. suffice for estimating 0.5 grm. of sulphur. After the combustion of about 0.5 grm. of pyrites, using a combustion tube about 40 cm. long, the absorbing liquid is acidified with hydrochloric acid, boiled to expel bromine, and the sulphuric acid precipitated as barium sulphate. The author obtained by this process very satisfactory results with the sample of pyrites used as standard, but he considers it too tedious a method for adoption as a technical analytical process. Another drawback to it is that it is not practical to carry on several estimations at the same time, as can be done in processes not requiring a combustion furnace. It is useful for determining sulphur existing as sulphide in substances containing other non-volatile but soluble sulphur compounds, such as calcium sulphate. Zulkowski recommends it for estimating sulphur in spent oxide from gas purifiers. For this purpose he employs a

tube about 60 cm. long, drawn out to a fine tube at the end at which the connection with the absorption apparatus is made, and also somewhat narrowed at a point near this end. A layer of platinised asbestos, 20 to 25 cm. long, is first placed in the tube, and shaken down to the constricted part, then at a distance of 7—10 cm. is placed a porcelain boat containing about 0.4 grm. of the gas oxide. The platinised asbestos is first heated, and moist oxygen led through the tube at the same time. After this the gas oxide is heated, beginning with the end nearest the platinised asbestos, and continuing the heating gradually towards the end at which the oxygen enters. The author does not state whether he finds the process satisfactory or not in the case of gas oxide.

*Volumetric Methods of Fohr* (*Chem. tech. Zeitung*, 1887, 195), and *von Klobukow* (*Ber.* 18, 1861; this Journal, 1885, 613). Fohr's method is one of the many methods for determining sulphur existing as sulphides, depending upon the decomposition of the sulphides with an acid, and estimation of the sulphuretted hydrogen evolved. In this instance the sulphuretted hydrogen is absorbed by an ammoniacal solution of zinc, then the absorbing liquid together with the precipitated zinc sulphide is saturated directly with a dilute cold solution of ferric sulphate in sulphuric acid, and the reduced ferrous salt titrated with permanganate. The author did not obtain satisfactory results with this method.

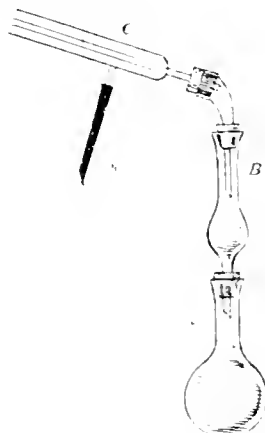
Neither did he find von Klobukow's method of any value. This was suggested for estimating sulphur in the higher sulphides, which when treated with acid, besides liberating sulphuretted hydrogen, precipitate sulphur, as also in the hyposulphites, sulphites, &c. The process attempts the conversion of all the elementary precipitated sulphur into sulphuretted hydrogen, by decomposing the substance mixed with zinc, by means of hydrochloric acid, and boiling to expel the sulphuretted hydrogen; but in the event of sulphur separating, the flask is cooled, allowed to stand and again boiled, and so on until the sulphur is removed. The author tested the method with a solution of polysulphide of potassium and with sodium sulphite, but in every case sulphur separated which could not be completely removed, and the results were too low.—H. S. P.

#### *Estimation of Sulphur in recovered Gas-purifying Material.* F. Meyer, *Chem. Zeit.* 17, 1319.

The method recommended by "Post" in his chemisch-technische analyse is very tedious. The following has been found much more convenient.

20—30 cc. carbon disulphide are introduced into a flask A, of about 150 cc., with which a small calcium chloride tube B is connected. In the bulb of this tube are placed 2—3 grms. of the purifying material, dried and wrapped in filter paper. The tube is then connected with a reflex condenser.

Instead of the calcium chloride tube B, a Soxhlet's extraction apparatus may be used.



When titred up in this way, the flask is heated on a water bath at about  $80^{\circ}\text{C}$ ., until the drops falling back into the flask appear quite colourless ( $\frac{1}{2}$  hour to 1 hour). The carbon disulphide is then distilled off, the last traces being removed by blowing air into the flask. The sulphur which remains, is oxidised with fuming nitric acid and a little potassium chlorate. The nitric acid is driven off with hydrochloric, and the sulphuric acid precipitated with barium chloride.—H. M.

### ORGANIC CHEMISTRY.—QUALITATIVE.

*Reactions and Constitution of Morphine.* G. N. Vis.  
J. prakt. Chem. **47** (1893) 584—587.

- (1.) The nitrogen-atom in morphine is methylated.
- (2.) Morphine is a tertiary base.
- (3.) By certain reactions morphine yields pyridine: hence it probably contains the pyridine-nucleus.
- (4.) Morphine derivatives are frequently known to become converted into those of the coal tar hydrocarbon, phenanthrene; if morphine does not therefore already contain the phenanthrene nucleus, it must at any rate be capable of producing it with great ease.
- (5.) When oxidised in a suitable manner morphine yields picric acid, and when fused with caustic alkali it yields protocatechic acid. Both reactions point to the existence of a benzene-ring in morphine. The second reaction indicates that morphine has a constitution analogous to that of papaverine, which also yields protocatechic acid when fused with alkali.
- (6.) Morphine contains two phenol hydroxyls, one of which behaves as a normal phenol hydroxyl whilst the other appears to behave as an alcohol hydroxyl. The third oxygen atom in morphine is of a neutral character.—D. E. J.

*The Detection of Eosin in Coloured Tomatoes.* K. P. McElroy and W. D. Bigelow. J. Amer. Chem. Soc. 1893, **4**, 191—195.

A SAMPLE of "tomato colour" made in Cincinnati was identified as an aqueous solution of bromeosin. Other articles of food have been known to be coloured with this and similar synthetic colouring matters, but hitherto the fact has not been recorded in the case of tomatoes. A method was elaborated for the detection of this colouring matter, the plan finally adopted being as follows. The solid portion of the tinned fruit is strained off and the juice is acidulated with hydrochloric acid and shaken out with ether. The ethereal solution is washed and shaken up with a dilute solution of caustic soda. Should this solution be strongly coloured it is acidified and extracted with ether, and the ether shaken out with caustic alkali as before, the operations being repeated until the colouring matter other than that sought has been eliminated. The fact that the natural colouring matter of tomatoes is not completely separated from its acid solution by ether nor removed wholly from ether by alkali allows of its separation from the artificial dyestuff by repeated treatment. The presence of a yellow-green fluorescence in the final alkali extract is indicative of bromeosin. As iodeosin does not give a fluorescence in alkaline solution its presence has to be determined by an additional test. The alkaline liquid resulting from the final extraction is gently warmed with sodium amalgam, and to it is then added a drop or two of very dilute solution of potassium permanganate. The presence of either iodeosin or bromeosin is indicated by the development of a green fluorescence.—B. B.

*On the Xanthine Bodies.* P. Balke. Journ. prakt. Chem. **47**, (1893), 537—567.

The author has investigated the effects produced by various reagents upon xanthine compounds, and the results are given in the following table:—

	Solubility in Water.	Action of Concentrated Caustic Soda Solution.	Action of Lead Acetate and Ammonia.	Action of Copper Acetate.	Fehling's Solution and $\text{NH}_4\text{OH}$ , $\text{HCl}$ .	Phosphomolybdic Acid.	Mercuric Chloride.	Silver Nitrate.	Picric Acid.	Weidel's Test. Evaporated with $\text{HNO}_3$ and $\text{Cl}$ water, and introduced into an Atmosphere of Ammonia.	Xanthine Reaction. Evaporated with $\text{HNO}_3$ .
1. Uric acid..	Almost insol.	$\text{C}_4\text{H}_3\text{N}_3\text{O}_6\text{H}_2\text{O}$ with difficulty sol.	..	..	..	..	..	..	..	..	..
2. Xanthine..	With difficulty	$\text{C}_4\text{H}_3\text{N}_3\text{O}_6\text{H}_2\text{O}$ with difficulty sol.	Bluish-green ppt.	White ppt. oxidising in air.	..	..	..	..	..	Yellow-red coloration.	Yellow residue. Purple-red colour in $\text{NaOH}$ .
3. Heteroxanth	"	White crystals	White crystalline precipitates.	"	..	White precipitates.	White precipitates.	White precipitates.	..	..	..
4. Paraxanth.	Easily soluble	"		"	..				Yellow ppt.	..	..
5. Theobromine	With difficulty	..		..	..				..	..	..
6. Caffeine...	"	..		..	White ppt. oxidising in air.				..	..	..
7. Guanine...	Insoluble	..		Blue to green ppt.	"				Yellow ppt.	..	Yellow. With $\text{NaOH}$ Purple-red.
8. Carnine...	Easily sol. in 1 pt $\text{H}_2\text{O}$ .	..		"	..				..	..	..
9. Hypoxanth	With difficulty	..		"	..				..	..	..
10. Adenine...	Soluble	..		"	..				..	..	..
11. Episarkine.	Insol. in cold $\text{H}_2\text{O}$ sol. with difficulty in hot.	..		..	..				..	..	..

—D. E. J.

*Tests of Purity of Rectified Wood Vinegar.* G. Buchner. (Chem. Zeit. **17**, 1319.)

THE German "Pharmacopœia," edition III., requires that 10 cc. rectified wood acetic acid decolorise 100 cc. permanganate solution (1 cc. = 0.1 grm.  $\text{KMnO}_4$ ).

The author has found that the reduction of the permanganate is due to creosote (guaiacol, cresol, &c.) contained in the acid. The other constituents, acetic acid, methyl alcohol, acetone, &c., decolorise permanganate solution only very slightly.

1 cc. of a 1 per cent. solution of creosote in acetic acid immediately decolorises 20 cc. of permanganate solution, so that what the Pharmacopœia demands is a minimum of 0.5 per cent. creosote in rectified wood vinegar. As the acid varies so much in composition, it is suggested that it would be more expedient to substitute an acid containing a fixed percentage of creosote.—H. M.

## ORGANIC CHEMISTRY.—QUANTITATIVE.

*Evaluation of Glue-stuffs.* F. Gautier. Zeits. Anal. Chem. **32**, 113—115.

THE value of glue-making materials, more especially of tanners' spetches, is not only dependent upon the absolute amount of glue-making substance, but also upon the relation existing between this and the non glue-making constituents of the raw material. These latter, which may be here regarded as impurities, are of two kinds,—the coarse impurities insoluble in water, such as sand, carbonate of lime, and fat; and those impurities that are soluble in water. In spetches therefore there are three things to be estimated; 1, the coarse impurities insoluble in water; 2, the raw glue, *i.e.*, the sum of the constituents soluble in water; and 3, the actual glue-yielding substance, *i.e.*, the pure glue.

If spetches be boiled with water, and the solution allowed to clear itself by standing, the insoluble matters, such as sand, carbonate of lime, &c., settle to the bottom, the fat floats on the surface, and the crude glue-making extract remains in solution. The latter when evaporated to dryness gives the raw glue. This can now be separated into matter precipitated from solution by tannin, *i.e.*, the pure glue-making constituents, and soluble impurities not precipitated by tannin. To estimate the pure glue-making constituents the amount of ash in the raw glue and in the tannin precipitate must also be determined. The process is as follows:—100 grms. of the sample are boiled in an evaporation dish with 1 litre of water and a few drops of caustic soda solution, until the extraction is complete. The solution is poured into a suitable vessel, made up to 2 litres, and allowed to settle for 10 hours. 20 cc. (= 1 grm. of the sample) of the clear extract are then evaporated to dryness, dried at  $105^\circ\text{C}$ ., and the dry residue weighed. It is then incinerated and the ash weighed. This gives the amount of ash-free raw glue. A further quantity of 20 cc. of the clear extract are then measured into a 100 cc. graduated flask, diluted with about 30 cc. of water, neutralised with acetic acid, and a solution of tannin added until a precipitate ceases to be formed; the liquid is then shaken, and its volume made up to 100 cc. It is then filtered on a dry filter, and a sufficient quantity of hide powder is added to the filtrate to precipitate the small excess of tannin that has been added, and the precipitate allowed to settle during ten hours. If the solution at the end of this time gives no indication of tannin in solution it is again filtered on a dry filter, and 50 cc. (= 0.5 grm. of the sample) are evaporated to dryness, weighed, and the ash determined by incinerating and weighing. This gives the amount of ash-free substance in the extract not precipitated by tannin, and by deducting this from the amount of ash-free raw glue, the amount of pure glue-making material is found. As an example:—

1. 20 cc. of extract (= 1 grm. of the sample) give—

0.760 grm. dry residue.

0.133 grm. ash.

0.627 grm. ash-free residue.

= 62.7 per cent. ash-free raw glue.

2. 20 cc. precipitated with tannin, made up to 100 cc., filtered, and excess of tannin removed and filtered. 50 cc. of filtrate (= 0.5 grm. of sample) give

0.054 grm. dry residue.

0.044 grm. ash.

0.010 grm. ash-free residue.

= 1.20 per cent. ash-free non glue-making substance.

Deducting (2) from (1): 62.7 — 1.2 = 58.5 per cent. pure glue; and the sample of spetch contains therefore—

58.5 per cent. pure glue-making material.

1.2 " non glue-making material.

13.3 " ash.

56.0 " of raw glue.

—H. S. P.

*Estimation of the Quantity of Juice contained in Beetroots.* A. Vivien. Bull. de l'Assoc. des Chim. **10**, 833.

See under XVI., page 264.

*Quantitative Colorimetric Estimation of Carbohydrates.*

E. Neitzel. Neue Zeits. Rubenzucker Ind. **32**, 13.

THE method which forms the subject of a German patent (72,982 of 1892), depends upon the reactions described by Ili (Chem. Zeit., 1885, **9**, 231; this Journal, 1887, 306, and 455), and by Molisch (Silzber. Wien. Akad., 6 Mai, 1886; this Journal, 1887, 149), namely, that when carbohydrates are heated alone, treated with acids, or dehydrating agents humus substances are formed which give rise to colorations when treated with aromatic compounds—phenols, alcohols, and amines.

The liquid in which the carbohydrate is dissolved or suspended is measured into a test tube and a known volume of an alcoholic solution of the aromatic compound added to it, and subsequently sulphuric acid, when on shaking the mixture together a coloration is developed varying in intensity with the amount of carbohydrate present. The coloration reaches its maximum only after the lapse of several minutes.

The point observed is the interval which elapses, when, after shaking together the mixture, a ray of light, which at first penetrates the solution is no longer able to do so. This is proportional to the percentage of carbohydrate. A table is constructed on tests carried out with solutions containing known amounts of sugar, and from it the percentage of sugar in a solution of unknown strength can be ascertained by again carrying out an experiment with it under the standard conditions.

The following precautions are to be observed:—The ray of light must be of constant intensity; the distance of the observer from the source of light must always be the same; the width of the test tubes employed must be equal.

The method serves for the estimation of sugar in drainage waters of the sugar factory, in urine, wine and beer.

—A. R. L.

*Rectified Wood Vinegar.* W. Brandes. Apoth. Zeit. **1893**, **8**, 534.

IT is required in Germany that 10 cc. of rectified wood vinegar shall immediately decolorise 100 cc. of potassium permanganate solution (1 in 1000).

The author finds that, owing to the simultaneous formation of manganese hydrate, it is difficult to decide as to this point, and recommends the addition of 10 cc. of dilute sulphuric acid to the permanganate solution before the addition of the wood acid.—J. G. W.

*The Oxygen Absorption, Acid and Iodine Numbers of various drying Oils.* L. Lettenmayer. Chem. Zeit. **17**, 1506.

See under XII., page 258.



*The Analysis of Lubricating Oils containing blown Rape Oil and blown Cotton Seed Oil.* T. B. Stillman. J. Amer. Chem. Soc. 1893, 15, 265-270.

The author states that a difficulty arises in the analysis of mixed lubricating oils when it is required that the specific gravity of the mineral oil that has been used should be ascertained, as the oil recovered from the ethereal extract of the products of saponification of the mixed oil is not necessarily identical in specific gravity with that which was originally taken. The determination of the nature of the fatty oil and the calculation of the specific gravity of the mineral oil from that of the mixture and that of the fatty oil are preferable for this purpose.

The large variation in the specific gravity of blown rape oil (0.930-0.960) makes the method useless when the lubricating mixed oil has been prepared with blown rape oil, but some information can be obtained by comparing the properties (notably the viscosity) of different mixtures of blown oil of known specific gravity and mineral oil, with those of the sample under examination.

The use of blown rape oil is said to be diminishing in favour of that of blown cotton seed oil—which is less liable than the unblown oil to develop acidity. The effect of blowing of the two oils on their iodine absorption is shown below—

	Iodine Absorption.
Cotton-seed oil.....	104-114
Blown cotton-seed oil.....	93-103
Rape oil.....	102-104
Blown rape oil.....	94-100

—B. B.

*Kapok Oil.* R. Henriques. Chem. Zeit. 17, 1283.

See under XII., page 258.

*Cotton Shavin and Maize Oil.* F. Hart. Chem. Zeit. 17, 1522.

See under XII., page 257.

*The Estimation of Paraffins.* R. Holand. Chem. Zeit. 17, 1833, 1473 and 1483-1484.

THE estimation of the amount of solid hydrocarbons or paraffins in oils is a problem of considerable importance, particularly to those engaged in the brown-coal tar trade. The paraffin is the most valuable constituent of this substance, and the various oils obtained on distilling it are cooled and pressed most carefully to extract every possible trace of solid matter. The first pressing takes place at the temperature of spring water—yielding hard paraffin, the second and third are conducted at the ordinary winter temperature, and at an artificially lowered one respectively—yielding softer products; and all the resulting press cakes and oils have to be tested to determine their commercial value.

The oldest process for this estimation is Grotowsky's modified by Engler and Bohm, and consists in dissolving the oil in the smallest possible amount of ether in the cold and precipitating the paraffins by means of absolute ethyl alcohol. The precipitate is filtered off, washed and more alcohol added repeatedly until oil begins to be thrown out, and the precipitates must be redissolved and precipitated to free them from oil. The author's experiments show that this process is not sufficiently exact, the paraffins still containing oil and *vice versa*, besides occupying too much time. Pawlewski and Filemonowicz working on petroleum and ozokerite products, precipitate the paraffins from their solutions by adding 10-20 times their volume of glacial acetic acid, washing the precipitate finally with ethyl alcohol of 75 Tr. This process is useless for brown coal tar as the softer paraffins are somewhat soluble in the liquid. Zalzieski (this Journal, 1888, 349) dissolves the oil to be tested in five times its quantity of amyl alcohol and precipitates the paraffins by the addition of the same amount of ethyl alcohol of 75 Tr., keeping the mixture

for several hours at a temperature not exceeding 4° C. with occasional agitation, filtering off the precipitate through a cold dry paper, and washing with a well-cooled mixture of two parts of amyl and one of ethyl alcohol. He then dissolves the paraffins in ether, evaporates and dries the residue at 125° C. For oils containing little paraffin, he uses ten times the amount of the alcohol mixture, and allows it to stand 10-12 hours. Modified somewhat to suit the brown coal tar products, this process has only the disadvantage attending the use of such a high temperature for drying, beside the unpleasantness of the operation.

While ethyl alcohol of not more than 98 per cent. strength only yields turbid solutions with brown coal tar oils, 99.5-100 per cent. alcohol dissolves in all proportions these oils when free from paraffin up to the specific gravity 0.850; from 0.850 to 0.880 they require five times, and from 0.880 upwards ten times their weight of solvent, and all the solutions may be cooled to the ordinary laboratory temperature without any separation taking place. On the other hand paraffin of 60° C. melting point when boiled with alcohol, separates out all but slight traces when the liquid is cooled to 25° C., and at 0° C. it is absolutely insoluble. Softer paraffins are more soluble as their melting point falls, but even that melting at 34° is absolutely insoluble in alcohol at 0°.

In his paper the author gives a number of experiments carried out on various known mixtures of paraffins and paraffin-free oils, by dissolving 20 grms. of the substance in 100 cc. of absolute ethyl alcohol, cooling the solution for two hours in melting ice and filtering through a double filter, which show very gratifying results; the extracted paraffins having within a degree or two the same melting point as the original, and the oils on distillation remaining liquid up to the very last when the distillate was allowed to fall on a piece of ice, proving that they were perfectly separated; a result which he has never been able to obtain by Zalzieski's method. The experiments being only conducted on brown coal tar products, the author expresses a hope that other investigators will take up the matter in other directions.—F. H. L.

*The Determination of Malic Acid in Wines, &c.* C. Mücke. Zeits. d. allgemeinen österr. Apotheker ver. 30, 289. Zeits. Anal. Chem. 32, 481.

THIS article is an expansion of the author's previous paper on the separation of malic acid from succinic, citric and tartaric acids (Zeits. Anal. Chem. 31, 465), and describes in what way his original method should be modified in order to the estimation of malic acid in wine. The extractive matters and inorganic acids present in wine, are in part precipitated along with the malic acid when wine is treated with lead acetate and alcohol. To obviate this the process is modified so as to partly remove by precipitation and partly to decompose these interfering substances. The tanning and colouring matters in the wine in particular are acted upon so that ultimately a very faintly coloured, or, as in the case of ciders, almost colourless filtrate is obtained. The extractive matters that then remain in solution give no precipitate with acetic acid and lead acetate in the presence of an excess of 80-85 per cent. alcohol. The process is as follows: 100 cc. of wine are evaporated on a water-bath to a few cubic centimeters volume, transferred to a flask, four or five cc. of twice normal sulphuric acid added and allowed to stand at the ordinary temperature for one hour. After this interval a mixture of 50 cc. of strong alcohol and 50 cc. of ether is gradually added during continuous shaking, and the liquid allowed to stand for from six to ten hours. In this time the matters insoluble in ether-alcohol will have well settled or subsided. The precipitate is filtered and washed with ether-alcohol, and to the filtrate 100 cc. of water is added, to prevent as far as possible the etherification of the acids present, and it is then distilled on the water bath to remove the greater part of the ether-alcohol, which is finally removed by distilling *in vacuo*. After the liquid free from alcohol has cooled to from 50 to 60°, the chlorine present is separated by addition of as small as possible an excess of a freshly prepared solution of silver sulphate. At this

temperature, in the presence of free sulphuric acid, only a slight reduction of silver takes place. After the precipitate of silver chloride has been filtered, the filtrate is neutralised with potassium carbonate and evaporated to small bulk. It is then treated exactly as described in the author's method for separating succinic, citric and tartaric acids from malic acid (*loc. cit.*). In the case of fruit wines containing no tartaric acid, it is only necessary to pass carbonic acid once through the filtrate obtained after the first treatment with barium hydrate, until it shows but a slight alkalinity. In analysing cider it is better to take not more than 50 cc. of the liquid, on account of the large quantity of malic acid contained in it.—H. S. P.

*Detection of the Higher Alcohols in Spirits of Wine.*  
Hardy. *Deutsch-Amerik. Apotheker-Zeit.* **13**, 73; *Zeits. Anal. Chem.* **32**, 477.

The spirits of wine is to be mixed with  $4\frac{1}{2}$  times its volume of a saturated solution of sodium chloride and  $\frac{1}{2}$  its volume of water, and is then several times shaken up with carbon bisulphide. The latter is then separated from the saline solution, and some concentrated sulphuric acid added to it. The carbon bisulphide and sulphuric acid are now separated and air is blown through the sulphuric acid to completely expel the carbon bisulphide, after which the sulphuric acid is mixed with an equal volume of glacial acetic acid and boiled for a quarter of an hour in conjunction with a reflux condenser. On now adding a volume of saturated salt solution equal to the original volume of spirits of wine taken, the acetic ethers of the butyl and amyl alcohol present separate out in oily drops. From the volume of these ethers collected and measured in a narrow graduated tube, the quantity of the originally present higher alcohols is found by multiplying the volume by 0.8.—H. S. P.

*Estimation of Succinic Acid formed in Fermentations.* A. Bau. *Archiv. f. Hygiene*, **14**, 225; *Zeits. Anal. Chem.* **32**, 482—486.

THE author has investigated the question, in what way the quantity of succinic acid formed in fermentation is affected by the conditions under which the fermentation is accomplished, and in order to do this he found it necessary to devise a suitable method for estimating succinic acid, especially in wine or other fermented liquids. He examined and found unsatisfactory the methods of Pasteur, Macagno, Kayser, and of Schmitt and Hiefe. As the result of his own experiments upon the solubility of succinic acid in various liquids and upon its behaviour with various reagents, he proposes the following method. 100 cc. of wine are evaporated to a syrup, repeatedly extracted on the water-bath with boiling alcohol, and each of the alcoholic extracts filtered after cooling. These extracts are mixed together and distilled, and the residue is dissolved in a little hot water and, if turbid after cooling, the solution is filtered. The clear filtrate is then treated with barium nitrate and with 3 to 4 times its volume of 90 per cent. alcohol and vigorously stirred. A precipitate containing the tartaric, malic and succinic acids forms, and after subsidence it is thrown on a filter and washed with 70 per cent. alcohol. It is now warmed for some time with a sufficient quantity of sodium carbonate solution, and filtered. The filtrate is neutralised with nitric acid, evaporated to a small bulk, and after neutralising with ammonia, it is saturated with a magnesia mixture containing magnesium nitrate, ammonium nitrate, and ammonia. After standing for from 3 to 4 hours, the precipitate due to tartaric acid is filtered off and the filtrate heated with potassium hydrate solution until all the ammonia has been expelled. The precipitate of magnesia is then filtered off, the filtrate exactly neutralised with nitric acid, diluted to from 100 to 150 cc. and silver nitrate solution (1:20) added which precipitates the succinic acid. The precipitate is collected on a tared filter, well washed, dried, and weighed. As a check it is burnt off in a porcelain crucible and the metallic silver remaining is weighed. The results calculated from the two weighings should agree.

Silver nitrate only precipitates tartaric acid from concentrated solutions and only precipitates malic acid from solutions containing more than 1 part in 800. In some cases (as when in the precipitations of the mixed acids with barium nitrate and alcohol, too much of the latter has been added and the precipitate has been allowed to stand too long for the purpose of subsiding before filtration) chlorine is present in the solution from which the silver succinate has to be separated, and when this is the case, 25 cc. of the solution must be evaporated to dryness, incinerated, and the chlorine determined in the aqueous solution of the ash, and the corresponding amount of silver chloride must then be subtracted from the weight of the precipitate of silver succinate.—H. S. P.

*A Study of the Methods of Estimating Glycerol.* E. Sohr. *Archiv. f. Hygiene*, **14**, 395; *Zeits. Anal. Chem.* **32**, 486—488.

THE methods of Diez, von Törning, Planchon, and the so-called "Reichs Methode," are particularly examined.

With regard to Diez's method (*Zeits. Anal. Chem.* **27**, 519; this Journal, 1888, 397, 696), the author finds two objections, viz., that the separated mixture of esters (consisting of glycerol tri and di-benzoate) which is obtained by treating the diluted solution of glycerol with benzoyl chloride and caustic soda solution cannot be washed free from benzoyl chloride, according to the originally prescribed procedure, because it is often a resinous or sticky mass which retains the benzoyl chloride; and, further, that the drying of the mixture of esters at 100° leads to error as it begins to decompose at this temperature. He proposes to overcome the first difficulty by washing the mixture of esters on the filter with water at 80° C. and a little caustic potash solution, until the smell of benzoyl chloride is gone, and finally to wash with water until the washings are quite neutral. In this way the mixture of esters becomes melted and then more readily is washed free from benzoyl chloride. Instead of drying and weighing the mixture of esters, he recommends saponifying it with half normal alcoholic solution of potash, using only a slight excess of the latter. After boiling a short time on the water-bath, the excess of potash is titrated with hydrochloric acid. He shows that substantially only the tribenzoate is formed, when the proportion of potash used attains almost exactly 3 molecules of potassium hydrate, 168 to 1 molecule of glycerol, 92.

Von Törning's method (*Zeits. Anal. Chem.* **28**, 363; this Journal, 1889, 124) he has modified to some slight extent in regard to the apparatus used. Instead of an air-bath of sheet iron, he uses a copper mantle packed with asbestos, which incloses the retort. An aperture is made in the cover to admit a thermometer. He prefers to use a retort without a tubulus, and when additions of water have to be made he allows the retort to cool, and after disconnecting the retort from the condenser adds the water by the neck. When using retorts with a tubulus and cork, he always found the cork became carbonated. In the distillate he estimates the glycerol by his modification of Diez's method, and obtains good results.

He also has modified Planchon's apparatus somewhat (*Zeits. Anal. Chem.* **28**, 356; this Journal, 1888, 779; 1889, 308, 480), chiefly with a view to preventing the water vapour passing over into the absorption apparatus. His arrangement is as follows:—The flask in which the glycerol is oxidised with potassium permanganate and sulphuric acid, is closed with a double-bored india-rubber cork, through one hole of which a tube reaching to the bottom of the flask passes, serving to allow of a current of air free from carbonic acid being drawn through the flask, whilst through the second hole of the cork is passed the condensation tube of a Liebig's condenser which is attached in an upwards inclined direction, so that the water vapour from the flask becoming condensed, flows back to the flask, whilst the carbonic acid evolved by the oxidation of the glycerol passes on to the drying and absorbing apparatus. This consists of first of all a U-tube in the bottom of which are placed a few pieces of calcium chloride, next a U-tube, one half filled with calcium chloride and the other half with pumice impregnated with copper

sulphate (which has been heated until white), after this a Liebig's potash apparatus containing 30 per cent. solution of potassium hydrate, which is followed by a small U-tube containing potassium hydrate, and finally a wash-bottle containing potassium hydrate solution which serves as a safety-flask preventing access of carbonic acid from the outside air. An intermediate flask is also introduced between this safety flask and the aspirator which is attached to the end of the train.

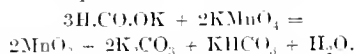
The operation is begun by slightly warming the liquid in the evolution flask; afterwards the heat is gradually increased until the evolution of carbon dioxide becomes small. The aspirator is then connected and a current of air is drawn through the apparatus. The results obtained and given by the author are very good.

The author then compares results he obtains in estimating glycerol in wine, mixtures of glycerol and sugar, &c., by the so-called "Reichs Methode" (*Zeits. Anal. Chem.* **23**, 392), and by von Töring's method, determining the glycerol in the distillate yielded by the latter process partly by Diez's process and partly by Planchon's, both modified by himself as above described. Confirming the results of other chemists, he finds that the glycerol separated by the "Reichs Methode" is far from pure, and that von Töring's method combined with Diez's gives by far the best results.

He recommends Planchon's method for estimating the amount of glycerol in aqueous solutions of glycerol, such as the "glycerine" of commerce, &c.—H. S. P.

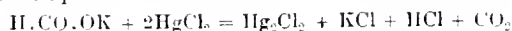
*The Determination of Formic Acid.* A. Lieben. *Monatsh. Chem.* 1893, **14**, 746—754.

THE volumetric determination of formic acid by oxidation with potassium permanganate cannot be carried out in acid solutions, since the oxidation is too slow in the cold, whilst heating volatilises a portion of the formic acid. The author's results go to show however that accurate results may be obtained in a solution of the acid containing an excess of sodium carbonate, the oxidation proceeding according to the equation:—



The accuracy of the results are not influenced by the quantity of sodium carbonate added. The titration is carried out by a permanganate solution, the strength of which has been fixed by means of iron. It is advantageous to warm the solution on the water-bath during the titration, as this hastens the reaction and causes the precipitated manganese dioxide to separate more readily, thus allowing the coloration, due to an excess of permanganate, to be more distinctly seen in the supernatant liquid. It is necessary to allow the solution to stand a little after the titration is completed to make sure that an excess of permanganate has been added. A number of analytical data show the accuracy of the method, which holds for salts of formic acid as well as for the free acid, but which is inapplicable in presence of other oxidisable substances.

The author also gives details of experiments on the value of Seala's method (*Gazzetta Chim.* 1890, **20**, 393) for the determination of formic acid by heating a neutralised solution of the acid with mercuric chloride, and weighing the mercurous chloride formed after drying it at 100° C. These experiments show that the process is only approximately accurate (within 1 to 2 per cent. on the formic acid present) if a large excess of mercuric chloride be employed and the mixture be heated on the water bath for at least 6 to 8 hours, instead of 2 hours, as recommended by Seala. Otherwise the results are much too low. The quantity of mercuric chloride added should be 50 times the weight of the formic acid, or 4 times the theoretical quantity required, according to the equation:—



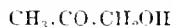
The addition of potassium butyrate or the heating of the mixture in a sealed tube to 102° C. does not increase the accuracy of the method.—C. A. K.

*The Quantitative Estimation of Acetone and Salicylic Acid in "Sal acetol."* H. Eckenroth and K. Kock. *Pharm. Zeit.* 1893, **38**, 593.

"SAL ACETOL" is the product of the combination to an ester of salicylic acid and acetone, its formula being  $\text{OHC}_6\text{H}_4\text{COOCH}_3\text{COCH}_3$ . It is saponified by caustic alkali with formation of acetone and alkaline salicylate. To examine it, 0.5 grm. is dissolved in water and titrated with decinormal potash, using phenolphthalein as an indicator, the flask being warmed; 26 cc. of alkali should be neutralised.—F. H. L.

*The Quantitative Estimation of Acetone and Salicylic Acid in "Sal acetol."* H. Helbing and F. W. Passmore. *Pharm. Zeit.* 1893, **38**, 621. Compare preceding abstract.

THE authors state that the preceding account of the decomposition of "sal acetol" on saponification is incorrect. The products are a salicylate, and acetone-alcohol—



Sal acetol is readily soluble in weak alkalis, and the colourless solution contains the hydrolysed products which reduce Fehling's solution in the cold. By gentle warming the alkaline liquid becomes brown in colour, and the characteristic smell caused by the action of alkalis on sugar is produced. This reaction is due neither to acetone nor salicylic acid, and as they do not reduce Fehling's liquid, these tests show the presence of the acetone-alcohol. Sal acetol should contain 71.16 per cent. of salicylic acid, and it should be examined for chlorine compounds, which may be introduced during the manufacture.—F. H. L.

*The Analytical Determination of Probably "Available" Mineral Plant Food in Soils.* Bernard Dyer. *Proc. Chem. Soc.* 1894, [134], 36.

PRINCIPALLY refers to experiments made by the author in endeavouring to decide what was the most useful solvent for extracting "available" (as distinguished from "total") mineral constituents of plant food from soil.

These experiments led him to seek to determine the average acidity of the root sap which plants have at their disposal as a natural solvent for such constituents. Determinations involved in the examination of about 100 plants, made in order to arrive at this, are recorded in detail.

As these determinations appeared to indicate the suitability as an analytical soil solvent of a 1 per cent. solution of citric acid—the same solution that, on grounds quite differently approached, had been suggested by Tollens and Stutzer as an agent for like purposes in testing manures—the author decided to try the effect of the solution on soils of known history and recorded fertility.

For the purposes of this trial, 22 separate plots of the experimental barley field at Rothamsted were sampled, with the kind permission and assistance of Sir John Lawes, and Sir J. H. Gilbert, and the samples were all subjected to separate investigation. A detailed account of this work is given, and its results discussed, the general conclusion being that the use of the suggested solution does give valuable indications of comparative ("mineral") soil fertility such as are not obtained in ordinary soil analysis.

Appended to the account of the work on soils is an account of some experiments on phosphatic manurial materials, comparing the solvent action of alkaline ammonium citrate and dilute citric acid, the result of which is to confirm the views of Tollens and Stutzer, to the effect that, although ammonium citrate may suffice to dissolve "reverted" or "retrograde" phosphate, it does not suffice to indicate "available" phosphate, and that its use ought to be superseded by that of a weak solution of citric acid.

# ANALYTICAL AND SCIENTIFIC NOTES.

*A Coal containing Vanadium.* A. Mourlot. *Comptes rend.* 1893, **117**, 546—548.

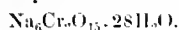
THE analysis of coal containing vanadium and which was obtained from Mendoza in the Argentine Republic has been given by Kyle (*Chem. News*, 1892). The author has examined a similar coal and found that it contains 0.21 per cent. calculated on the coal, and 38.5 per cent. calculated on the ash.—A. K. M.

*The Physiological action of Tellurous Acid.* T. Bokorny. *Chem. Zeit.* **17**, 1598—1599.

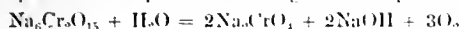
THE result of experiments on the toxic power of tellurous acid with respect to lower forms of life (*Spirogyres Zygnema*, *Diatomaceae* and *Infusoria*) is to show that the action is but faintly marked, or not at all observable. Selenious acid is somewhat more active, especially in solutions not neutralised; whilst sulphurous acid is still more energetic, especially when free.—C. R. A. W.

*Sodium Salt of Perchromic Acid.* J. Häussermann. *J. Prakt. Chem.* **48**, 70—72.

WHEN sodium peroxide is added to chromic hydroxide stirred up with water to a thin paste and kept cooled below 20° C., a violent reaction takes place, the hydroxide dissolving with formation of a yellowish brown liquid. On standing for some time in a cool place the solution deposits shining brownish-red crystals with the formula—



The substance may be heated with alkaline solutions without decomposition. Sulphuric acid at once gives the characteristic colour of perchromic acid, and hot water decomposes the compound according to the equation—



—J. W.

## New Books.

THE ALCHEMICAL ESSENCE AND THE CHEMICAL ELEMENT. An Episode in the Quest of the Unchanging. By M. M. PATRISON MURK. London: Longmans, Green, and Co. New York: 15, East 16th Street. 1894. 4s. 6d.

OCTAVO volume, bound in cloth, containing 94 pages of subject-matter. In these pages is portrayed the development of alchemy, and it is shown that all that alchemy did or could do, was to spread "a wondrous veil" over nature. This development is traced up to the period when chemistry commenced "hesitatingly to withdraw" that veil.

DIE SELBSTENTZÜNDUNG VON SCHIFFSLADUNGEN, BAUMWOLLE UND ANDEREN FASERSTOFFEN, STEINKOHLEN, HEUHAUFEN, TARAK, &c., SO WIE DEREN VERHÜTUNG. Von Dr. L. HÄPKE. Zweite, erweiterte Auflage, mit 10 Holzschnitten. Bremen: C. Ed. Müller's Verlagsbuchhandlung. 1893. London: H. Grevel and Co., 33, King Street, Covent Garden. 2 40 M., or about 2s. 5d.

A MONOGRAPH on the subject of Spontaneous Combustion. The work is stoutly bound in cloth, is of 8vo. size, and contains 105 pages. The subject matter is subdivided as follows:—Introduction. Origin and Causes of Spontaneous Combustion. I. Coals. II. Coal-dust, Briquettes, Soot, and Bone-black. III. Cotton. IV. Wool, Silk, Flax, Hemp, and Jute. V. Hay and Grain. VI. Various Substances. The work is illustrated by 10 wood-engravings.

UNIVERSAL EXPOSITION AT PARIS, 1889. REPORT ON CHEMICAL AND PHARMACEUTICAL PRODUCTS. By SPENCER R. NEWBERRY. Washington: Government Printing Office, 1893.

THE following branches are reported upon, the report covering some 140 pages:—I. Sulphur and its Derivatives. II. Sulphuric Acid. III. Alkali Manufacture. IV. General Chemical Products. V. Explosives. VI. Gelatin, Glue, Bone-black, and Phosphorus. VII. Soaps, Glycerin, and Stearin. VIII. India-rubber and Gutta Percha. IX. Pigments, Paints, and Varnishes. X. Pharmaceutical Products. XI. Products of Petroleum Distillation. XII. Products of Forestry. XIII. Agricultural Products not used as Food. XIV. Chemical Methods of Bleaching and Dyeing. XV. Leather and Skins. XVI. Fertilisers of Organic or Mineral Origin.

PRINCIPLES AND PRACTICE OF AGRICULTURAL ANALYSIS. A Manual for the Examination of Soils, Fertilisers, and Agricultural Products. For the Use of Analysts, Teachers and Students of Agricultural Chemistry. By HARVEY W. WILLEY, Chemist of the U.S. Department of Agriculture. Vol. I. No. 1. 25 cents. Easton, Pa., Chemical Publishing Co.

THIS is Part 1, of a series of monthly issues to follow, of the work under consideration, and is mainly confined, in the course of its 48 pages, to general definitions, and a descriptive account of the rocks, the micro-structure of which is illustrated in a series of admirably executed engravings from photographs.

## Trade Report.

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

PROPOSED NEW COMMERCIAL TREATY BETWEEN RUSSIA AND GERMANY.

*A—Russian Concessions to Germany.*

[Note.—Poud = 36 lbs. avoirdupois; Funt = 0.902 lbs. avoirdupois; Rouble = 3s. 2d.]

Tariff Classification.	Rates proposed to be levied under Russo-German Treaty.	Rates at present leviable under Russian Minimum Tariff.
Potato fecule .....	Poud Rbls. cop. 0.60	Poud Rbls. cop. 1.40
Starch of all kinds; dextrine. „	1.15 „	1.40
[Starch and dextrine imported in boxes, packets, or other small receptacles „	Poud gross 1.50	Poud gross 1.75
Cement of all kinds (natural or artificial Portland, Roman, cement made from scoria, and all others); bricks, slabs, and pipes of cement.	Poud 0.08	Poud 0.09
Baryta; sulphate of baryta ( <i>blanc fixe</i> ) and carbonate of baryta, artificial.	„ 0.80 „	1.00
Salts of chromic acid soluble in water; e.g., bichromate of potash, neutral chromate of potash, chromate of soda.	„ 2.15 „	2.65
Tannic acid (tannin) .....	„ 5.00 „	6.00
Blue coppers, (except anhydrous coppers, Salzbürg coppers (a mixture of sulphates of iron and copper), white coppers, or sulphate of zinc; chloride of zinc.	„ 0.80 „	1.00
Tartar emetic .....	„ 3.00 „	4.00

Tariff Classification.	Rates proposed to be levied under Russo-German Treaty.	Rates at present leviable under Russian Minimum Tariff.
	Rbbs. cop. Poud gross 1500	Rbbs. cop. Poud gross 1500
Chemical and pharmaceutical products not specially tariffed.	" 16'00	" 16'00
Preparatory medicines the importation of which is authorised in accordance with a special list.	" 0'12	" 0'15
Precipitated or washed chalk; chalk and talc, ground.	Poud 0'20	Poud 0'50
White lead and zinc white.	" 0'35	" 0'40
Colours with base of copper (except verdigris) or of arsenic.	" 3'00	" 4'00
Verdigris (basic acetate of copper).	" 3'50	" 4'00
Painting substances (pigments) prepared with products from the distillation of coal tar; alizarine, extract of madder, lac of madder or alizarine; cochineal carmine, carmine lac; indigo; turmeric and saffron extract in a dry state.	" 14'00	" 17'00
Colouring substances mixed with non-colouring materials such as clay and oil pay duty at the rate of 3 roubles per poud when the colouring substance does not exceed 10 per cent. of the total weight of the mixture.		
Fine colours for miniatures in china or porcelain cups or saucers, in tubes, or in tin capsules; Indian ink, liquid, in flasks.	" 5'00	" 5'00

## NORWAY.

## Tariff Changes.

Mr. A. S. MacGregor, Acting British Consul-General at Christiania, in a report to the Foreign Office, dated the 9th February, encloses the following list of alterations made by the Storting, on the 6th February, and coming immediately into operation in the customs duty and excise on spirits and malt:—

On spirits in bottle or jars, without any respect to strength, kr. 1.90 (2s. 1d.) per litre (formerly kr. 1.6 = 1s. 9d.)

On spirits in receptacles other than the above mentioned, kr. 2.03 (2s. 3d.) per litre of 100 per cent. alcoholic strength (formerly kr. 1.71 = 1s. 10½d.)

On spirits containing substances affecting the alcoholic strength, kr. 2.55 (2s. 10d.) per kilos, tare; as regards casks, 15 per cent. (formerly kr. 2.15 = 2s. 5d.)

On ether and naphtha, kr. 3.60 (4s.) per kilo. (formerly kr. 3.10 = 3s. 5½d.)

On vinegar-ether ("Eddike æter,") kr. 1.35 (1s. 6d.) per kilos. (formerly kr. 1.15 = 1s. 3½d.)

On "Ether spirituosus" and other ethers containing spirits, kr. 2.55 (2s. 10d.) per kilo. (formerly kr. 2.15 = 2s. 4½d.)

On perfumed waters and aromatic vinegar (including packing), kr. 2.35 (2s. 7½d.) per kilos. (formerly kr. 2.00 = 2s. 2½d.)

On varnishes and polish, kr. 2.35 (2s. 7½d.) per kilos, tare; metal packing 8 per cent. (formerly kr. 2.00 = 2s. 2½d.)

On methylated spirit—Spirit, 10 öre (1½d.) per litre; varnish, 10 öre (1½d.) per litre.

## FINLAND.

## The Duty on Sulphate of Potash and Soda.

Mr. J. Michell, Her Majesty's Consul at St. Petersburg, in a report to the Foreign Office, dated the 20th February,

states that, according to information supplied by Mr. C. J. Cooke, British Vice-Consul at Helsingfors, the Imperial Senate of Finland has established a duty of 1 mark 20 penni Finnish currency per 100 kilos, of sulphate of potash and soda (natrium sulphuric) imported into the Grand-Duchy, the above duty being leviable from the 16th February.

## EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

## NEW BELGIAN REGULATIONS AFFECTING THE ADULTERATION OF BEER.

Sir F. R. Plunkett, Her Majesty's Minister at Brussels, in a despatch to the Foreign Office, dated the 9th February, encloses copy of a royal decree published in the *Moniteur Belge* establishing new regulations for preventing the adulteration of beer, and for ensuring, as far as possible, that the beer should reach the consumer in good condition. The following is a translation of these regulations:—

Art. 1. It is strictly prohibited to use in the manufacture and preparation of beers products containing matters injurious to health, as also to sell, expose for sale, detain or transport for sale beers containing these matters, or beers otherwise adulterated.

For the application of the present regulations, such antiseptics as salicylic acid, sulphuric acid, or their saline preparations are considered as injurious to health. The presence of sulphuric acid is, however, permissible in beers when the proportion does not exceed 14 mgrms. per litre, probably originating in a careful disinfection of the casks.

Art. 2. Casks, bottles, or other receptacles, in which beers are contained for the wholesale trade, or in demi-johns, must bear in distinct characters the name and description, as well as the address of the maker or merchant.

Every factory or trade-mark, regularly deposited, will be sufficient for the purposes of the above regulations.

Art. 3. Taps, pipes, pumps, carbonic acid apparatus, &c., used for the serving of the beer must be kept perfectly clean.

The air admitted to the beers in casks for sale must come from a place free from any cause of contamination and well ventilated if possible from outside the houses.

Art. 4. It is prohibited to sell, expose for sale, detain or transport for sale *stortbier*, collected in the shops at the bottoms of glasses or on the tables and counters, unless these liquids are denaturalised in such a way that they cannot be utilised as beer, nor serve for mixing with beers, nor be employed in the manufacture of vinegar.

Art. 5. Infractions of the preceding regulations will be met with the penalties provided in the law of the 4th August 1890, without prejudice to the application of the penalties provided by the Penal Code.

## INTERNATIONAL BOOK AND PAPER INDUSTRIES EXHIBITION AT PARIS.

According to an announcement in the *Bulletin du Musée Commercial* for the 10th February, an international book and paper exhibition will be open in Paris at the Palais de l'Industrie from the 23rd July to the 23rd November next.

## THE FOREIGN TRADE OF GERMANY IN 1893.

The following is a comparative statement of the values of each class of imports into Germany in 1893 and 1892, as enumerated in the official returns:—

Category.	1893.	1892.
	Marks.	Marks.
Refuse (rags, &c.)	62,488,000	47,324,000
Lead and lead wares	5,717,000	4,322,000
Drugs and dyestuffs	243,445,000	235,433,000
Earths, ores, precious metals, and asbestos	298,296,000	317,752,000

THE FOREIGN TRADE OF GERMANY IN 1893—*cont.*

Category.	1893.	1892.
	Marks.	Marks.
Glass and glass wares.....	8,165,000	8,201,000
Hides and skins.....	117,543,000	126,620,000
Caoutchouc, gutta percha, and wares thereof.....	37,135,000	33,165,000
Copper and copper wares.....	56,931,000	52,198,000
Leather and leather wares.....	17,176,000	13,998,000
Candles.....	131,000	96,000
Oils, excluding petroleum and fatty substances.	158,775,000	175,067,000
Paper and paper wares.....	12,239,000	10,816,000
Petroleum.....	76,926,000	73,508,000
Soap and perfumery.....	2,331,000	2,287,000
Stone and stoneware.....	38,505,000	39,858,000
Coal, lignite, &c.....	98,737,000	95,101,000
Straw and bast goods.....	6,243,000	5,722,000
Tar, pitch, resin, and asphalt.....	30,856,000	27,927,000
Earthenware.....	5,772,000	5,343,000
Floorecloth, &c.....	992,000	1,160,000
Zinc and zinc wares.....	6,035,000	5,874,000
Tin and tin wares.....	20,066,000	17,633,000

The following statement shows the values of each category of exports from Germany in 1893 and 1892 :—

Category.	1893.	1892.
	Marks.	Marks.
Refuse (rags, &c.).....	12,558,000	10,311,000
Lead and lead wares.....	9,508,000	9,367,000
Drugs and dyestuffs.....	277,588,000	262,011,000
Earths, ores, precious metals, and asbestos	232,410,000	237,077,000
Glass and glass wares.....	43,552,000	37,752,000
Hides and skins.....	61,956,000	62,401,000
Caoutchouc, gutta percha, and wares thereof.....	28,868,000	26,953,000
Copper and copper wares.....	61,277,000	56,226,000
Leather and leather wares.....	117,768,000	110,116,000
Candles.....	799,000	800,000
Oils, excluding petroleum, and fatty substances.	30,615,000	23,518,000
Paper and paper wares.....	95,843,000	91,639,000
Petroleum.....	673,000	575,000
Soap and perfumery.....	10,338,000	9,284,000
Stone and stoneware.....	22,913,000	25,396,000
Coal, lignite, &c.....	142,720,000	131,212,000
Straw and bast goods.....	3,940,000	4,027,000
Tar, pitch, resin, and asphalt.....	6,828,000	7,585,000
Earthenware.....	37,831,000	35,205,000
Floorecloth, &c.....	1,103,000	928,000
Zinc and zinc wares.....	37,581,000	32,929,000
Tin and tin wares.....	4,669,000	4,157,000

## CHEMICALS AND DYES ON FREE LIST IN THE NETHERLANDS.

The United States Minister, under date of January 9, 1894, informs the Department that the Netherlands Govern-

ment has granted exemption from Customs duties to chemicals, dyes, and other material of a similar nature used as auxiliaries in operations in factories or trades, and from spirits, excise, and Customs duties to chemicals imported to that country destined for use in institutions for the instruction of natural sciences.

## GENERAL TRADE NOTES.

## THE SOLIDIFICATION OF PETROLEUM.

The *Moniteur Officiel du Commerce* for the 8th February gives the following as the formula of M. Maestraci, of the French navy, for obtaining briquettes of petroleum similar to those of coal.

With a litre of petroleum there is mixed 150 grams of triturated soap, 10 per cent. of resin, and 333 grams of caustic soda. This mixture is heated, care being taken to stir it; when solidification commences, which takes place at the end of about 10 minutes, observation is made as to the progress of the operation. If the mixture shows a tendency to overflow, some drops of soda are placed in the receptacle. The stirring is continued until solidification is complete.

The operation being finished, the material is poured out into moulds so as to make the briquettes, which are then placed from 10 to 15 minutes in a stove. All that remains is to let them cool; these briquettes can be used a few hours after manufacture.

To the three elements constituting the mixture, M. Maestraci recommends further the addition of 20 per cent. of wood shavings, and 20 per cent. of clay or sand which makes them firmer and more lasting.

Some trials in heating have recently been made at Marseilles on several tug-boats with these briquettes. An equal weight supplied three times the heat of ordinary coal briquettes, and there is no waste.

It is hoped with very simple modifications in the fire-places to arrive at still better results, the suppression of smoke and an increased production of heat, so that 1 kilog. of solidified petroleum will be equivalent to 4 kilogs. of coal.

## THE USE OF ALUMINIUM FOR LITHOGRAPHY.

The New York *Iron Age* for the 18th January publishes the following :—

Aluminium has been found adaptable to many purposes, and constant announcements are made of some new use to which the metal has been put. The latest is in lithography, in which process it is claimed to possess many advantages over the stone now used, while fulfilling all the requisite conditions. The only stone used for lithographing is found in Bavaria, and as the supply is diminishing its cost is increasing. Moreover, it is very brittle, and being rigid, can only be used on flat surfaces. Under a recently patented process, we learn that aluminium plates, weighing from 2 to 10 lb. and costing from 5 dols. to 10 dols., do the work equally well, and that, being flexible, the plates can be moulded into forms for cylinder presses. Aluminium plates are also coming into use in engraving in place of steel.

## RUSSIAN SUGAR PRODUCTION.

According to a recent report of the French Ambassador at St. Petersburg, extracts from which are given in the *Moniteur Officiel du Commerce* for the 25th January, the quantity of moist-sugar submitted for refining during the 1892-3 sugar season in Russia was 1.7 per cent. higher than in 1891-2, the total production of refined sugar having been 15,558,000 pounds (pound = 36 lbs. avoirdupois), or 4.6 per cent. more than in the preceding season. The quantity of sugar placed on the market by the refineries represented 15,077,000 pounds, as compared with 15,612,000 pounds in 1891-2, or a decrease of 535,000 pounds.

A new refinery commenced working in 1893 in the government of Livonia. The provinces of Kiew, Kharkow, and Podolia have supplied the best results, while on the other hand those of the provinces of Kherson and Moscow have been less satisfactory.

It is estimated that the beet crop in Russia in 1893 yielded 55 per cent. more than in 1892 (34,000,000 berkovets = berkovet = 360 lbs. avoirdupois—as compared with 21,800,000 berkovets in 1892). The average production per deciatine (1 deciatine = 2·7 acres) in 1893 was 111·32 berkovets against 81·15 in 1892. Although the crop was satisfactory as regards quantity, such was not the case as regards quality, which left much to be desired. On the average the density of the beets harvested in 1893 was only 17·52 in place of 17·92 in 1892; again the saccharin richness represented only 14·40 in place of 14·94 for 1892. It is only in the Polish provinces that the beets are of a superior quality to those harvested in 1892.

#### GERMAN SUGAR-BEET RETURNS.

The United States Consul at Magdeburg, in a recent report to his government, deals with the official statistics of the German beet-sugar industry and states as follows:—

The year 1892–93 has been of special importance to the sugar growers, as the system of taxation was changed by law of May 31st, 1891, to go into effect August 1st, 1892. Under the old law, beets were taxed 80 pfennigs per 100 kilogrammes, and an additional tax of 12 marks was levied on every 100 kilogrammes of sugar for domestic consumption. This tax of 80 pfennigs on the beets was refunded on exported sugars by means of a premium. The new law dropped the beet tax of 80 pfennigs entirely and substituted a tax of 18 marks per 100 kilogrammes on sugars for domestic consumption. The sugars which are exported are, as heretofore, exempted from this tax, but the export premium, which amounted under the old law to 8·50 marks per 100 kilogrammes, is reduced as follows:—

Description.	August 1st, 1892, to July 31st, 1895.	August 1st, 1895, to July 31st, 1897.
	Marks. 1·25	Marks. 1·00
On raw sugar of at least 90 per cent. and refined sugar of from 90 to 98 per cent. purity.	2·00	1·75
On candies and sugars in white, full, hard leaves, blocks, sticks, and crystals, and other sugars of at least 90 per cent. purity.	1·65	1·40
On all other hard sugars		

After July 31st, 1897, all bounties are to be discontinued. It is stated, however, in the press that the sugar growers and refiners are putting out feelers already to have this premium system continued after July 31st, 1897, claiming that they would be at too great a disadvantage with other countries, principally France and Belgium, if the premium was dropped entirely.

By the same law the duty on imported sugars was raised from 30 marks to 36 marks, excepting on such imported sugars as enter again into the manufacture here. Such sugars are credited with the consumption tax of 18 marks, thus paying only a net duty of 18 marks per 100 kilogrammes.

#### FRENCH MARGARINE PRODUCTION.

The *Monde Economique* for the 3rd February states that eight Bordeaux houses, including the manufacturers of seed oils, merchants, and ships' captains, have petitioned the members of the Chamber of Deputies in favour of margarine and against the proposals made to suppress it. It is, the petitioners state, perfectly wholesome, and by reason of its low price, being in fact from 30 to 40 per cent. less than butter, it can be purchased by the poorer classes who cannot afford butter. It is of importance to the Bordeaux trade, which imports ground-nuts from Senegambia for the works,

making a superior oil, these nuts being also used in other countries to give to the margarine-butter a nutty taste and elasticity; it is of interest also to agriculture, the milk and fat being used. Adulteration, it is said, is not practised by the large factories nor in the warehouses, but by certain firms who mix calf fat or similar substances with their butter in order to deceive purchasers.

#### INDIGO CULTIVATION IN THE PUNJAB.

It appears that an unusually large crop of indigo was obtained last year in the Punjab. The *Calcutta Englishman* in its issue of the 24th January states that the area cultivated is estimated at over 110,000 acres, an increase of 70 per cent. over the previous year, and the amount of dye obtained at 817,000 seers, an increase of about 60 per cent. Statistics of the Punjab indigo crop are available only for the past three years; and we have therefore no exact information concerning the normal out-turn. The area under indigo this year is unusually large, and the out-turn in dye is estimated at 15 annas.

#### THE BASIC STEEL PRODUCT IN 1893.

The growth of the production of steel by the Thomas-Gilchrist or basic process is shown by the returns just received for the *Mineral Industry*, giving the output for 1893 under the patents covering that process in the different iron-making countries of the world. The figures are in metric tons, and we compare them here with those for 1892:—

#### Total Production of Thomas-Gilchrist (Basic) Steel.

	1892.	1893.
Germany and Luxemburg.....	2,013,184	2,308,937
England.....	406,839	363,800
France.....	287,529	332,407
Austria-Hungary.....	288,522	320,063
United States.....	91,729	262,408
Russia.....	58,661	
Belgium.....	56,274	
Total.....	3,202,640	3,587,615

This shows an increase of 384,975 tons, or 10·7 per cent. in a year which has not been a favourable one for the industry, since it has been in almost all countries one in which the iron and steel industries have shown a temporary decline, or have at least been stationary. The total production of steel by this process since its first introduction is given in the *Mineral Industry*, Vol. 2, now in press.

The growth has been constant and marked, except in the especially unfavourable year of 1888. Germany continues to be the country where the process is most in favour, its especial adaptation to the phosphoric ores of some of the leading iron districts having given it a standing there and brought it into favour at an early date. In England there was a decrease of some 10·5 per cent. in the output, a contrast to the showing in all the other European countries.

The figures for the United States in 1893 are as yet only estimated. The increased use of basic steel in this country in the future is probable.

A feature of considerable economic importance in this process is the large proportion—about 36 per cent.—of phosphate of lime contained in the slag. Last year a production of some 863,000 tons of this slag was reported, and nearly all of that made at the European works was ground and sold for use as a fertiliser, forming a considerable item in the profits of those works.—*Engineering and Mining Journal*.



## UNITED STATES EXPORTS OF PARAFFIN WAX.

There is no article produced in the United States, nor even refined petroleum, which shows such an enormous increase in the amount exported as does paraffin wax. This article, which is of comparatively recent introduction, has reached an important place in the exports which was not even dreamed of 10 years ago. Indeed, the production at that time did not reach one-half of the amount exported during the last calendar year, when the total exports amounted to over 99,000,000 pounds. The rapid growth of the consumption of this article is due mainly to the fact that new uses have been found for the material, chief among which is its employment in the electrical industry. The exports of nearly all the products of petroleum have shown remarkable increase during the past few years, but even those of illuminating oil bear no comparison with the enormous expansion in the demand for paraffin wax. This is probably due to the fact that there is practically no competition, the Russian petroleum producing no wax, and the only other source being the Scotch shale wax, or paraffin scale, as it is known commercially, but production of which is limited, and does not cut much of a figure even in the requirements for home consumption, and it is not suitable for the more important uses to which paraffin wax is applied. To show the steady growth of the United States foreign trade in this product we append the following table, giving the amounts exported during each of the past 10 fiscal years. It will be seen from this table, taken from the official reports of the Bureau of Statistics at Washington, that the increase in the shipments has been to the United Kingdom, although the requirements of Germany show some increase. The amounts given are in pounds:—

Year.	To United Kingdom.	To Germany.	Total.
1884	16,230,251	771,092	17,001,343
1885	21,826,882	2,202,221	24,029,103
1886	22,798,003	1,402,427	24,200,430
1887	29,130,847	2,205,530	31,336,377
1888	33,815,143	1,576,354	35,391,497
1889	29,424,089	3,751,830	33,175,919
1890	42,343,246	4,823,073	47,166,319
1891	53,562,105	3,673,045	57,235,150
1892	54,816,457	6,491,864	61,308,321
1893	63,404,093	4,862,210	68,266,303
Total, 10 years ..	378,351,716	31,768,676	410,120,392

It appears that the increase in exports is still going on at the same ratio, as is evidenced by the large shipments during the last half of 1893. While the total exports during the fiscal year (ended June 30, 1893) amounted to 82,675,140 pounds, those during the 12 calendar months reached the enormous total of 99,061,034 pounds.—*Oil, Paint, and Drug Reporter*.

## IMPERIAL INSTITUTE RESEARCH COMMITTEE.

A committee of advice has been appointed in connection with the Scientific and practical Research Department of the Imperial Institute, and the members have adopted a scheme of action which it is hoped will result in making the public acquainted with the resources of various countries, to enable men connected with commerce and industry to compare the products of particular classes furnished by the several countries, and also to direct the attention of those practically interested in them to new or little-known natural products and to furnish trustworthy information as to their nature, properties, and value. The following are the members of the committee of advice and the subjects with which they will deal:—Professor Armstrong, F.R.S., Dr. W. H. Perkin, F.R.S., and Professor Meldola, F.R.S. (dyes,

tanning materials, and organic products); Dr. Hugo Müller, F.R.S. (mineralogy, botany, and fibres); Mr. C. F. Cross (fibres); Professor Dunstan, F.R.S. (pharmaceutical products and drugs); Mr. David Howard (alkaloids and drugs); Professor A. H. Church, F.R.S. (agriculture); Mr. Boyerton Redwood (petroleum and oils); Professor Fisher's-Auster, F.R.S. (metallurgy, fuel, and oils); Mr. Bennett Brough (metallurgy, mining, and minerals); Professor W. C. Unwin, F.R.S. (structural materials); Professor Hummel (dyes and tans); Professor W. A. Tilden, F.R.S. (gums and resins); Mr. W. H. Dering (oils, waxes, and fats); Mr. H. A. Miers (mineralogy); and Mr. Horace T. Brown, F.R.S. (food-grains). Sir F. Abel, F.R.S., is director, and Mr. Theodore Cooke, L.D., is secretary of the committee.—*Chemist and Druggist*.

## BOARD OF TRADE RETURNS

## SUMMARY OF IMPORTS.

Articles.	Month ending 28th February	
	1893.	1894.
	£	£
Metals.....	1,682,538	1,529,112
Chemicals and dyestuffs.....	751,160	721,477
Oils.....	615,811	509,931
Raw materials for non-textile industries.	2,093,116	2,438,774
Total value of all imports....	24,759,610	33,981,085

## SUMMARY OF EXPORTS.

Articles.	Month ending 28th February	
	1893.	1894.
	£	£
Metals (other than machinery) ....	2,301,823	2,117,208
Chemicals and medicines .....	735,736	646,158
Miscellaneous articles.....	2,256,704	2,032,185
Total value of all exports.....	17,093,369	17,679,149

## IMPORTS OF OILS FOR MONTH ENDING 28TH FEBRUARY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Cocoa-nut..... Cwt.	12,700	22,614	16,152	28,257
Olive .....	2,122	1,966	89,283	51,628
Palm .....	80,549	70,323	108,345	84,228
Petroleum .....	13,760,479	9,112,680	231,436	139,214
Seed .....	2,301	1,636	58,915	41,951
Train, &c..... Tuns	391	1,336	19,385	23,585
Turpentine .....	31,144	28,419	56,685	43,232
Other articles .. Value £	..	..	61,549	101,683
Total value of oils...	..	..	615,811	509,931

## IMPORTS OF METALS FOR MONTH ENDING 28TH FEBRUARY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Copper:—			£	£
Ore..... Tons	2,114	3,354	13,372	24,454
Regulus..... "	7,569	4,707	189,778	124,376
Unwrought .... "	3,547	4,301	169,453	184,685
Iron:—				
Ore..... "	572,711	333,461	264,419	239,626
Bolt, bar, &c.... "	1,226	2,555	10,107	19,363
Steel, unwrought.. "	162	488	2,125	4,907
Lead, pig and sheet "	12,666	15,895	126,625	127,880
Pyrites..... "	47,048	53,133	80,881	86,828
Quicksilver..... Lb.	947,515	455,750	80,692	40,998
Silver ore..... Value £	..	..	214,723	200,382
Tin..... Cwt.	67,029	44,565	307,535	161,148
Zinc..... Tons	3,820	3,730	70,104	61,201
Other articles... Value £	..	..	152,744	167,204
Total value of metals	..	..	1,682,738	1,529,142

## IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDING 28TH FEBRUARY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Bark, Peruvian.. Cwt.	17,042	8,577	31,294	15,160
Bristles..... Lb.	182,789	180,630	22,770	20,013
Caoutchouc..... Cwt.	19,385	24,188	228,302	294,394
Gum:—				
Arabic..... "	4,718	5,721	12,759	13,905
Lac, &c..... "	12,592	4,781	58,301	25,257
Gutta-percha .... "	2,656	3,890	25,739	29,026
Hides, raw:—				
Dry..... Cwt.	26,037	37,713	64,672	88,566
Wet..... "	29,744	49,459	64,080	101,873
Ivory..... "	498	506	24,729	22,363
Manure:—				
Guano..... Tons	983	3,623	7,205	29,707
Bones..... "	5,117	12,395	22,931	58,774
Nitrate of soda... "	5,703	13,079	51,498	120,749
Phosphate of lime "	33,606	27,269	60,588	55,827
Paraffin..... Cwt.	59,727	48,319	55,497	52,614
Linen rags..... Tons	2,438	1,254	27,029	12,436
Esparto..... "	14,522	19,315	68,753	84,933
Palp of wood .... "	11,037	19,777	54,860	104,062
Rosin..... Cwt.	136,166	155,505	25,440	35,988
Tallow and stearin "	56,219	89,658	82,282	105,628
Tar..... Barrels	708	1,655	317	1,063
Wood:—				
Hewn..... Loads	93,240	120,356	161,179	185,943
Sawn..... "	37,344	64,451	108,541	154,989
Staves..... "	2,612	3,838	13,231	19,578
Mahogany..... Tons	5,119	4,750	44,745	40,176
Other articles.... Value £	..	..	781,813	764,220
Total value.....	..	..	2,699,446	2,438,774

Besides the above, drugs to the value of 68,366<sup>l</sup>. were imported, as against 73,974<sup>l</sup>. in February 1893.

## IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH ENDING 28TH FEBRUARY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Alkali..... Cwt.	4,209	5,695	3,844	5,143
Bark (tanners', &c.) "	14,556	17,324	5,399	7,126
Brimstone..... "	62,736	46,505	15,031	10,377
Chemicals..... Value £	..	..	102,450	122,963
Cochineal ..... Cwt.	250	421	1,471	2,532
Cutch and gambier Tons	1,162	3,761	25,968	66,903
Dyes:—				
Aniline ..... Value £	..	..	11,628	16,138
Alizarin ..... "	..	..	20,456	20,422
Other ..... "	..	..	2,050	3,872
Indigo ..... Cwt.	17,168	12,707	368,664	262,219
Nitrate of potash. "	28,050	26,478	24,665	24,118
Valonia ..... Tons	4,133	2,986	55,214	39,173
Other articles... Value £	..	..	118,610	140,485
Total value of chemicals	..	..	754,160	721,477

## EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDING 28TH FEBRUARY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Gunpowder..... Lb.	449,300	661,800	11,547	15,956
Military stores.. Value £	..	..	85,362	59,610
Candles..... Lb.	1,708,800	1,309,700	30,480	25,023
Caoutchouc..... Value £	..	..	92,362	81,817
Cement..... Tons	27,155	26,822	46,968	44,299
Products of coal Value £	..	..	93,398	87,012
Earthenware ... "	..	..	153,563	102,155
Stoneware..... "	..	..	13,135	13,551
Glass:—				
Plate..... Sq. Ft.	137,749	100,990	6,017	5,304
Flint..... Cwt.	6,085	6,320	14,607	14,774
Bottles..... "	48,310	49,627	22,071	24,092
Other kinds.... "	15,517	10,927	18,694	8,620
Leather:—				
Unwrought .... "	12,216	11,139	121,082	102,913
Wrought ..... Value £	..	..	22,410	16,040
Seed oil..... Tons	5,041	5,888	108,620	123,552
Floorecloth ..... Sq. Yds.	1,547,600	1,861,600	64,279	66,639
Painters' materials Val. £	..	..	110,233	105,579
Paper ..... Cwt.	64,484	65,663	111,916	104,239
Rags..... Tons	5,510	4,161	42,591	22,559
Soap..... Cwt.	39,315	46,840	41,736	46,455
Total value.....	..	..	2,257,704	2,032,485

**EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING  
28TH FEBRUARY.**

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Alkali..... Cwt.	492,028	424,758	161,681	126,952
Bleaching materials ..	112,493	77,257	46,576	30,455
Chemical manures. Tons	33,426	33,839	222,307	246,125
Medicines..... Value £	..	..	71,607	77,845
Other articles ... ..	..	..	230,562	215,931
<b>Total value .....</b>	<b>..</b>	<b>..</b>	<b>735,536</b>	<b>696,458</b>

**EXPORTS OF METALS (OTHER THAN MACHINERY) FOR  
MONTH ENDING 28TH FEBRUARY.**

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Brass..... Cwt.	7,055	8,129	31,117	31,615
Copper:—				
Unwrought..... ..	37,116	23,841	91,014	58,593
Wrought..... ..	21,873	33,590	65,471	82,594
Mixed metal..... ..	24,749	31,471	69,406	70,048
Hardware..... Value £	..	..	156,439	140,298
Implements..... ..	..	..	89,141	90,556
Iron and steel..... Tons	191,223	173,861	1,554,251	1,346,379
Lead .....	2,274	3,606	27,622	42,274
Plated wares... Value £	..	..	17,228	17,291
Telegraph wires ..	..	..	97,496	133,118
Tin .....	8,761	10,078	41,787	38,593
Zinc .....	10,670	10,611	14,047	7,825
Other articles .. Value £	..	..	61,783	58,624
<b>Total value .....</b>	<b>..</b>	<b>..</b>	<b>2,301,823</b>	<b>2,117,208</b>

## Monthly Patent List.

\* The dates given are the dates of the Official Journals, in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

### L—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### APPLICATIONS.

3414. A. J. Boulé.—From P. de Molle et A. Willbaux, Belgium. Improvements in or relating to the filtration and purification of liquids, and apparatus therefor. February 17.

3665. H. Evers-Swindell. Improved manufacture of gas-retorts also applicable to other tubular objects of fire-clay or earthenware, and apparatus therefor. February 20.

3702. W. Charley. Improvements in machinery for the compression of air or other elastic fluids. February 21.

3792. S. H. Sparkes. Improvement in hydro-extractors. February 22.

3799. W. Under and J. Willoughby. Improvements in the method of and apparatus for controlling the supply of air to furnaces. February 22.

3805. J. W. Brown. Float for determining specific gravity and consistency of boiling tar, asphalt, bitumen, and other liquids. February 22.

3887. W. A. Kirkham. Improvements in or appertaining to plant for evaporating brine. February 24.

4069. O. Hamilton. An improved heating apparatus more particularly applicable to chemical processes. February 26.

4094. S. C. Hauberg. Improvements in centrifugal liquid separators. Complete Specification. February 26.

4098. H. H. Lake.—From the Actien-Gesellschaft für Kohlenäure-Industrie, Germany. An improved cap for cylinders or receptacles for containing liquid or gas under pressure. February 26.

4350. A. Normandy. Improvements in apparatus for evaporating and condensing water. March 1.

4549. W. Smethurst and The Smokeless Heat and Light Syndicate, Ltd. Improvements in the process of and apparatus for evaporating liquids. March 3.

4622. B. Andrew. See Class XII.

4700. L. B. Burnett. Apparatus capable of ready application to ordinary gas burners to convert them into Bunsen burners. March 6.

4895. H. Digne. An apparatus for melting tallow and analogous products for the desiccation of fruits, and for other operations requiring an equal and regular temperature comprised between 20° and 200° C. March 8.

5119. D. Drummond. Improvements in apparatus for expressing sap or juice from fibrous canes and other materials. March 12.

5187. C. Long and W. H. Borkitt. An improvement in acid raisers of steam-jet pumps for the raising of acids, alkalis, and other liquids. March 13.

5262. W. Henneberg. Improvements in apparatus for condensing vapours and heating or vaporising liquids respectively. Complete Specification. March 13.

5568. C. E. Miles. Improved means for and method of condensing vapour and for producing a vacuum in pans or like vessels used for evaporating or concentrating liquids. March 16.

5569. E. Edwards.—From F. P. Hofmeister, Germany. Improvements in apparatus for effecting the absorption of gases by liquids. March 16.

5613. T. Drost. An improved process for saturating or mixing solid substances with liquids. March 17.

## COMPLETE SPECIFICATIONS ACCEPTED.\*

1893.

3170. J. A. Morrell and J. Fromberz. Improvements in apparatus applicable for evaporating liquids or saccharine solutions. February 21.

4245. A. Shields. Improvements connected with refrigerating apparatus. February 28.

7525. J. Drummond. Improvements in apparatus for filtering liquids or for separating liquids from or drying crystallised or other granular matters. February 21.

7912. T. R. Murray. Improvements in pumps for pumping or compressing gases or vapours. February 28.

8125. W. H. Webb. Improvements in or connected with apparatus for cooling liquids. February 28.

9808. N. Yagn. Improvements in apparatus for sterilising water. March 7.

16,663. G. Morris and W. Edney. See Class XVII. March 14.

17,737. F. S. Cripps. Improvements in valves for tar, ammoniacal liquor, and other liquids. March 21.

24,106. W. P. Thompson.—From H. L. Fuge. Improvements in pressure regulators or reducers for gases, liquids, or the like. February 21.

21,526. H. Reiser. Process and apparatus for cleansing filtering material. March 7.

1894.

70. G. Frere and M. Meslans. Improvements in the method of and apparatus for determining the density of gases. March 7.

## II.—FUEL, GAS, AND LIGHT.

## APPLICATIONS.

5018. A. Farinetti. An automatic apparatus to produce gas for the purpose of light connected therewith. February 12.

3078. J. Jameson. An appliance for increasing, or increasing and regulating, the pressure of illuminating gas in houses, factories, or shops. February 13.

3223. W. Donaldson. See Class XI.

3395. W. R. Herring. Improvements in and in apparatus for manufacturing oil-gas. February 16.

3420. A. A. Routledge and W. Usher. An improved carburetter, for saving gas and increasing its illuminating power. February 17.

3558. F. W. Golby.—From H. Leffler, Germany. Improvements relating to the purification of combustion gases and the utilisation of the heat contained therein. February 19.

3571. F. Ilavati and F. Slacký. See Class VII.

3771. W. H. White. An improved composition for fire-lighters. February 21.

3794. R. C. Wilford. Improvements in apparatus for producing and regulating a combustible mixture of petroleum and atmospheric air for the purpose of operating explosion engines. February 22.

4053. J. Laing. Improvements in the manufacture of gas from mineral and other oils and tars, and in apparatus therefor. February 26.

4111. J. F. Duke. Improvements in the means for automatically lighting gas. February 27.

4189. C. W. Pinkney. Improvements in and apparatus for the manufacture of gas suitable for use in gas engines or for heating purposes. February 27.

4281. J. Mactear. Improvements in deodorising the exhaust gases from gas or oil motors or engines. February 28.

\* See Note (\*) on previous page.

4283. O. Imry.—From La Société Anonyme des Moteurs Thermique Gardie, France. Process for the manufacture of combustible gas. February 28.

4376. H. Palmer and E. Spain. Improvements in night-lights. March 1.

4506. R. Dunlop. Improvements in treating and utilising certain gases resulting from carbonisation of coal. March 3.

4661. A. MacLaine. Improvements in the combined manufacture of coal-gas and water-gas in gasworks and the carburetting of same. March 6.

4719. E. Fleischhauer and M. Bernstein. Improvements in and relating to the manufacture of gas. Complete Specification. March 6.

4812. W. P. Thompson. Improvements in obtaining or separating certain gases and in apparatus therefor. March 7.

4995. G. Buckley. Improvements in the manufacture of compressed fuel. March 9.

5092. F. Williams. An improved method of using small coke or breeze in the manufacture of coke and coke bricks. March 12.

5214. F. E. Baron. Improvements in liquid fuel and in means or apparatus employed in utilising the same. March 13.

5540. G. Wilton. Improvements in the method of and apparatus for the carbonisation of coal in the manufacture of coal-gas. March 16.

5656. A. Schneller and W. J. Wisse. Improvements in the production of ozone and in apparatus therefor. March 17.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

4475. F. Brunck. Improvements in horizontal coke-ovens. March 7.

4510. F. W. Clark and A. Forbes. Improvements relating to the distillation and breaking up of liquid hydrocarbons and similar substances, and to apparatus therefor. February 28.

5546. P. Drorkovitz. Improvements in the manufacture of coke, coal-gas, and the obtaining of by-products, and in apparatus employed therein. March 14.

8510. J. Moeller. A process for the manufacture of oil-gas. February 21.

23,381. T. Deiches and J. Komaike. Improvements in candles and wax and such like matches. March 21.

23,471. C. C. Walker. Improvements in or connected with the lids of gas retorts or the like. March 7.

1894.

1029. G. St. J. Kneller. Refractory balls and the like for incandescent gas fires or stoves. February 21.

1991. A. J. Boulton. From W. A. Koneman and A. F. Hatch. Process of and apparatus for utilising producer gas as fuel for calcining limestone. March 21.

3223. W. Donaldson. See Class XI.

## IV.—COLOURING MATTERS AND DYES.

## APPLICATIONS.

25,074A. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of a new dihydroxy-naphthalene disulpho acid. February 21.

3026. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of new bases containing sulphur of their sulphonic-acids, and of colouring matters derived therefrom. February 12.

3032. H. E. Newton.—From The Farbentabriken vormals F. Bayer and Co., Germany. The manufacture or production of azo dyestuffs. February 12.

3036. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. The manufacture and production of new mordant-dyeing colouring matters and intermediate products relating thereto. February 12.

3113. H. E. Newton.—From The Farbentabriken vormals F. Bayer and Co., Germany. The manufacture or production of colouring matters. February 13.

3301. S. Pitt.—From L. Cassella and Co., Germany. Tris-azo dyestuffs. February 15.

3397. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. The manufacture and production of new nitrosamine compounds suited for use in the preparation of diazo compounds. February 16.

3474. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. The manufacture and production of new mordant-dyeing colouring matters. February 17.

3828. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in the manufacture of naphtha-zarine and of new intermediate products related thereto. February 22.

3842. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of new bases and their sulpho-acids. February 22.

3843. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of colouring matters. February 22.

3844. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of a new naphthylenediaminedisulpho acid and of colouring matters therefrom. February 22.

3886. W. E. Heys.—From Sandoz and Co., Switzerland. Improvements in the production of colouring matters dyeing with mordants. Complete Specification. February 23.

4373. H. E. Newton.—From The Farbentabriken vormals F. Bayer and Co., Germany. The manufacture or production of azo colouring matters. March 1.

4459. S. B. Boulton, T. B. Haywood, H. E. Boulton, and H. Fergusson. Processes for the production of certain salts of beta-sulphonaphthalene and for the separation of alpha- and beta-sulphonaphthalenes. March 2.

4460. O. Imray.—From The Society of Chemical Industry in Basle, Switzerland. Manufacture of polyazo colouring matters. March 2.

4542. R. E. Evans. Manufacture of a new colouring matter. March 3.

4629. H. Imray.—From The Basle Chemical Works, Bindschedler, Switzerland. Manufacture of a new dioxynaphthalene-monosulpho acid and of colouring matters therefrom. Complete Specification. March 5.

4630. H. Imray.—From The Basle Chemical Works, Bindschedler, Switzerland. Manufacture of a new dioxynaphthoic monosulpho acid and of colouring matters therefrom. Complete Specification. March 5.

4870. J. Altschul. Improvements in the manufacture and application of a new diazo compound for use in dyeing and printing. March 8.

4893. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Improvements in the production of phenol-carbon acids. March 8.

5056. H. E. Newton.—From The Farbentabriken vormals F. Bayer and Co., Germany. The manufacture or production of colouring matters. March 10.

5143. R. Koreff. Manufacture of new amido bases and of azo colouring matters therefrom. Complete Specification. March 12.

5144. H. E. Newton.—From The Farbentabriken vormals F. Bayer and Co., Germany. The manufacture or production of azo colouring matters and materials thereof. March 12.

5159. H. H. Lake.—From Wirth and Co., Agents for A. Leenhardt and Co., Germany. Improvements in the manufacture of colouring matters. March 12.

5267. H. E. Newton.—From The Farbentabriken vormals F. Bayer and Co., Germany. The manufacture or production of azo dyestuffs and materials thereof. March 14.

5318. L. Koechl and J. Altschul. Production of new yellow colouring matters. March 14.

5444. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Binding, Germany. Improved manufacture of dry alizarine. March 15.

5546. C. D. Abel.—From A. Fischesser and Co., Germany. Manufacture of naphthylamine 1, 3, 5 trisulpho acid, and of two new naphthylamine 1, 3, 5 trisulpho acids derived therefrom. March 16.

5547. C. D. Abel.—From A. Fischesser and Co., Germany. Manufacture of 1, 8 amidonaphthyl 4, 6 disulpho acid. March 16.

5636. H. E. Newton.—From The Farbentabriken vormals F. Bayer and Co., Germany. The manufacture or production of polyazo dyestuffs. March 17.

#### COMPLETE SPECIFICATION ACCEPTED.

1893.

5693. J. H. Ziegler. Manufacture of new colouring matters by the action of diazo compounds on the hydrazine derivatives of oxalylacetic ether. February 21.

6035. H. E. Newton.—From The Farbentabriken vormals F. Bayer and Co. The production or manufacture of colouring matters. February 28.

6035A. H. E. Newton.—From The Farbentabriken vormals F. Bayer and Co. The manufacture or production of colouring matters. February 28.

7330. H. E. Newton.—From The Farbentabriken vormals F. Bayer and Co. The manufacture or production of dye stuffs. March 21.

8181. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik. The manufacture of new sulpho-acids of phenylrosinduline and analogues and homologues thereof. February 28.

8221. W. H. Claus. Manufacture of blue colouring matter containing sulphur. March 7.

8824. H. E. Newton.—From The Farbentabriken vormals F. Bayer and Co. Improvements in the manufacture or production of basic compounds derived from pyrazine. March 14.

8898. H. E. Newton.—From The Farbentabriken vormals F. Bayer and Co. The manufacture or production of colouring matters and materials thereof. March 14.

9689. Read, Holliday, and Sons, Lim., and R. Holliday. The production of new colouring matters for dyeing and printing. March 14.

9894. H. E. Newton.—From The Farbentabriken vormals F. Bayer and Co. The manufacture or production of azo colouring matters. March 21.

9969. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation. Manufacture of basic colouring matters. March 21.

1894.

2232. W. G. Thompson and I. Moore. Improvements in the manufacture of a diazido base and of tetraazo colouring matters derived therefrom. March 7.

## V.—TEXTILES, COTTON, WOOL, SILK, Etc.

## APPLICATIONS.

3100. J. Smith and H. F. Clayton. A new method of means for treating yarn slubbing and woven or felted fabrics. February 12.

3103. S. F. Hughes. An improved means for removing grease or other fatty compounds from all raw material used in the manufacture of woollen, cotton, silk, linen, or other fabrics, and the fabrics themselves, as well as to increase the efficiency of oxidising agents when used for the purpose of kaulsing and bleaching. February 14.

3184. O. Reichenbach. An improved process and apparatus for cleaning wool and other textile products. Complete Specification. February 17.

3534. J. V. Eves. Improvements in the treatment of wet spun flax yarn. February 19.

3569. J. Faust. Extracting the fibres out of "ramie-plants" and the like. February 27.

3642. D. Emsley and J. Robinson. An improved method of clearing or preparing rice fibre. February 24.

3644. T. Cheetham. Improvements in means for facilitating the examination of textile fabrics. February 26.

3727. J. Lorraine. Improvements in the strengthening of textile materials or substances subject to tensile strain. February 28.

3832. J. S. Farmer, H. L. Storey, and I. H. Storey. Improvements in the manufacture of solid-colour floor-cloths, and in machinery employed in such manufacture. March 2.

4481. A. Drew. Improvement in treating dyed or printed textile fabrics. March 3.

4665. E. de Pass.—From J. C. McLauchlin and A. A. Hand, United States. Improvements in the manufacture of fabrics from fibre. March 5.

4697. A. Zimmermann.—From J. Hoffert, Germany. Improvements in treating tissues, fibres, paper, and the like. March 6.

4879. J. Anderson, jun. Improvements in the method and means of washing wool and similar substances for extracting greasy, oily, and other fatty matters therefrom and recovering products therefrom. March 8.

5128. J. Harrison and C. Wolfendale. Improvements in and apparatus for the manufacture of inlaid or through-colour floor coverings. March 12.

5150. J. Harrison and C. Wolfendale. Improvements in and apparatus for the manufacture of inlaid or through-colour floor coverings. March 12.

5151. H. S. Close and W. O. Roff. An improved process of retting flax, jute, or other similar fibrous materials and eliminating the gummy or other matter therefrom and utilising the resultant residuals. March 12.

5167. J. Harrison and C. Wolfendale. Improvements in and apparatus for the manufacture of inlaid or through-colour floor coverings. March 12.

5334. R. J. H. Rastick. Improvements in the treatment of reed and other fibrous plants. March 11.

## COMPLETE SPECIFICATION ACCEPTED.

1893.

8717. J. C. Mewburn.—From J. Cardon. Improvements in machinery for breaking, softening, and removing the gummy matter from hemp, ramie, and other fibrous materials. March 7.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

## APPLICATIONS.

3103. S. P. Eastick and W. J. Stonhill. A process for disinfecting, bleaching, and converting rags into a pulpy or "half-stuff" condition. February 13.

3193. S. F. Hughes. See Class V.

3378. W. P. Thompson.—From E. Haefely, Germany. An improved process of dyeing for producing genuine Turkey red and rose colour in yarn hanks and fabrics. Complete Specification. February 16.

5186. G. Keighley. A new or improved means or method of dyeing warp on the beam. March 13.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

4437. C. Owens and J. Whiteley. Improvements in machines for washing, bleaching, mordanting, dyeing cotton, wool, or other fibres in a raw, spun, or manufactured state. February 28.

5291. Rhodes, Rhodes, jun., and Perkins. Improvements in apparatus for dyeing and washing yarn in the hank. March 7.

7487. Read, Holliday, and Sons, Lim., and H. Bind-schaller. Improvements in the production of an indigo vat or bath employed in the dyeing with indigo. February 21.

7919. Hay and Park. Improvements in dyeing certain colours on cotton fabrics and yarns. February 28.

7841. E. Gessler. Improvements in dyeing apparatus. February 28.

8167. F. R. Wells. A process for colouring paper and cardboard. February 21.

9682. J. Relitt. Improvements in the method of and apparatus for dyeing, mordanting, and otherwise treating woven or felted fabrics and yarns. March 21.

23,559. W. P. Thompson.—From E. Holken and Co. An improved process of dyeing for producing genuine Turkey-red and rose-colour in vegetable yarns in cops, hanks, carded strips, and the like. February 21.

1894.

3378. W. P. Thompson.—From E. Haefely, Germany. An improved process of dyeing for producing genuine Turkey red and rose colour in yarn hanks and fabrics. Complete Specification. February 16.

## VII.—ACIDS, ALKALIS, AND SALTS.

## APPLICATIONS.

3637. J. Brock, V. C. Driffeld, A. Carey, and F. Brown. Improvements in and apparatus for the treatment of "caustic salts" obtained as a by-product in the manufacture of caustic soda. February 12.

3372. S. Z. de Ferranti and J. H. Noad. Improvements in the manufacture of oxides and carbonates of lead. February 16.

3560. F. W. Golby.—From L. Schrader, Germany. A process for making chloride of strontium from sulphate of strontium. February 19.

3571. F. Ilavati and F. Sladky. Processes for the production from air, with or without admixture of oxygen, of nitric acid, nitrates, and other nitrogen salts; also the production from air, together with water-gas or hydrogen of ammonia, and compounds thereof. February 19.

3767. H. R. Angel. Improvements in the manufacture of carbonic acid gas, bicarbonate of soda, and metallic sulphur. February 21.

4013. A. R. Davis. Improvements in the distillation of ammonia. February 24.

4034. W. Mills. Improvements in the manufacture of soda and potassa. February 26.

4341. G. Foulcr. Improvements in and apparatus for the manufacture of ammonia, and of by-products obtained therein. March 1.

4423. R. Wolfenstein. Improvements in the development or preparation of high percentage and of chemically-pure super-oxide of hydrogen. March 2.

4573. W. Garraway. Improvements in the manufacture of caustic soda, nitric and sulphuric acids. March 5.

4791. H. Wächter. A bleaching preparation or mixture applicable also for other purposes. Complete Specification. March 7.

4916. A. J. Boulton.—From E. Warzee, Belgium. Process for the separation and precipitation of salts of iron and zinc from solutions containing both. Complete Specification. March 8.

5168. J. Y. Johnson.—From J. Marx, Germany. Improvements in or connected with the manufacture of plumbates and plumbic acid (peroxide and other oxides of lead). March 12.

5178. M. W. Beylikgy. A new and improved process of manufacturing alkaline bichromates. March 13.

5237. R. Campion. Improvements in the production of soda. March 13.

5300. W. Mills. Improvements in utilising spent iron pyrites and apparatus therefor. March 14.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

4310. A. Gray. See Class X.

7485. F. M. Spence, D. D. Spence, and J. Gilchrist. Improvements in certain apparatus for the distillation of ammoniacal liquor. March 7.

7512. J. Brock and F. Hurter. Improvements in the manufacture of permanganates of soda or potash from manganates of soda or potash. February 21.

7846. C. Raspe. Improved process for the manufacture of carbonate of ammonia. February 21.

8907. H. R. Browne and M. Guthrie. Improvements in the manufacture or production of soda crystals. March 7.

9271. G. S. Albright and J. J. Hood. Improvements in the manufacture of manganese peroxide. March 14.

12,997. La Société Marcheville Daguin and Co. Improvements in calcining apparatus for the conversion of bicarbonate of soda into carbonate of soda, and other uses. March 14.

#### VIII.—GLASS, POTTERY, AND EARTHENWARE.

##### APPLICATIONS.

3677. J. Hawthorn. Improvements in pottery kilns. February 20.

3695. E. H. Pearce, J. Brettell, and G. Thomas. Improvements in the art of glass decoration. February 21.

4081. C. Margot. An improved process for producing indelible ornamentations upon glass or glazed surfaces. February 26.

#### COMPLETE SPECIFICATION ACCEPTED.

1893.

4385. H. Hilde. Improved method and machinery for blowing and casting articles of glass. February 21.

#### IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

##### APPLICATIONS.

3496. L. Tabbatt. New method of covering bricks, walls, and similar surfaces with a glazed or enamelled surface. February 14.

3738. J. McDonnell. Improved method of making bricks for street and other paving from fibre or fibrous waste and bitumen or asphalt or similar substances. February 21.

3843. T. D. Harries. An improved artificial stone. Complete Specification. February 22.

4223. P. Baumert and A. Pieck. Improvements in the manufacture of artificial stone or marble. Complete Specification. February 28.

4275. F. Turner. A novel compound adaptable as a cement. February 28.

4377. R. Astley. Improvements in the construction of fireproof floors. March 1.

4632. A. Kuhnwein. Manufacture of asbestos cement and of coverings or moulded objects made therefrom. March 5.

5071. G. W. A. Stein. Improved process of manufacturing hydraulic cement. Complete Specification. March 10.

5137. E. Benard. Improvements in the manufacture of cements. March 12.

5160. C. J. Potter. An improved material for paving and like purposes. March 12.

5590. J. I. Cox. Improvements in fire-clay and other bricks. March 17.

5595. C. A. Ropes and J. C. Sellars. Improvements in or connected with cements or cement work. March 17.

5626. T. L. Banks. Improvements in the construction of fireproof floors. March 17.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

7499. W. Sykes. Improvements in the manufacture of compositions suitable for paving, building, pipe-jointing, and other like purposes. February 28.

7501. R. Astley. An improved form and method of construction of fireproof floors. February 21.

8441. N. Rigby. An improved material for covering or decorating walls and the like, and the method of producing the same. February 28.

9044. H. H. Leigh.—From A. Jumeau. Improvements in the mosaic reproduction of decorative painting. March 14.

16,415. W. A. Barr. Improvements in and connected with fireproof ceilings. February 21.

1894.

477. G. W. A. Stein. Improved method of manufacturing hydraulic cement. March 14.

1396. H. H. Leigh.—From G. Bagge. Improvements in paving blocks. February 28.

#### X.—METALLURGY, MINING, Etc.

##### APPLICATIONS.

3224. J. M. White. Improvements in the manufacture of steel or iron plates and apparatus therefor. February 14.

3228. J. W. Swan. Improvements in and apparatus for desilverising lead. February 14.

3680. J. Nicholas. Improvements in obtaining gold and silver from ores and other compounds, and apparatus therefor. February 20.



3711. H. J. Phillips. A method of preparing, desulphurising, and carbonising bricks, for a process of desulphurising and carbonising molten iron or steel in one operation. February 21.

3705. F. Tscherrin. Manufacture of metallic alloys and mechanical mixtures of metals of high specific gravity, applicable for projectiles and cores of projectiles. February 23.

3936. W. F. Reid. A new or improved process and material for the production of articles of metal. February 24.

3991. J. Bedford. Improvements in the manufacture of steel and in alloys for use therein. February 24.

4199. H. W. Gabbett-Fairfax and W. Beardmore. Improved method of and apparatus for hardening or tempering steel plates and other articles. February 27.

4335. J. Stuart and J. Mason. Improvements in the production of aluminium. March 1.

4533. T. Hampton. Improved method of and moulds for casting compounds, ingots for armour plates and other plates. Complete Specification. March 3.

4761. C. T. J. Oppermann. Improvements in and connected with the deposition of metals upon aluminium. March 7.

4840. G. Hookham. A new metallic alloy for cartridge cases and articles subjected to high temperatures. March 7.

4980. W. P. Thompson.—From O. Nicolai, Germany. A new or improved solder for soldering aluminium, aluminium alloys, and other metals. Complete Specification. March 9.

5090. E. C. Broadwell. Improvements in soldering materials for aluminium. March 10.

5235. D. Blyth. Improvements in the treatment of iron ores for use in the manufacture of steel, for purifying and other purposes. March 13.

5345. J. A. Mays. Improvements in apparatus for oxidising comminuted lead or zinc. March 14.

5346. J. A. Mays. Apparatus for comminuting and converting molten materials. March 14.

5379. W. D. Allen. An improved furnace for treating ingots, blooms, or billets of iron or steel. March 14.

5525. C. W. Buckle and J. Stuart. A new or improved metallic alloy. March 16.

5530. E. Singer, F. Donat, and P. Kircheisen. Process for soft-soldering aluminium. Complete Specification. March 16.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

4310. A. Gray. Improvements in and relating to the manufacture of zinc oxides. February 28.

5218. J. S. McArthur.—From The Cassel Gold Extracting Company, Limited, and C. J. Ellis. Improvements in extracting gold and silver from ores and the like. March 14.

6219. H. Wild. Improvements in or relating to the "Thomas" metallurgical process. February 28.

7453. W. Wilkinson. Improvements in ingot moulds, and in the process of casting hollow metal ingots. February 21.

7730. A. Longsdon.—From L. Grambow. A new or improved process for treating steel armour plates. February 21.

8057. A. Sattmann and A. Homatsch. Improvements in the manufacture of iron and steel. February 28.

8456. B. H. Thwaite. Improvements in the manufacture of steel and in the refining of fluid iron. March 14.

9085. S. Meredith. Improvements in puddling furnaces used in the manufacture of iron and steel. March 14.

9406. J. H. Ladd. Improvements in treating malleable or wrought iron. March 14.

12,825. R. Lewis and G. Morgan. Improvements in apparatus for coating iron and steel plates with tin or other metals or alloys. March 14.

25,071. A. Trug. Method and apparatus for extracting gold and silver from their ores by the combined action of amalgamation and centrifugal force. March 21.

1894.

2041. J. C. Fell.—From The Emmens Zinc Company. Improvements in the treatment of zinc-lead-sulphide ores carrying gold or silver or gold and silver. March 7.

#### XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

##### APPLICATIONS.

3223. W. Donaldson. Improvements in heating of gases by electricity. Complete Specification. February 14.

3674. W. H. Longsdorf.—From G. Hewett, United States. Improvements in primary galvanic batteries and in the liquids used therein. February 20.

3725. T. Blackhurst. Dry or semi-dry galvanic elements. February 21.

3749. A. J. Boulton.—From C. Cudell, I. Cudell, and M. Cudell, Germany. Improvements in galvanic batteries. February 21.

3922. M. Roskell. Improvements in or relating to the electro-deposition of metals. February 23.

4113. H. T. Barnett. Improvements in electric batteries. February 27.

4168. A. W. Hussey. Improved construction of electric battery. February 27.

4325. A. H. Harris. Improvements in the electro-deposition of aluminium. March 1.

4472. T. Parker. Improvement in electrolytic processes in the manufacture of chlorine alkali and derived products. March 3.

4635. H. Wehmann. Improvements in galvanic batteries. March 5.

4724. O. March. Improvements in and relating to secondary batteries. March 6.

5537. R. Heathfield and W. S. Rawson. Electrical deposition of zinc and alloys of zinc, and apparatus therefor. March 16.

5579. J. Kirkwood. Improved solutions for the electric deposition, separation, and refinement of aluminium. March 17.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1892.

23,101. C. A. J. H. Schroeder and H. E. R. Schroeder. New primary batteries. March 21.

1893.

1114. A. W. Armstrong. An improvement in the electrodes of electric, galvanic, and other batteries. February 21.

5197. J. Hargreaves and T. Bird. Improvements in the production of alkali and in apparatus therefor, the said apparatus being applicable for electrolysis generally. March 14.

5198. J. Hargreaves and T. Bird. Improvements in and in making electrolytic apparatus. March 14.

7594. F. M. Lyte. Improvements in the electrolytical decomposition of chlorides and mixtures thereof, and in apparatus for the purpose. March 7.

8571. H. C. W. Emery. Improvements in or connected with cells for electric batteries. March 7.

10,584. H. Y. Castner. Improvements in connection with electrolytic apparatus. March 14.

10,942. W. Walker, jun., and F. R. Wilkins. Improvements in primary voltaic batteries. March 24.

23,881. W. P. Thompson.—From C. L. Coffin. Improved apparatus for heating metals electrically. March 7.

24,274. C. Kellner. Improvements in apparatus for the electrolytic decomposition of metallic salts. March 7.

24,871. H. T. Johnson. Improvements in dry batteries. February 21.

1894.

2900. J. Rudholzner. Improvements relating to the electro-deposition of metals. March 14.

3223. W. Donaldson. Improvements in the heating of gases by electricity. March 14.

### XII.—FATS, OILS, AND SOAP MANUFACTURE.

#### APPLICATIONS.

3025. C. Weygang. Manufacture of saponaceous products from petroleum. February 12.

3197. H. Lambert. Improvements in the method of and machinery or apparatus for extracting oil from seeds and the like, and for forming cakes from such or any suitable substance. February 14.

4622. R. Andrew. Improvements in purifying liquids and fats, and apparatus therefor. March 5.

4819. A. Blackie. Improvements in preparing, packing, and dissolving mixed powders to form liquids or washes, suitable for dipping animals and for washing or syringing plants for destroying germs, parasites, and mildew, and for other uses. March 7.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

7742. J. E. Bedford and C. S. Bedford. Improvements in the manufacture of linoleum oil. February 28.

8621. H. Hamilton. A new or improved composition of soap. February 21.

12,884. C. R. Illingworth. A superfatted biniodide of mercury soap. March 21.

18,658. J. Mellinger. Improvements in soap for removing hair from the skin. February 21.

### XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

#### APPLICATIONS.

3372. S. Z. de Ferranti and J. H. Noad. See Class VII.

4059. J. Masters. A special preparation of rubber. February 26.

4201. F. A. W. Sly. An improved manufacture of varnish. February 27.

4505. J. S. Macarthur. New or improved composition for varnishing or polishing. March 3.

5168. J. Y. Johnson.—From J. Marx, Germany. See Class VII.

5347. J. A. Mays. Process of and apparatus for the manufacture of white lead. March 14.

5414. A. R. von Pischof. An improved process for manufacturing a permanently elastic substance. March 15.

5440. E. Hérisse. An elastic composition for moulds, waterproofing, and other purposes. March 15.

5608. J. D. Noble. A new or improved fireproof and insulating paint. March 17.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

1113. S. J. Simpkin. Improvement in the manufacture of refined indigo. February 21.

3886. W. M. Walters. Improvements in the composition and treatment of substances for use as glue, putty, and cement. February 28.

7119. A. C. J. Charlier. Improvements in apparatus for the manufacture of metallic pigments or compounds, and in the production of lead and zinc pigments or compounds, and combinations of these by means of such apparatus. March 14.

9611. W. P. Thompson.—From G. Leuchs and C. Leuchs. Improvements in the manufacture of glazes, enamels, and the like. March 21.

1894.

166. O. Inray.—From J. Sachs, C. E. Meier, and M. Gerstenborfer. Improvements in the manufacture of pigments. March 14.

### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

#### APPLICATIONS.

3615. E. H. Lewis. Improvement in the art or process of waterproofing leather and in the compounds employed in same. Complete Specification. February 20.

3616. E. H. Lewis. Improvement in the art or process of waterproofing leather. Complete Specification. February 20.

4193. A. Levinstein. Improvements in the tanning of hides. February 27.

4679. H. Walker and J. J. Wilson. A new or improved process in or connected with the currying of skins and appliances therefor. March 6.

4696. A. Zimmermann.—From The Chemische Fabrik auf Actien vormals E. Schering, Germany. Improvements in the treatment and testing of gelatinous matter. March 6.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

3886. W. M. Walters. See Class XIII.

21,088. T. J. W. Mackenzie and W. J. Nodin. A new ornamental leather and process for preparing the same. March 21.

1894.

2079. G. E. Wiese. Improvements in the manufacture of liquid glue. March 7.

2175. J. Grillo and M. Schroeder. Process for the production of glue and gelatin from bones. March 7.

### XV.—AGRICULTURE AND MANURES.

#### APPLICATIONS.

4988. E. E. Dietz. Improvements in the manufacture of phosphatic slags of a high percentage of soluble phosphate with or without magnesia or potash. March 9.

5517. D. Blyth. Improvements in the preparation of manures. March 16.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

7637 W. Brothers. Improved manure or fertiliser. February 21.

1894.

2624 J. J. Seldner and J. Watson, jun. Improvement in the manufacture of fertilisers. March 14.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

## APPLICATIONS.

3733. T. Sheldon. Improvements applicable to treacle, honey, and similar viscous syrups. February 21.

3782. E. Langen. Improvements in refining sugar. Complete Specification. February 21.

3813. G. Pott. Improvements in apparatus for mixing and heating or cooling moist sugar or like substances. February 22.

5119. D. Drummond. See Class I.

5512. E. Leconte. Process for purifying and decolorising saccharine juices in the manufacture of beetroot and other sugar. March 16.

5518. D. Blyth. Improvements in the method of purifying and decolorising saccharine, amylaceous, and other fluid bodies. March 16.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

3170. J. A. Morrell and J. Fromberz. See Class I.

5793. J. Hanson, P. C. D. Castle, and J. H. Morrison. Improvements in or appertaining to the utilisation of a waste or nearly waste product, and the manufacture of a useful gum or gummy compound therefrom. March 7.

## XVII.—BREWING, WINES, SPIRITS, Etc.

## APPLICATIONS.

3055. L. L. Thomas. Solidified herb beer extracts and wine essences. February 13.

3076. D. A. Blair. Improvements in and relating to continuous working spirit stills. February 13.

3296. J. B. Bouyoud and A. Vellnet. Waterless liqueur. February 15.

3642. J. C. Pennington. Improved process for the manufacture of beer. Complete Specification. February 20.

3997. C. Rach. Apparatus for the preparation of wort. February 24.

3998. C. Rach. Apparatus for brewing. February 24.

4678. C. P. Hendy. Apparatus to prevent waste when fining beers and ales in casks. February 26.

4894. P. Pavrez. Apparatus for pasteurising or sterilising and cooling beers, and for aerating or charging beers with gas before placing them in cask or in bottle. Filed March 8. Date claimed August 23, 1893, being date of application in France.

5618. G. H. U. Harrow. Improvements in brewing and distilling. March 17.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

6168. G. de Geyter. Improvements relating to the saccharification of wort or the like, and to the extraction and subsequent treatment thereof in brewing, distilling, and in the manufacture of maltose, syrups, and other extracts, and apparatus therefor. March 21.

16,063. G. Morris and W. Edney. An improved still or apparatus for distilling spirits and all kinds of other liquids. March 14.

24,548. R. George. Improvements in the preparation of malt and in apparatus therefor. March 7.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

## APPLICATIONS.

*A.—Chemistry of Foods.*

3413. R. C. Wilson. Improvements in or connected with the manufacture of salt. February 17.

3791. J. F. Duke and A. Foucard. An improved method of treating preserved eggs. February 22.

4049. A. L. Lebet. An improved food compound having for its base milk. February 26.

4050. A. L. Lebet. An improved food compound having for its base milk. February 26.

4193. F. Goracci and A. Anselmi. A new or improved compound for use as an ingredient for soup or other food. February 26.

4548. J. Raschen. Improved substitutes for butter and lard. March 3.

4977. E. W. Beech and T. Wilson. Improved means, method of, and appliances for the preservation of food extracts. March 9.

*B.—Sanitary Chemistry.*

3333. W. Ambler. Improvements in the method of and apparatus for annihilating fumes emitted from destructors for destroying towns' refuse and the like. February 16.

3969. H. Lockwood. Improvements in the purification of sewage and other foul waters. February 24.

4169. A. J. Bonlt.—From F. S. Salberg, England, and M. Neumann, Austria. A sanitary process and apparatus for towns and trades. Complete Specification. February 27.

4195. H. H. Lake.—From Seaburg and Johnson, United States. Improvements relating to the burning of sulphur for disinfecting purposes. Complete Specification. February 27.

4210. J. Brierley. Improvements in filters for treating sewage or other refuse or fouled waters. February 28.

4725. H. Rieusch. Improved plant for purifying waste water. March 6.

4727. E. H. L. Ostermann. Improved process for purifying waste waters. March 6.

5105. E. C. Ives. Improved means or apparatus for purifying sewage or other foul or polluted waters. March 12.

*C.—Disinfectants.*

5066. R. J. Stephens and J. Hewes. Improvements in or relating to disinfectants and deodorants. March 10.

5288. A. Kraus and A. Chevrier. An improved manufacture of an antiseptic and microbicide. March 13.

## COMPLETE SPECIFICATIONS ACCEPTED.

*A.—Chemistry of Foods.*

1893.

15,161. W. F. E. Casse. Process for preserving milk and cream for a length of time, rendering it suitable for lengthy transit. March 14.

1894.

1170. R. F. Maclosky.—From J. H. Niemann. A new or improved meat extract. March 21.

**B.—Sanitary Chemistry.**

1893.

6423. J. I. Meldrum and T. F. Meldrum. Improvements in or connected with the purification of sewage and analogous foul or impure liquids. March 7.

9063. R. C. Tanner. A new or improved method of treating sewage by means of ferric compounds. March 21.

**C.—Disinfectants.**

1893.

6553. P. Molyneux and G. R. Potts. Improvements in or connected with disinfectants. March 7.

8129. S. Pitt.—From The Chemische Fabrik auf Actien vormals E. Schering. Improvements in the preparation of antiseptics. March 7.

24,709. S. F. Kattenhoy. Apparatus for the production of ozone from phosphorus for disinfecting purposes. March 7.

**XIX.—PAPER, PASTEBOARD, Etc.**

**APPLICATIONS.**

3103. S. P. Eastick and W. J. Stonhill. A process for disinfecting, bleaching, and converting rags into a pulpy or "half stuff" condition. February 13.

3105. J. Baier and R. W. Baxter. Improvements in the manufacture of imitation whalebone. February 13.

3440. E. Partington. Improvements in the treatment of "sulphite pulp" used in the manufacture of paper and the like from wood. February 17.

3906. C. Beadie. A process for treating paper so as to provide against falsification of documents. February 23.

4697. A. Zimmermann.—From J. Hoffert, Germany. See Class V.

5054. A. E. Healey and J. Williams. Improvements in treating paper and other fabrics, and in the preparation of solutions for that purpose, and the obtaining of a by-product in the said preparation. March 10.

5557. P. W. Wilkinson. An improved manufacture of paper for bank notes and other documents. March 16.

**COMPLETE SPECIFICATIONS ACCEPTED.**

1893.

9827. S. Wheeler. Improvements in the manufacture of paper and like materials. March 21.

23,502. W. P. Thompson.—From C. Schauffelen. An improved finely-granulated paper and process of producing the same. March 21.

24,275. C. Kellner. Improvements in the manufacture of cellulose. March 7.

24,287. C. Kellner. Improvements in the manufacture of cellulose. March 14.

1894.

824. W. White. An improved method of preparing pigment paper for photographic printing. February 21.

**XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.**

**APPLICATIONS.**

3055. L. L. Thomas. Solidified herb beer extracts and wine essences. February 13.

3179. S. Radlauer. A new or improved anti-pyretic and anti-neuralgic preparation. Complete Specification. February 14.

3290. J. B. Bonyard and A. Velluet. See Class XVII.

3553. R. Haddan.—From C. Strohler and Co., Germany. A new or improved herbal extract and process of producing the same. February 19.

4018. W. Malthmann. An improved manufacture of ketone musk. Complete Specification. February 24.

4997. J. A. Johnson.—From C. F. Boehringer and Sohn, Germany. The treatment of ergot of rye and the production therefrom of the substance which imparts to it its valuable properties. February 26.

**COMPLETE SPECIFICATIONS ACCEPTED.**

1893.

8735. J. C. W. F. Tiemann. Manufacture of a new chemical substance from orris-root. March 14.

8736. J. C. W. F. Tiemann. Manufacture of new chemical substances suitable for perfumery, confectionery, and the like. February 28.

1894.

684. F. W. Warrick. An improvement in capsule fillings containing ferrous salts. February 21.

3179. S. Radlauer. A new or improved anti-pyretic and anti-neuralgic preparation. Complete Specification. February 14.

**XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.**

**APPLICATIONS.**

3121. M. Russo and G. B. Zanardo. A process for the production of objects, either in relief or in intaglio with the aid of photography. February 13.

4276. A. Zimmermann.—From The Chemische Fabrik auf Actien vormals E. Schering, Germany. Improvements in the manufacture of photographic printing plates. February 28.

4346. F. Slavin and S. Pollastrini. A new or improved method or means of photographing in colours. March 1.

4606. C. E. Pettitt. Improvements in the manufacture of sensitive plates and films for photographic purposes. March 5.

4627. T. H. Blair and The European Blair Camera Company, Limited. Improvements in or relating to appliances for the manufacture of photographic films. March 5.

4689. J. R. C. Gale and J. W. T. Cadett. Improvements in the preparation, packing, and storing of photographic chemical compounds. March 6.

5061. S. H. A. Oakes and R. R. Gibbs. Improvements in or appertaining to photographs. March 10.

5511. T. Moore. New method of reproducing prints from photographic negatives. March 16.

5561. A. Zimmermann.—From The Chemische Fabrik auf Actien vormals E. Schering, Germany. Improvements in photography. March 16.

**COMPLETE SPECIFICATIONS ACCEPTED.**

1893.

5504. A. J. Boulton.—From T. H. Blair. Improvements in or relating to the manufacture of films for photographic purposes. March 21.

1894.

824. W. White. See Class XIX.

## XXII.—EXPLOSIVES, MATCHES, ETC.

## COMPLETE SPECIFICATIONS ACCEPTED.

## APPLICATIONS.

3024. C. D. Abel.—From L. König, Germany. Improved manufacture of explosives from nitrate of ammonia and resins. Complete Specification. February 12.

3773. E. Hesketh and A. Mareet. Improvements in or connected with the manufacture of gun-cotton or other manufactures in which cooling water liable to become acidulated is employed. February 21.

4543. E. de Poorter, A. Walton, and T. H. Andreal. The manufacture of an improved smokeless explosive compound. March 3.

4626. A. Manrette. An explosive composition. Complete Specification. March 5.

1893.

22,992. H. C. Seddon. Improvements in percussion fuses for projectiles. March 21.

23,381. T. Deiches and J. Komaike. See Class II.

1894.

1982. V. I. Feeny.—From The United States Smokeless Powder Co. Smokeless powder. March 7.

3024. C. D. Abel.—From L. König, Germany. Improved manufacture of explosives from nitrate of ammonia and resins. Complete Specification. February 12.

# THE JOURNAL

OF THE

# Society of Chemical Industry:

## A MONTHLY RECORD

FOR ALL INTERESTED IN CHEMICAL MANUFACTURES.

No. 4.—Vol. XIII.]

APRIL 30, 1894.

[Non-Members 30/- per annum; Members 21/- per Set of extra or back numbers; Single Copies (Members only) 2 6.

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		D.Sc., F.R.S. ....	XI., XII.

## NOTICES.

In accordance with the provisions of Rule 18 of the By-Laws, notice is hereby given that those Members of Council whose names are placed in italics in the annexed list, will retire from their respective offices at the forthcoming Annual General Meeting.

Dr. T. E. Thorpe, F.R.S., has been nominated to the office of President; and Mr. E. C. C. Stanford has been nominated Vice-President under Rule 11.

Mr. John Calderwood, Dr. C. Dreyfus, Dr. A. Dupré, F.R.S., and Prof. W. A. Tilden, F.R.S., have been nominated Vice-Presidents under Rule 8; and Mr. Wm. Thorp has been nominated an Ordinary Member of Council under Rule 17, in the place of Dr. C. Dreyfus, nominated a Vice-President.

## THE JOURNAL.

### Publication Committee:

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John Heron.	John Spiller.
D. B. Hewitt, M.D.	Wm. Thorp.
David Howard.	Thomas Tyrer.
Prof. J. J. Hummel.	

Mr. C. A. FEARNS has been nominated an Ordinary Member of Council under Rule 24, and the Treasurer and Foreign Secretary have been nominated for re-election to their respective offices.

Members are hereby invited to nominate fit and proper persons to fill four vacancies among the Ordinary Members of Council under Rule 18. Special nomination forms for this purpose can be obtained from the General Secretary upon application.

*Extract from Rule 18:*—"No such nomination shall be valid unless it be signed by at least ten Members of the Society who are not in arrears with their subscriptions, nor unless it be received by the General Secretary, at the Society's Office, at least one month before the date of the Annual General Meeting, to the election to take place at which it refers. Nor shall any such nomination be valid if the person nominated be ineligible for election under Rules 12 or 15. No member shall sign more than one nomination form."

#### ALTERATION OF BY-LAWS

Notice is hereby given, in accordance with Rule 54 of the By-Laws, that the Council will propose to the forthcoming Annual Meeting the amendment of the Society's By-Laws as follows:—

1. That *Rule 7* be amended by the deletion of "and" in the first line thereof, and by the addition of the words "and 47" in the same line.

2. That *Rule 15* shall read as follows:—

"Four Ordinary Members of Council shall retire each year, and those who thus retire shall not be eligible for re-election to the Council, except under By-laws 14, 23, and 24, until the Annual General Meeting next following that at which they retire. When an Ordinary Member of Council is nominated for election to the office of President, he shall not be counted among those who are to retire."

3. That the first sentence of *Rule 16* read as follows:—

"Except that any Ordinary Member of Council may be nominated for election to the office of President, and that such member, if any, shall not be included in the operation of the present By-law, the Ordinary Members of Council shall retire as far as may be possible in the order in which they were elected."

4. That *Rule 17* shall read as follows:—

"When an Ordinary Member of Council is nominated for election to the office of President, the Council shall nominate a Member for election as an Ordinary Member of Council in his place."

5. That under *Rule 18* the eighth line be amended to read as follows:—"and also the name of the person, if any, who has been nominated, &c., &c."; that the form of nomination of candidates for the Council be amended by transferring the candidate's declaration to the end thereof; and that the words "and by the nominee" be added in the third line after the above-mentioned form after the word "subscriptions."

6. That the proviso at the end of *Rule 22* be deleted.

7. That in *Rule 26* the Nomination Form for candidates for Membership be amended to read as follows:—

"We, the undersigned members of the Society of Chemical Industry, hereby propose  
(candidate's name in full) as a fit and proper person to become a member of the Society.

For names only of members }  
recommending the candidate. }

"And I, the said candidate for membership, hereby declare that if elected I will do my best to support the Society and will observe its By-Laws.

(Signature) \_\_\_\_\_ (Profession) \_\_\_\_\_

(Address) \_\_\_\_\_ (Date) \_\_\_\_\_

*Extract from By-Laws (Rules 27, 29, and 30).*

8. That *Rule 27* should read as follows:—

"Each member shall pay an annual subscription of, twenty-five shillings, due on the 1st January in each year; except that any member may pay a life composition fee of twenty pounds in lieu of an annual subscription. He shall also pay on receipt of notice of election an entrance fee of one guinea in addition to his first year's subscription; except that should a member who has paid an entrance fee resign and subsequently seek re-election the Council may dispense with a further entrance fee."

9. That in the second line of *Rule 28* the words after "and" read as follows:—"issued to the members once every year."

10. That *Rule 29* read as follows:—

"No person shall be entitled to the privileges of membership who has not paid his subscription for the current year. He shall not attend meetings nor vote, nor shall he receive gratuitously the publications of the Society. Should the subscription remain unpaid eight months after it is due, the Secretary shall give the member notice that unless payment be made within four months after the date of such notice, his name will be struck off the register. At the expiry of the said period, if the member should still remain in default, the Council may order his name to be removed from the register, whereupon he shall cease to have any right, privilege, or interest in the Society, and the Council may notify the same in the Journal and recover the arrears of subscription at law."

11. That *Rule 30* read as follows:—

"Any member may withdraw from the Society at the end of any year by giving previous notice to the General Secretary of his intention to withdraw; always provided that such notice to be valid must be accompanied by the payment of all subscriptions, if any, which may be due from him to the Society at the date of the notice. By such notice and payment he shall be released from all further liability as a member."

12. That the following Rule be added after *Rule 53* of the present Rules:—

"All communications between Local Sections and Public Bodies or Societies shall be conducted through or be at the discretion of the Council; and no publication shall be issued by any Section to the general public without the previous consent of the Council."

13. That *Rules 54* and *55* be placed after *Rules 34* and *38* respectively, and that the succeeding Rules be renumbered accordingly."

#### ANNUAL GENERAL MEETING.

The Annual General Meeting will be held in Edinburgh on the 18th, 19th, and 20th July next. An outline draft of the programme will appear in the May issue. Tickets of membership will be issued in time for the meeting, and will form, as heretofore, vouchers for visits to works and excursions.

#### BANKERS' ORDERS.

For the convenience of Members, the Treasurer has arranged with the Bankers of the Society that they shall collect subscriptions from Bankers in town and country, and Members, who have not already done so, are invited to fill up and sign the Banker's Order enclosed with the December number of the Journal, which should then be sent to the Honorary Treasurer, Mr. E. Rider Cook, East London Soap Works, Bow, E.



## INTERNATIONAL CONGRESS OF APPLIED CHEMISTRY.

We are requested by the Foreign Office to state that an International Congress, under the patronage of the Belgian Government, will open in Brussels on the 4th August next, in connection with the Antwerp Exhibition, on the subject of applied Chemistry.

The work of the Congress will be divided into four sections corresponding to those of the Belgian Chemists' Association, viz.:—Sugar, Agricultural Chemistry, Food-stuffs, and Biological Chemistry.

Those who desire to attend the Congress should apply to M. Sachs, 68, Rue d'Allemagne, Brussels (Sugar and Agricultural Chemistry), or to M. H. van Laer, 15, Rue de Hollande, Brussels (Adulteration of Food-stuffs, and Biological Chemistry), who will forward full particulars on application.

Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 41 of the bye-laws, the Society has the right of priority of publication for three months of all such papers. Infringement of this bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. EYRE and SPOTTISWOODE, the Society's printers and publishers, to whom all communications respecting them should be addressed.

## LIST OF MEMBERS ELECTED 23rd APRIL 1894.

Barnsley, G., jun., Broomfield, Sheffield, steel manufacturer.

Blackmore, H. S., 206, South 9th Avenue, Mount Vernon, N.Y., U.S.A., chemist.

Coward, Percy, 68, Brundett's Road, Chorlton-cum-Hardy, Manchester, chemist.

Gemmell, G. H., 4, Lindsay Place, George IV. Bridge, Edinburgh, chemist.

Heriot, Jas., 18, Dumbiedykes Road, Edinburgh, mineral water manufacturer.

Johuston, G. L., Kingswood, Kingswood Road, Sydenham Hill, S.E., student.

Johnston, W. E., Kingswood, Kingswood Road, Sydenham Hill, S.E., student.

King, W. Grant, Cornell University, Ithaca, N.Y., U.S.A., chemical engineer.

Lazarus, Jos., Opern Ring 13, Vienna, Austria, civil engineer.

Mitchell, Robt., 4, Marchhall Road, Edinburgh, gas engineer.

Sawyer, Harris E., Chemical Laboratory, Harvard College, Cambridge, Mass., U.S.A., instructor in chemistry.

Skelton, J. R., Thorpe St. Andrew, Norwich, technical chemist.

Swan, David, c/o The Swan Colour Co., Ltd., Petershill Road, Glasgow, colour manufacturer.

Thackray, R. B., Laurel Bank, Newby Wood, Horsforth, Leeds, leather manufacturer.

Topley, Wm., F.R.S., Geological Survey Office, 23, Jernyn Street, London, S.W., geologist.

Uhlig, E. C., Millville, Cumberland Co., N.J., U.S.A., glassworks chemist.

## CHANGES OF ADDRESS.

Baly, E. C. C., 10 London; Ardmore, Ritherden Road, Tooting, S.W.

Barratt, W. S., 10 Moss Side; St. Margaret's Schools, Whalley Range, Manchester.

Bell, Percy Carter, 10 Park Row; 13, Ann Street, Park Row, New York, U.S.A.

Bhaduri, K., 10 Baroda; c/o S. K. Lahiri and Co., 51, College Street, Calcutta, India.

Boulton, T. S., 10 Seven Sisters Road; 14, Freegrove Road, Caledonian Road, N.

Brown, Horace T., F.R.S., 10 Add'son Road; 15, Cromwell Crescent, Earl's Court, Kensington, S.W.

Burnham, J. C., 10 Lewisham; Gunpowder Factory, Kirkee, near Poona, India.

Buttfield, H. V., 10 Clapton; 13, Wellington Road, Bush Hill Park, Enfield, N.

Coste, F. H. Perry, 10 Great Tower Street; 142, Burnt Ash Hill, S.E.

Crossley, L., 10 Liverpool; Beachwood, Arnside, Carnforth.

Davis, A. R., 10 Kuntsford; Lynton, Wellington Road North, Stockport.

Dixon, M. T., 10 Newcastle; P.O. Box 1816, Johannesburg, S.A.R.

Dysen, Herbert, 10 Tamworth; 2, Exchange Place, Middlesbrough.

Homfray, D., 10 South Street; 6, Dartmouth Row, Greenwich, S.E.

Houston, R. S., 10 Glasgow; Briar Villa, Greenlaw Drive, Paisley.

Jackman, E. J., 10 Beckton; c/o London County Council, 40, Craven Street, W.C.

Jenkins, T. H., 10 Manchester; c/o Mrs. Waite, Lindow Street, Lancaster.

Jones, Professor D. E., 10 Stafford; 7, Marine Terrace, Aberystwith.

Lewis, G. T.; Journals to 505, Provident Building, South and Chestnut Streets, Philadelphia, Pa., U.S.A.

Macfarlane, R. F., 10 Heaton; c/o Northumbrian Chemical Co., Ltd., Heworth Shore, Felling-on-Tyne.

McGeorge, A. J.; Journals to 215, West 78th Street, New York City, U.S.A.

McMillan, W. G., 10 Calcutta; Metallurgical Laboratory, Mason College, Birmingham.

Marchlewski, Dr. L., 10 Peru Street; 25, Wellington Street, Higher Broughton, Manchester.

Matos, L. J., 10 Fairmount Avenue; 850, Union Street, Philadelphia, Pa., U.S.A.

Morgan, J. J., 10 Milton Lodge; Riverdale, Abergavenny.

O'Sullivan, Jas., 10 Spring Terrace; 26, Ashley Road, Burton-on-Trent.

Ray, Wm., 10 Sloane Street; 141, Duke Street, Chorlton Road, Manchester.

Rideal, Dr. S., 10 St. George's Hospital; 28, Victoria Street, Westminster, S.W.

Roberts, Ernest H., 10 Wicklow; 15, Herndon Road, East Hill, Wandsworth, S.W.

Roberts, J. H. M., 10 Trinity Square; 14, St. James' Road, Brixton, S.W.

Rouillard, R. A., 10 Dundonald Road; 2, Springfield Road, Wimbledon.

Smith, A. J., 10 Pittmoor; 7, Carwood Road, Sheffield.

Smith, Jas. F., 10 Stratford Road; 240, Brunswick Road, Sparkbrook, Birmingham.

Sowerby, W. M., 10 Runcorn; 12, Upper Queen's Terrace, Fleetwood, Lancashire.

Strangman, J. Pim, 10 28; 38, Rue Desbordes-Valmore, Passy, Paris.

Thorpe, Professor T. E., F.R.S., 10 South Kensington; 61, Ladbroke Gardens, Kensington, W.

Wates, E. A., 10 St. John's; Secunderabad, Deccan, India.

Wells, J. G., 10 St. Paul's Street West; Selwood House, Slobnall Street, Burton-on-Trent.

Whiteley, R. Lloyd, 10 Bowers Avenue; 239, Woodborough Road, Nottingham.

Wilson, C. J., 10 Little Queen Street; 14, Old Queen Street, Westminster, S.W.

#### CHANGE OF ADDRESS REQUIRED.

Stephens, H. O'R., 10 20, Brunswick Gardens, Kensington, W.

#### CHANGE OF NAME.

Jones, Herbert, to Sefton-Jones, Herbert, 21, Mincing Lane, E.C.

#### Death.

Bell, H. S., St. Anne's Park Villas, Wandsworth, S.W.

## London Section.

THE CHEMICAL SOCIETY'S ROOMS, BURLINGTON HOUSE, W.

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*Vice-Chairman: W. Crowder.*

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W. G. Blagden.  
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T. A. Lawson.  
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*W. Ramsay.*

*F. G. Blair Roberts.*  
A. Gordon Salamon.  
*G. N. Stoker.*  
*F. Napier Sutton.*  
T. Tyrer.  
Frank Wilson.  
C. R. Alder Wrixlit.

*Hon. Local Secretary: John Heron,*  
74, North Side, Clapham Common, S.W.

The names in italics are those of Members of Committee who retire at the end of the current session.

The following have been elected to fill the vacancies, and will take office in July next:—*Chairman: B. E. R. Newlands. Committee: E. Grant Cooper, H. ce Mosenthal, E. O'Neill, S. Rideal, and A. Shearer.*

SESSION 1893-94.

#### REMAINING MEETINGS.

May 5th:—

Mr. Claude Vautin. "The Commercial Electrolysis of Fused Salts."  
Messrs. W. H. Stanger, M.I.C.E., F.C.S., and B. Blount, F.I.C., F.C.S. "The Testing of Hydraulic Cements."

May 21st:—

Mr. W. Gowland, A.R.S.M., F.I.C., F.C.S. "A By-product obtained in treating Copper with Lead."  
Mr. Oscar Guttman, M.I.C.E. "The Manufacture of Smokeless Powder."

June 4th:—

Mr. W. G. McMillan:  
(a.) "Experiments on the Strength of Leather."  
(b.) "Note on the Colour of Brass."  
Dr. P. Dvorkovitch. "Distillation of Peat."

*Meeting held Monday, April 2nd, 1894.*

MR. WM. THORP IN THE CHAIR.

#### COLORIMETRIC ANALYSIS, OR COLOUR AS A MEANS OF QUANTITATIVE ESTIMATION.

BY JOSEPH W. LOVIDOND.

I HAVE the honour to bring under the notice of this Society some observations bearing on the variation of colour as a means of quantitative estimations.

The object of the paper is an attempt to measure the relation between changes of colour in substances, and their causes, whether the causes be of a chemical, or of a physical nature, such as changes of density, temperature, &c., or of qualities too subtle to be detected by the balance, for example amongst others, the fading of dyes, and changes in organic substances.

When either of these changes is accompanied by a change of colour, then, under conditions which are to be defined, the changes of colour can be measured, and the variations used to define quantitatively the cause of the change.

The method is founded on the fact that selective colour absorption is constant in a given substance so long as the physical and other conditions remain unchanged.

The successful use of this new power of investigation depends on the co-incidence of the following conditions :—

- 1st. A normal light.
- 2nd. A common standard of colour for reference.
- 3rd. A normal vision.
- 4th. A substance having a characteristic colour.

#### A Normal Light.

may be defined as a light made up of the six spectrum colours in equal proportions. Such a perfect light is not available under ordinary conditions, but as this class of colour work can only be carried on in daylight, we have no option but to accept daylight for the purpose. We can, however, ascertain the limits of luminous intensity, and of colour in the light, under which uniform measurements can

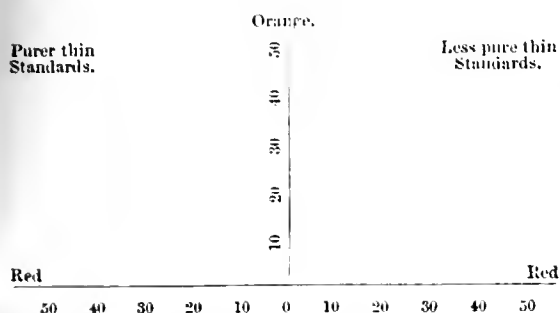
be made, and when the daylight is outside these limits, to a certain extent modify the excess of luminosity by a suitable shading, or collect low lights by suitable reflections. All the work hitherto done has been under these conditions, and without noticeable variations, there is as yet little reason for suspecting that diffused daylight will not meet all practical requirements.

#### A Common Standard of Colour

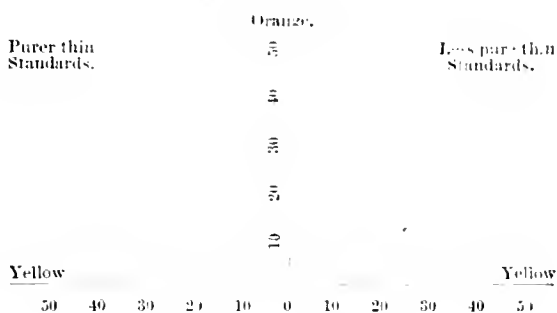
is found in the standard red, yellow, and blue glass colour scales which were brought under your notice two years ago.

It was then shown how, by grading the power of selective colour absorption of suitably selected coloured glass into equal units of colour equivalence, a beam of diffused daylight can be analysed into its six component colour rays, and how pigmentary colours can be measured, their colour value numerically expressed by a scientific nomenclature, and their position found on one or another of the six colour charts on the wall.

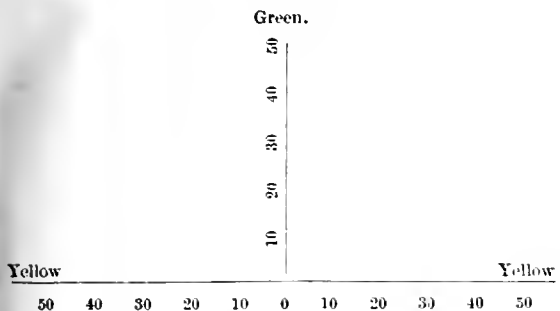
RED ORANGE CHART.



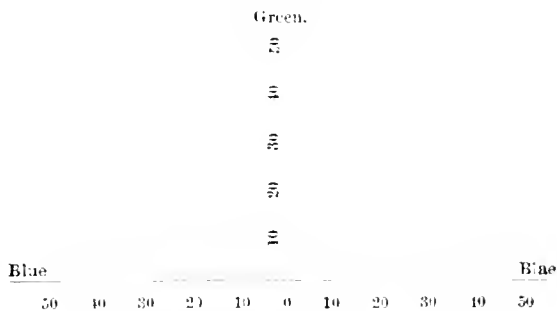
YELLOW ORANGE CHART.



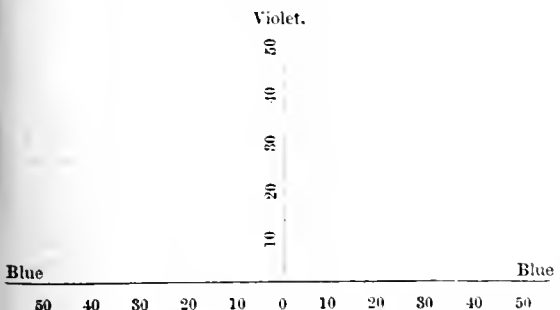
YELLOW GREEN CHART.



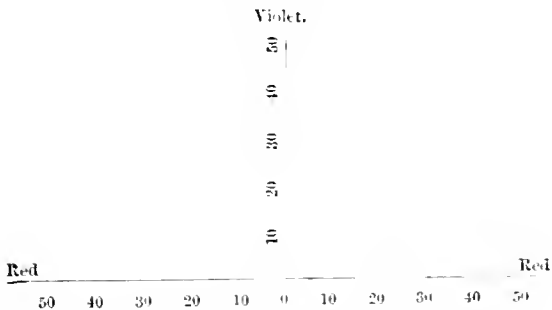
BLUE GREEN CHART.



RED VIOLET CHART.



BLUE VIOLET CHART.



NOTE.—These charts, together with the set of colour circles, have already been before this Society, and are only inserted here for reference.

The illustration by a series of colour circles of the tri-chromatic nature of the red, yellow, and blue, together with

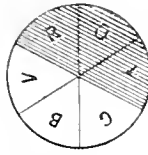
the monochromatic character of the orange, green, and violet standard colours.

RED GLASS.		YELLOW GLASS.		BLUE GLASS.	
Absorbs.	Transmits.	Absorbs.	Transmits.	Absorbs.	Transmits.
Yellow	Violet	Blue	Orange	Red	Green
Green	Red	Violet	Yellow	Orange	Blue
Blue	Orange	Red	Green	Yellow	Violet

Red only distinguishable.

Yellow only distinguishable.

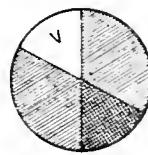
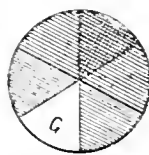
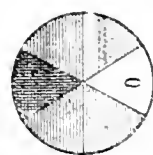
Blue only distinguishable.



Red and yellow glass combined transmit only orange.

Blue and yellow glass combined transmit only green.

Blue and red glass combined transmit only violet.



The work of the last two years has confirmed the truth of the principles upon which the system is based, but the subdivision of the units in the colour scales has been extended. A trained vision can now differentiate the units of each scale into hundredths up to 4.

Hundredths up to 4.	
Twentieths between 1 and 6.	
Tenths	6 " 8.
Fifths	8 " 11.
Halves	11 " 20.

making 493 distinct colour sensations in a single scale of 20 units intensity.

Although there are but three colour scales to begin with, we can by their combinations of two, and of three, produce a variety of other colour scales containing, in fact, all the colours to which the vision is sensitive.

The light available this evening is the electric arc, modified by a screen to approximate the intensity of a working diffused daylight, although this light is not in accord with the normal daylight, by means of which the glasses were graded. It is sufficiently near to illustrate their power of light and colour analysis, by resolving the light into its component colours.

The first illustration is a glass standard spectrum, there are intervals of five units between the shades, whose intensities are 20 units, a continuous spectrum made by all the sensations producible by the standard glasses would be more than 92 feet long.

The whole beam is resolveable by the glasses into five groups, the variations within the groups being of degree only.

Group 1 contains the colours transmitted by a single glass standard, and is divisible into three scales.

Red contains 493 separate colour sensations.

Yellow	493	"	"
Blue	493	"	"

1479 Total colour sensations in the group.

The rays producing the colours of this group are structurally trichromatic, but visually monochromatic. The colour sensations are all chartable on the horizontal lines of their respective colour charts.

Group 2 contains the colours transmitted by two equal glass standards, which are the normals of the system in contradistinction to the standards of Group 1. It is divisible into three scales.

Orange contains 493 separate colour sensations.

Green	493	"	"
Violet	493	"	"

1479 Total colour sensations in the group.

The rays producing these sensations are monochromatic, both structurally and visually, being the only ray transmitted in common by equal pairs of standards. The colour sensations are all chartable on the perpendiculars of their respective colour charts.

Group 3. Colours transmitted by two unequal glass standards divisible into six scales.

Red Orange contains 84,424 separate colour sensations.

Yellow	84,424	"	"
Yellow Green	86,424	"	"
Blue	86,424	"	"
Blue Violet	86,424	"	"
Red	86,424	"	"

518,544 Total colour sensations in the group.

The rays producing these sensations are bichromatic both structurally and visually, and are chartable between the lines of their respective colour charts according to the proportions of the two composing colours.

Group 4 contains the light transmitted by three equal glass standards. It has but one scale, which is simply gradations of neutral gray, ranging in intensity from the smallest reduction of the original light to the black of total absorption.

The scale contains 493 separate sensations, which are structurally hexachromatic and visually colourless.

When colour is accompanied by this absorption of light the colour appears dingy in proportion to the loss of light, producing an effect similar to the mixture of pigmentary black with artists pure colours. I therefore propose to call the units of this scale "black units" when accompanied by the colour of one of the other scales.

As the charts are only constructed to define two colour sensations on any given point, the value of black units accompanying colour must be written in numerals near the colour point.

Group 5.—Colours transmitted by three unequal glass standards. This group consists of the colours of groups 1, 2, and 3, in combination with the black units of group 4.

The rays producing the sensations are either quadachromatic or quinquachromatic structurally, and may be described visually as creating two colour sensations and one light sensation.

The group contains 61,004,491 separate sensations, comprising every colour, no matter how complex, which is less pure in colour than the glass standards.

TABLE OF SEPARATE COLOUR SENSATIONS in a BEAM of NORMAL DAYLIGHT of 20 N.T. UNITS in INTENSITY.

Group 1 contains	1,479
" 2 "	1,479
" 3 "	518,544
" 4 "	93
" 5 "	61,004,496
<hr/>	
61,526,491 Total sensations.	

Large as this number is every colour has a separate numerical value, which is represented by a combination of one, two, or three of the 450 standard glasses in the box on the table, each colour can be made evident to the vision by simply viewing a normal light through the suitable combination, and has a position on one of the six colour charts.

The value of colour transmitted by a single glass is correctly indicated by the unit number it bears, but the colour transmitted by a combination of two or three glasses is not directly represented by the numbers, the colour so transmitted being the remainder after their mutual absorption.

This remainder can be obtained by means of an equation the first half of which represents the numbers on the glasses and the second half determines the colours they transmit.

For example a 1 per cent. solution of alkaline litmus in a  $\frac{1}{8}$ -in. stratum was matched by—

Standard Is.		Coloured Light transmitted.	
Red.	Blue.	Violet.	Blue.
0.90 +	8.0 =	0.9 +	7.1

This colour is a blue-violet in the proportions of the second half of the equation.

A 2-in. stratum of the same solution was matched by—

Standard Glasses.			Coloured Light transmitted.	
Red.	Blue.		Violet.	Red.
33.0 +	13.5 =		13.5 +	29.5

being a red-violet in the proportion of the second half of the equation.

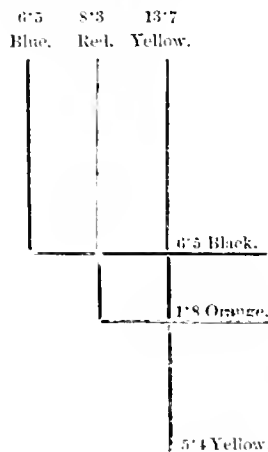
The colour of crude sewage after 20 minutes standing as recorded by the curves was matched by—

Standard Glasses.			Colour of Light transmitted.		
Red.	Yellow.	Blue.	Black.	Orange.	Yellow.
8.3 +	13.7 +	6.5 =	6.5 +	1.8 +	5.4

being a yellow orange in the proportion of 5.4 yellow to 1.8 orange dulled by 6.5 black.

Another way of representing the light transmitted is to draw three parallel lines in length proportionate to the colour of glasses used, then intersect these lines at their terminals, the sections intersected will in each case represent the quality of coloured light transmitted.

These methods hold good for any combinations.



#### A Normal Vision.

The usual tests for colour-blindness do not define a normal vision sufficiently close for quantitative colour work, as without being colour blind in the usual acceptance of the term, some visions vary in their degree of perception for one or more colours.

The method adopted to measure these minor abnormal variations, was to compare the readings of a number of persons at regularly increasing densities of the same coloured liquid, then by plotting the readings of each observer into separate curves on a diagram, the ordinates of which represent the colour units, and the abscissæ the increasing density by strata thickness.

When the curves of all the observers coincide, then all the visions are considered normal, but if one deviates from the others, then the deviating vision is considered abnormal for that particular colour, the amount of deviation being in fact a quantitative test of great delicacy for degree of abnormal colour perception.

The two diagrams containing the colour curves of one per cent. solutions of alkaline and acid litmus afford an instance.

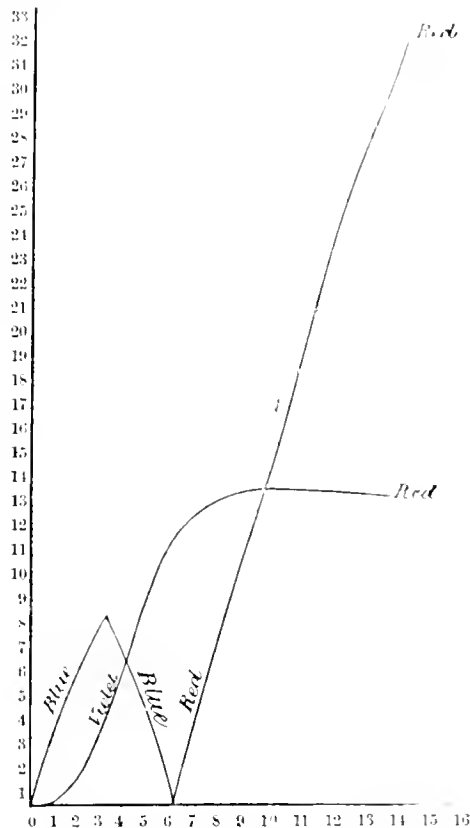
The observations were made by four independent observers, in thicknesses increasing by  $\frac{1}{8}$  in. from  $\frac{1}{8}$  in. to 2 ins.

The normal curves of the alkaline litmus chart are drawn through the coincident curves of A, B, C, and D.

#### ABSORPTION CURVE OF ALKALINE LITMUS.

1 per cent. Four observers, A, B, C, and D.

Colour Units.



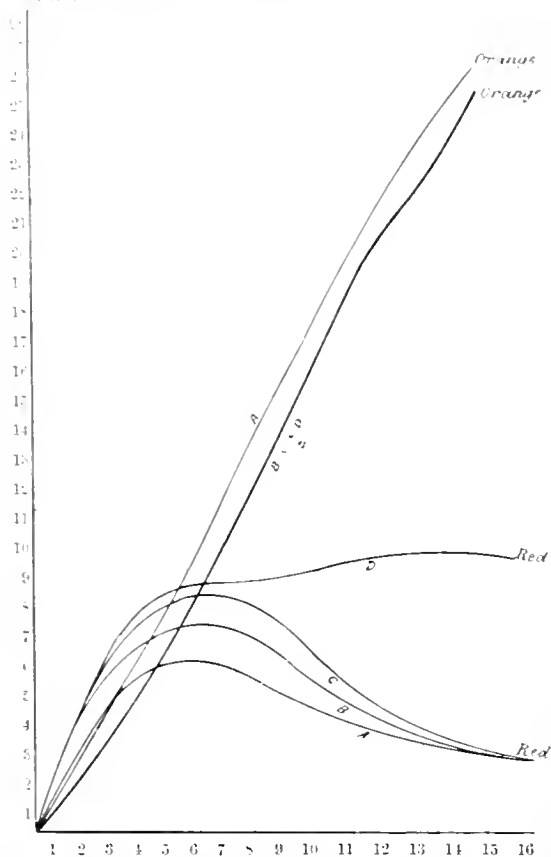
observers. Tracing these curves from zero the four visions are normal for the different combinations of blue violet through the violet-blue until the blue dies out in favour of red, they continue normal for the different degrees of violet-red, and red-violet throughout the whole course of the curves.

In the acid litmus chart the orange normal is drawn through the curves of B, C, and D; A showing an increased power of perception for this colour. A is therefore considered abnormal for orange. A also deviates from the other visions in having a lesser perception for red. A is also considered abnormal for red in this combination with orange, and his observations rejected in these colours for common reference.

It will be noted that whilst the four visions are abnormal for red in the red-orange combinations of the acid chart, they coincide for red in the red-violet combinations of the alkaline chart, pointing to a kind of selective colour-blindness for red according to the colour with which it is associated.

## ABSORPTION CURVE OF ACID LITMUS.

1 per cent. Four observers, A, B, C, and D.  
our Units.



A similar divergence in the red curves of B and C occurs in this chart at the respective colour depths of 6.8 and 8.8 units, whilst from the latter point the perception of D for red slightly increases, maintaining a wide line of deviation from the others, all of which decline in a somewhat symmetrical form until they unite at the depth of  $2\frac{1}{2}$  units.

The colour changes caused by the selective colour absorption of different thicknesses of alkaline, and of acid litmus is shown by their differences of colour in the two taper vessels containing the solutions which vary from zero to 2 ins. The coloured glasses used by the four visions in constructing the curves are shown separately in bands between the taper vessels. The first band containing the glasses which produce the alkaline normal, the four bands below contain the glasses used by each separate vision for the deviations of the acid chart.

As the observations for these two charts were made for the purpose of establishing a normal of colour perception for quantitative estimations, they were carried beyond the 8 units intensity below which a vision can distinguish to  $\frac{1}{17}$  of a unit. Such divergencies as B, C, and D above this depth seldom affect their normals below.

The colour curves of all the diagrams are the normals of my own staff, possibly a wider range of observations may necessitate some slight alterations.

The possession of an abnormal vision in this modified form does not necessarily disqualify the possessor for quantitative colour work, it only means that his work is not available for common reference, he can, by constructing his own curves of vision do quantitative colour work.

The personal equation of the observer sometimes causes minor deviations which are recognised by their more or less regularly alternating on either side of the true normal,

deviations from this cause are generally too small and too evenly distributed to interfere with the plotting of the true curve.

Deviations from normal having for cause an inaccurately gauged glass vessel, or a glass standard out in colour equivalence would be self-detecting, being necessarily recurrent when the faulty vessel or glass standard is used.

## A Characteristic Colour.

All work with colours done up to the present time points to a law of selective colour absorption for definite substances as rigid as the law of specific gravity and other physical constants, but the changes of colour may be brought about either by known physical changes of condition, or by unknown conditions which are accompanied by changes of practical importance in co-relation with changes in the colour values.

In order to define the specific colour absorption of a given substance in the form of a curve, which shall be available for reference in making quantitative estimations of unknown percentages, a series of colour measurements of regularly increasing thickness are made by direct experiment. This may be effected by a separate weighing for each percentage, or, by one weighing of the smallest percentage in the series, then increasing the proportions by a regular increase in the strata thickness of the vessels in which the measurements are made.

The several percentages, together with the strata thickness of the vessels, are marked on the horizontals of the curve chart, whilst the perpendiculars are divided into the units and fractions of units of the colour equivalent scales.

The four conditions previously described as necessarily coincident for making quantitative estimations having been complied with, I now give the full detail observed in producing the lead sulphide curves.

## Lead Sulphide Absorption Curves.

The measurements for these curves were made under the supervision of Dr. Bedson, at the Durham College of Science, Newcastle-on-Tyne. The solution contained 0.25 mgrs. lead per 50 cc. of water. The curves are plotted to a scale of one colour unit per inch.

The normal is formed by the co-incident curves of three observers, and the variations in density were obtained by a regular increase in strata thickness at  $\frac{1}{2}$  in. intervals, ranging from  $\frac{1}{2}$  in. to 2 in. The results are arranged in the following table where the—

- 1st column represents the regular increase of lead by strata thickness.
- 2nd column the colours in degrees of each stratum thickness.
- 3rd column the proportion of lead denoted by each colour when measured in the  $\frac{1}{2}$  in.
- 4th column the proportion of lead denoted by each colour when measured in the 2 in.

## Lead Sulphide Percentage for Colour Table.

Colour Points of Curves.		Percentage of Lead if Measured	
Orange.	Yellow.	In a $\frac{1}{2}$ in. Stratum.	In a 2 in. Stratum.
0.14 +	0.16	0.002	0.00025
0.26 +	0.13	0.003	0.000375
0.39 +	0.14	0.004	0.0005
0.5 +	0.15	0.005	0.000625
0.63 +	0.16	0.006	0.00075
0.75 +	0.19	0.007	0.000875
0.87 +	0.24	0.008	0.001
0.99 +	0.30	0.009	0.001125

## Lead Sulphide Percentage for Colour Table—cont.

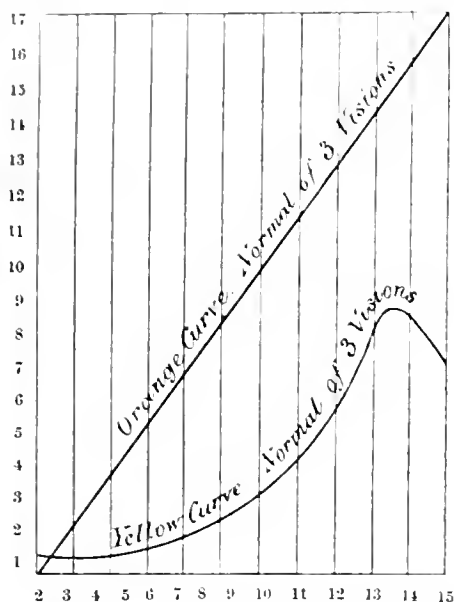
Colour Points of Curves.		Percentage of Lead if Measured	
Orange.	Yellow.	In a $\frac{1}{2}$ in. Stratum.	In a 2 in. Stratum.
1.0 +	0.35	0.010	0.00125
1.22 +	0.47	0.011	0.001375
1.35 +	0.60	0.012	0.0015
1.46 +	0.80	0.013	0.001625
1.58 +	0.82	0.014	0.00175
1.7 +	0.70	0.015	0.001875

To find the proportion of lead in a solution of lead sulphide of unknown percentages. If it contains more than 0.25 mgr. lead in 50 cc., it must be measured in the  $\frac{1}{2}$  in. cell, the proportion of lead will be found in the second column opposite the corresponding colour in the first column.

If the solution contains less than 0.25 mgr. lead in 50 cc., then it must be measured in the 2-in. cell, the proportion of lead will then be found in the third column opposite the corresponding colour in the first column.

## LEAD SULPHIDE ABSORPTION CURVE.

Colour Units.



It is obvious that having constructed the colour absorption curves for a definite compound substance, the method is applicable to a quantitative estimation of the whole compound, or to any definite part of it.

For instance, it is only necessary to supplement the percentage of lead column by percentage lead sulphide and sulphur columns to arrive at definite results in each case.

## Potassium Permanganate.

The absorption curves and resultant tables for estimating the quantity of permanganate and of oxygen consumed in an analysis were those of a solution prepared as follows:—0.395 grm. potash permanganate made up to 1 litre as a standard solution.

10 cc. of this standard solution added to 250 cc. of water and 10 cc. dilute sulphuric acid.

The curves are the mean of four normal observers.

## CURVES AND TABLES SHOWING THE QUANTITY OF POTASSIUM PERMANGANATE and of OXYGEN consumed in WATER ANALYSIS.

STANDARD SOLUTION, 0.395 grm. per litre.

Solution for curves, { 250 cc. ammonia free water,  
10 cc. dilute sulphuric acid,  
10 cc. standard solution of permanganate.

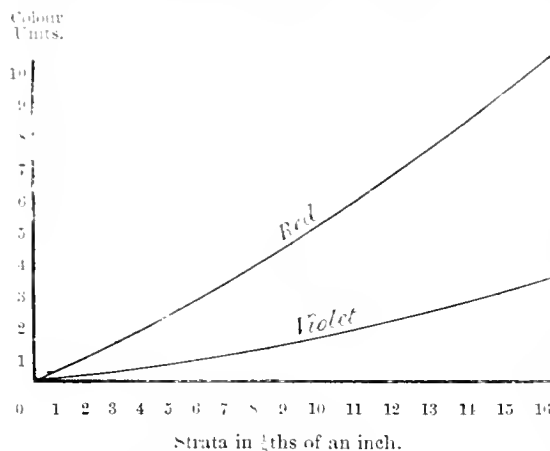
Colour Points of Curve.		If measured in the 2 in. Stratum.	
Violet.	Red.	Potassium Consumed.	Oxygen Consumed.
0.0	0.0	0.0	0.0
0.06	0.2	0.75	0.075
0.13	0.41	0.5	0.05
0.18	0.62	0.25	0.025
0.23	0.83	0.0	0.0
0.3	1.07	8.75	0.875
0.37	1.31	8.5	0.85
0.41	1.52	8.25	0.825
0.5	1.75	8.0	0.8
0.56	1.98	7.75	0.775
0.61	2.21	7.5	0.75
0.69	2.43	7.25	0.725
0.74	2.67	7.0	0.7
0.8	2.9	6.75	0.675
0.88	3.13	6.5	0.65
0.94	3.4	6.25	0.625
1.01	3.65	6.0	0.6
1.1	3.9	5.75	0.575
1.16	4.13	5.5	0.55
1.22	4.37	5.25	0.525
1.3	4.62	5.0	0.5
1.38	4.9	4.75	0.475
1.45	5.15	4.5	0.45
1.55	5.43	4.25	0.425
1.6	5.68	4.0	0.4
1.7	5.92	3.75	0.375
1.78	6.21	3.5	0.35
1.86	6.42	3.25	0.325
1.94	6.70	3.0	0.3
2.01	6.95	2.75	0.275
2.1	7.22	2.5	0.25
2.19	7.5	2.25	0.225
2.28	7.78	2.0	0.2
2.35	8.04	1.75	0.175
2.43	8.3	1.5	0.15
2.5	8.61	1.25	0.125
2.6	8.9	1.0	0.1
2.69	9.16	0.75	0.075
2.77	9.41	0.50	0.05
2.84	9.7	0.25	0.025
2.9	9.95	0.0	0.0



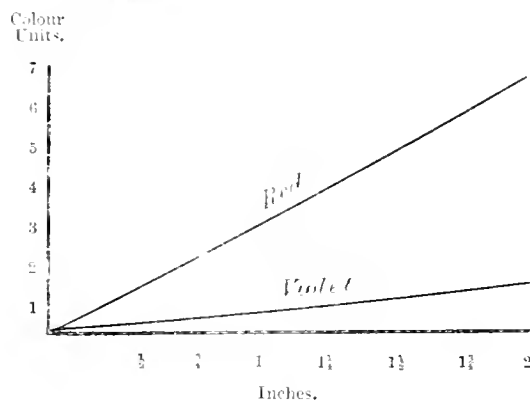
The first column contains the quantities of violet and red at 40 equidistant points of the curves.

The second column contains the cubic centimetres of permanganate consumed, and the third column the milligrammes of oxygen consumed by opposite corresponding colour number to that which may be found in an analysis by heating the mixture of water and permanganate to 27° C. for 15 minutes and for four hours in the usual way.

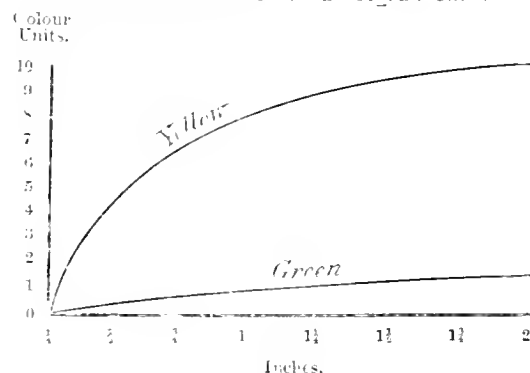
COLOUR CURVES OF 10 CC. STANDARD SOLUTION OF POTASSIUM PERMANGANATE ADDED TO 250 CC. WATER AND 10 CC. DILUTE SULPHURIC ACID.



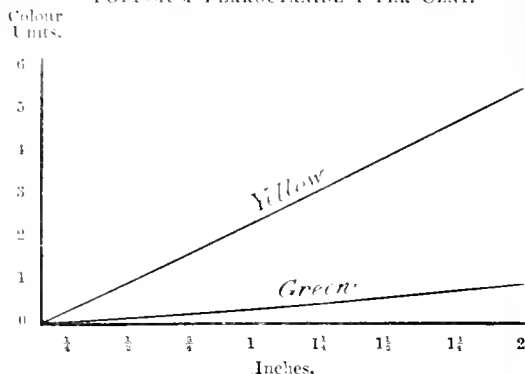
POTASSIUM PERMANGANATE '001 PER CENT.



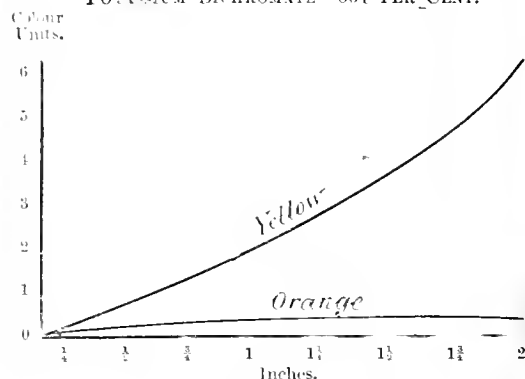
POTASSIUM FERROCYANIDE '01 PER CENT.



POTASSIUM FERROCYANIDE 1 PER CENT.



POTASSIUM BICHROMATE '001 PER CENT.



COLOUR ABSORPTION CURVES.

#### Copper Sulphate.

The variations of colour for density in these curves are too small for practical service, it would have been better measured as ammonia copper sulphate when the variations of colour for density are about six times greater. The open curve of this chart represents the degree of colour purity of copper sulphate in excess of the glass standard colours, that is, the solution was dulled by the values indicated on the curve by intercepting the light with neutral tint units.

I call attention to the curves of the 2 per cent. solutions of roasted malt marked respectively No. 7 and No. 10. The market value of No. 10 is one-third greater than No. 7. Their colour depths up to a  $\frac{3}{8}$  in. stratum practically coincide, after which the cheaper sample No. 7 becomes the darkest as having more orange, the dearer sample being characterised by an increase of yellow. This seems to suggest that depth of colour alone is not an infallible guide to fine quality in the caramels of roasted malts, and that the yellow preponderance is an important factor.

The curves of the two samples of log-wood is an illustration of measurement of dye value. The method is applicable to any kind of dyes either in the liquid or solid form.

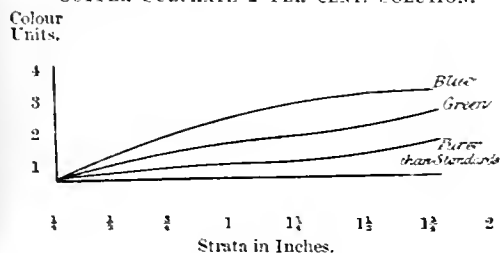
Percentage Table of Copper Sulphate Curve.

Colour Points of Curve in a 2-in. Stratum.		Per Cent. of Copper Sulphate.
Green.	Blue.	
0'00 +	0'16 denotes	0'05
0'17 +	0'3 "	0'1
0'28 +	0'42 "	0'15

## Percentage Table of Copper Sulphate Curve—cont.

Colour Points of Curve in a 2 in. Stratum.		Per Cent. of Copper Sulphate.
Green.	Blue.	
0.32 +	0.54 denotes	0.2
0.4 +	0.68 ..	0.25
0.47 +	0.8 ..	0.3
0.54 +	0.89 ..	0.35
0.61 +	1.03 ..	0.4
0.69 +	1.13 ..	0.45
0.73 +	1.23 ..	0.5
0.8 +	1.34 ..	0.55
0.87 +	1.43 ..	0.6
0.91 +	1.53 ..	0.65
0.98 +	1.62 ..	0.7
1.03 +	1.7 ..	0.75
1.07 +	1.8 ..	0.8
1.14 +	1.89 ..	0.85
1.2 +	1.97 ..	0.9
1.27 +	2.03 ..	0.95
1.31 +	2.11 ..	1.0
1.39 +	2.18 ..	1.05
1.4 +	2.25 ..	1.1
1.5 +	2.31 ..	1.15
1.57 +	2.39 ..	1.2
1.62 +	2.48 ..	1.25
1.7 +	2.54 ..	1.3
1.79 +	2.60 ..	1.35
1.83 +	2.68 ..	1.4
1.9 +	2.75 ..	1.45
1.96 +	2.83 ..	1.5
2.02 +	2.91 ..	1.55
2.1 +	2.99 ..	1.6
2.18 +	3.05 ..	1.65
2.24 +	3.13 ..	1.7
2.31 +	3.2 ..	1.75
2.4 +	3.3 ..	1.8
2.5 +	3.39 ..	1.85
2.58 +	3.48 ..	1.9
2.67 +	3.57 ..	1.95
2.75 +	3.67 ..	2.0

## COPPER SULPHATE 2 PER CENT. SOLUTION.



## Colour in Water.

In the examples already given we have as bases for comparison known chemical substances quantitatively prepared by means of the balance, but in measuring the colour and turbidity in water we have not these constants for comparison, and must therefore proceed in a tentative manner in order to obtain what information the system is capable of yielding.

If the water is clear it may be measured directly for colour, and in interpreting the value of a colour, consideration must be given to the source from which the water was obtained, as, if anything approaching the yellow orange of a peat water, or the yellow-green of a river water, is found in water from gravel or chalk, it is presumptive evidence of contamination.

Mr. Parry, of Liverpool, who has used the method for some years, informs me that the first intimation of changes of condition in the water of Lake Vrynwy is sometimes given by changes of colour in the daily record of colour measurements.

## Solid Matter in Suspension.

When a water contains solid matter in suspension it must be measured both before and after filtration;\* the loss of colour by filtering represents the light obstructed by the solid matter in suspension.

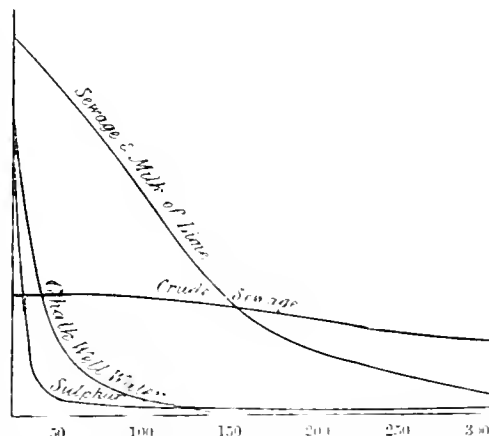
The following example is that of a water supplied to Newcastle-on-Tyne during the partial water famine of last summer. The measurements were made by Mr. Pattinson (who is sanitary inspector for that city) at his laboratory:—

	Black.	Orange.	Yellow.
Colour of water before filtration	2.6 +	1.0 +	3.4
" after "	1.6 +	0.4 +	3.4
Loss of colour by filtration	1.0 +	0.6	..

It is essential to find some substance of known specific gravity, whose variations in rate of deposit by time may be recorded in terms of obstructed light. The curves formed by these variations would be available for comparison with the light obstruction curves of unknown substances.

The curves in the deposit chart represent some preliminary work in this direction. The two substances here used as possible standards are: 1st. A 1 per cent. mixture of finely-divided sulphur and water; 2nd. The deposit of carbonates in a well water after boiling. The water contained 20 grains of carbonates per gallon.

## CHART SHOWING COMPARATIVE RATES OF DEPOSIT BY TIME.



\* NOTE.—At least 10 minutes must elapse after filtering before measuring, as the act of filtering causes a temporary loss of colour.

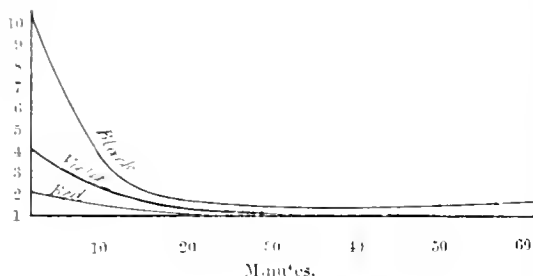
The unknown turbid solutions are crude sewage, and a portion of the same sewage treated with 1 per cent. of milk of lime. The variations in their rates of deposit is shown by the variations of the curves on the charts.

The black curves which in the main represents the light obstructed by the solid matter are brought together in a single chart to afford a better comparison.

The remarkable changes of colour during the liberation and deposit of sulphur in a 1 per cent. solution of soda hyposulphite and dilute hydrochloric acid are illustrated by the curves on the chart: the actual glasses used are placed in a frame for examination.

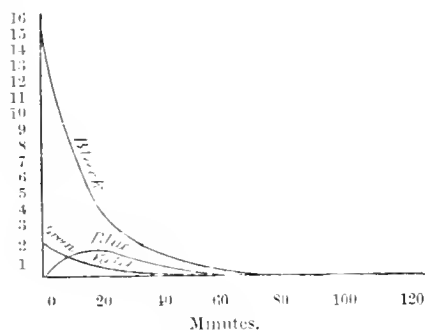
#### ONE PER CENT. OF SULPHUR IN DISTILLED WATER.

Colour Units.



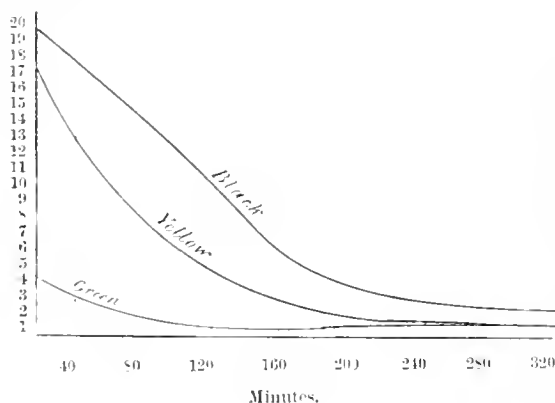
#### CHALK-WELL WATER. BEILED RATE OF DEPOSIT IN A 2 FT. STRATUM.

Colour Units.



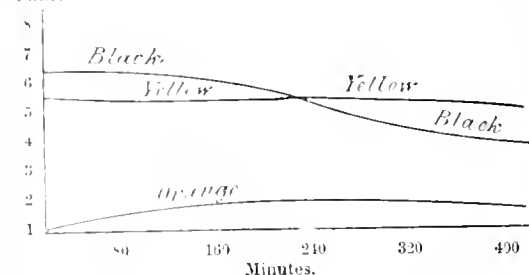
#### CRUDE SEWAGE TREATED WITH 1 PER CENT. OF MILK OF LIME. MEASURED IN 2 FT. STRATUM.

Colour Units.



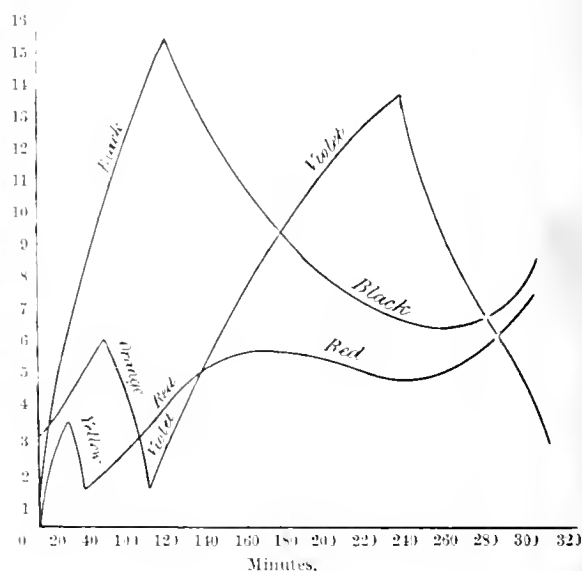
#### CRUDE SEWAGE. RATE OF DEPOSIT IN 2 FT. STRATUM.

Colour Units.



#### COLOUR CURVES OF 1 PER CENT. SOLUTION OF HYPOSULPHITE OF SODA IN DILUTE HYDROCHLORIC ACID WHILST DEVELOPING AND DEPOSITING SULPHUR.

Colour Units.



#### DISCUSSION.

The CHAIRMAN said that Mr. Lovibond's paper opened up a field for much further investigation. The most important point in the use of the tintometer was the permanence of its record.

They were all accustomed to more or less careful comparisons of colours, but beyond saying that one colour was like another they had no permanent record. If it were necessary to reproduce the same colour everything depended on the permanence of that selected as the standard. With these standard glasses, about whose permanence there could be very little doubt, if the same glasses were put together again, at whatever interval of time, the same colour would be obtained for comparison. He was not quite sure that he went quite all the way with Mr. Lovibond in some respects. For example, in such a case as lead sulphide the standard could be easily reproduced at any moment; a standard lead solution could be kept for an indefinite period, or could be at any time re-produced by means of the balance, and there he was not sure that the tintometer offered any great advantages. On another point he would ask for a little further information. Mr. Lovibond appeared to trust to his glasses as a means of analysing the constituent elements of a definite colour, and to say that a colour consisted of so much yellow, so much green, and so much blue, &c. But the same colour might be produced by a different arrange-

ment of constituent colours, and it was not quite fair to say because a certain colour was produced by so much green, so much blue, and so much yellow, that it contained these constituents, because one might have an exactly similar colour produced by another arrangement. Perhaps his physics might be defective and belonged rather to the old school, but he should have thought the proper way to state it was that the given colour was the colour of light of a given wave length, and that this method of mixtures was not quite scientifically accurate.

Dr. S. RIDEAL said Mr. Lovibond was to be congratulated on the progress he had made. He had now for the first time a means of standardising his glasses. In his previous papers he had never given any exact information by which glasses could be standardised in the laboratory, but by means of the diagrams he had prepared, one could ascertain whether glasses purchased from him to use with a tintometer were keeping their constant colour. Bichromate or other coloured solutions could be made up readily by means of the balance to 0.1 or 0.001 strength, and by means of the tables given in this paper chemists could ascertain whether the glasses were remaining permanent or not, since there was now on record the actual value of these glasses in ascertainable and reproducible quantities.

Mr. W. C. YOUNG said sometime back he endeavoured to apply a process of this kind to the determination of the organic matter in water, and for a time it seemed very promising. His experiments were confined to river water, and the standard was one made by a definite quantity of ammonium salt coloured by Nessler's solution. One litre of water was concentrated to 5 cc., all earthy matter was precipitated by sodium carbonate, filtered away, and a clear solution of 5 cc. volume remained. This did very well with river waters, but when he came to apply it to deep well waters, whether from the chalk or the new red sandstone, he found the organic matter in the water gave quite a different colour value, and consequently the method had to be abandoned. He might say also, that the organic matter in moorland water had a deeper colour for a given quantity than either river water or deep well waters.

Mr. D. HOWARD said the old certainty that there were three primary colours which, combined together, made all the others, had now disappeared, and if he might judge from the recent results which Captain Abney had given, it appeared that various combinations might make the same colour. At the same time there was this practical residue of knowledge left, that a definite record of a given colour was a very valuable thing to us, but it could not be too strongly impressed on all those who dealt with colours as a means of quantitative analysis, that the circumstances must be the same. The example which had been mentioned, which was a very admirable one, of the enormous difference which the presence of organic matter made in the colour of sulphide of lead precipitated under otherwise similar circumstances, was only one of those cases which occurred in all study of these matters. Other things therefore being equal, a constant record of colour was of the greatest possible importance, and, as far as he could judge, Mr. Lovibond had given in these glasses a better standard than any other which had been yet made. It must nevertheless be borne in mind that the personal equation came in with tremendous effect. There was not only that remarkable phenomenon colour blindness to be taken account of, but there was the fact that the sensitiveness to shade was extremely imperfectly developed in a great many eyes; and only those could speak with certainty of their judgment on a shade of colour who had trained their eyes, and proved that the training had been of any use. If an untrained person said the two colours were identical it was information of very little value. These two points seemed to be of prime importance—that the circumstances under which the colours were produced must be exactly identical, and that the personal equation must be carefully sought for and guarded against.

Dr. KOHN, Hon. Secretary of the Liverpool Section, said he had had some little experience in dealing with

the determination of lead by colour tests, and he would remind them that in a paper by Mr. Warrington, on the detection of lead in citric and tartaric acids, it was pointed out that a great deal depended on the constituents in the solution, and this would bear out the differences in the results mentioned by Mr. Young. In testing for lead in the presence of citric or tartaric acid, the deficiency of the colour test was immensely greater than with aqueous solutions. That was to his mind the great difficulty in applying the colorimetric tests in the laboratory, for they depended entirely on the conditions of the experiments. He would ask Mr. Lovibond whether, in the table for lead sulphide, which was undoubtedly most useful to anyone using a pure solution of lead, he had made similar tests in the presence of salts of any kind, and if so, what the influence of those salts had been on the colour values.

Mr. E. J. BRYAN said he could confirm what Dr. Kohn had said about the sensitiveness to colour of lead sulphide in the presence of tartaric acid or citric acid. Some time ago he had to determine small quantities of lead in mineral waters, and he found the effects were much increased by the addition of sugar to the solution. The intensity of the black colour due to the lead sulphide was increased about 10 times with the same quantity of lead present.

Mr. BRYAN CONGERY said the advance which Mr. Lovibond had made in this subject was very great, but one of the chief difficulties he found was the question of daylight, and he should like very much to know if Mr. Lovibond had succeeded in making or finding an artificial standard of light to work by.

Mr. BOVERTON REDWOOD said, hitherto the criticisms on this paper, though valuable, had been mainly of a destructive character, but he desired to give expression to his recognition of the importance which attached to the self-denying labours of Mr. Lovibond. This was not the first occasion on which he had taken upon himself to speak in somewhat eulogistic terms of the practical value of the instruments Mr. Lovibond had placed at their disposal. He had had undoubtedly to contend with great difficulties, and it might be that even greater difficulties lay ahead, but those who had followed the work he had done must have been impressed with the painstaking manner in which he had devoted himself to the subject, and there was good reason to hope that, on further investigation, all the points which had been raised would be satisfactorily dealt with; they certainly owed him a cordial vote of thanks, not merely for his paper, but for his labours in connection with this subject, which had now extended over many years. It was longer ago than he cared to remember since Mr. Lovibond first brought him the tintometer, and asked him whether it would be a useful instrument to employ in recording the colour of oils. Since that time it had been in constant use in his laboratory, and they would be sorry to be without it, for it was found to be of great utility.

Mr. B. E. R. NEWLANDS said he was himself quite a disciple of Mr. Lovibond. He believed he possessed the first instrument which had been sent to London, and this had been continuously used ever since. It was easy to criticise this tintometer, and to say that because it only gave an arbitrary standard of colour, and not one constructed on a purely scientific basis, it was of little or no use. He failed to see that entirely, and thought it was of slight importance whether the standard chosen was a scientific or an arbitrary one, so long as it was uniform and unalterable.

Mr. Lovibond had selected a material, viz., glass, which was a most permanent one to make a standard of; and, after spending an enormous amount of time in carefully graduating the colours of various glasses, he had introduced a standard which could be thoroughly relied upon so far as it went. He had much pleasure in seconding the vote of thanks to Mr. Lovibond for the labour he had undertaken, more especially as he was well aware that his work in connection with this invention was not so much with the idea of pecuniary profit as of rendering an important service to his fellow-men.

Mr. E. J. BRAY asked if Mr. Lovibond included in his scheme any method for the determination of ammonia in drinking water so as to compare with Nessler's solution?

Mr. J. W. DINHAM, speaking as a visitor, and connected with the grain trade, had great pleasure in supporting the vote of thanks. Chemistry had gone a great way to analyse grain, particularly barley and wheat, but in the matter of wheat, buyers in Mark Lane went on the question of colour in order to estimate the quality and quantity of gluten. The amount of soluble and insoluble gluten in the grain was important to millers, and the value of that was determined by the colour. Until they had a standard of colour in testing wheaten flour they could not recognise one sample from another, because of the bleaching qualities of the flour if kept as a standard sample. Mr. Lovibond had brought before them the means of estimating the colour, by standard glasses which were constant. It was a constant colour that they required in estimating value of breadstuffs, as colour indicated the development of the tissue of the grain and the quality of the insoluble gluten. They were able by this means to ascertain the quality of the grain, and to estimate the value of the flour manufactured from it.

Mr. E. F. HOOPER asked Mr. Lovibond whether in his investigations on this tintometer he was able to give any idea of the colour value of fluorescent bodies. Those who come in contact with oils would be very glad if they could fix on definite colours.

Mr. J. W. LOVIBOND, in reply, said in reference to the Chairman's suggestion that a colour might be produced in several different ways, he should say emphatically under daylight conditions it could not. As to what might be done by mixing the light of the various parts of the spectrum, his experience was too small to be of any weight, although he had never seen satisfactory results in any lecture he had attended, but with daylight the slightest variation of a single ray altered the colour. The set of glasses were so graded that they defined the slightest alteration, whether in the whole light or a part, and you could not disturb the constituents in any way without disturbing the visual sensation. He had had very little experience with regard to aqueous solutions; he had simply worked out the method of measuring the colour, and could only say that it recorded the slightest change of colour, and he had no hesitation in saying that, if there were a variation in a colour due to different properties of organic matter, the curve of absorption for the variation would show the difference, and in that way the method would be of very great value for estimating the qualities of water. It had yet to be worked out, however, and he had done nothing in that direction, except the instances given. The whole system of curves had been evolved since he gave notice of this paper, which was to have been on very different lines, and it would be seen that very little critical work could have been done except in the examples given. With reference to the influence of salts on lead sulphide, he could only go by analogy, but he should say that so long as the salts were colourless the influence would be nil, but the instant a coloured salt intervened then the system was useless. The colour of the foreign body, whatever it might be, must be eliminated before satisfactory results could be obtained. With reference to the simplicity of the yellow, and its being produced in various ways, these glasses would record the slightest change. No yellow varying from another yellow could be recorded by the same glass. The sodium line he did not regard as a pure yellow. He regarded it as a yellow orange, and any departure from that must be made evident by the glass on observation. Five minutes would convince anyone of the truth of this. The personal equation he granted was an important element, but it was one which could be measured and must be settled before uniform work for reference could be done. The curves in the acid litmus chart defined how near they could go towards settling it; these curves were formed by individuals without any communication with each other. As he pointed out in the alkaline litmus chart, the four coincided in the comparison of records after they were independently made. In the acid chart they varied to the extent shown, that is to say, they coincided at the beginning and at the finish with

one exception. Such a record would be necessary before any work for publication could be done. He believed it would be found that the variation from colour blindness was not so great as was frequently supposed. The personal equation was simply a matter of observation, and, like the balance, you might let it sway a little on one side or the other, but when you had a collection of vibrations on the two sides they balanced each other if it were simply a matter of personal equation. The question of artificial light was a difficult one to deal with, and from his experience he should say it was impossible to do critical colour work with any artificial light. In the first place the rays from artificial light were not in the same condition as diffused daylight. One got a different kind of energy in the intense direct rays from artificial light to what one did from the diffused light after it had been reflected and selectively absorbed backwards and forwards an innumerable number of times. It was quite possible that an artificial light might be made to do some simple kinds of colour work, but one would either have to regrade the present, or to make a separate set of glasses, and to find a different material for colouring them, because the cobalt of blue was peculiarly susceptible to the penetrative character of the red ray of artificial lights. This red ray seemed to have a property of penetration apart from its colour value, which can be neutralised by reflection. In fact, the work done by means of these glasses was quite different to that done by intense light through lenses and after reflection from mirrors. With reference to the value of this method for quantitative work, given the four coincident conditions defined in the paper, whatever the balance could do he could without hesitation pronounce to the millionth of 1 per cent. by this system for lead sulphide evolved under the direction of Dr. Bedson. He had no doubt it could be made available for estimating ammonia in water very rapidly. The first method for this purpose was by means of arbitrary glasses, and that was the one now mostly used in laboratories, where the system was adopted more than 25 years ago, but under the colour equivalent system, some little work would have to be expended upon it, and as he had but one pair of eyes he thought he might fairly ask some of the younger men to undertake such work. For oils it was in constant use, and he was fortunate in having the testimony of Mr. Redwood to its value, but as a matter of fact it was rapidly coming into general use for this purpose, and he thought its utility might be enlarged even to differentiating between the sources of oils. He had no doubt that oils from different sources would show different absorption curves; at any rate if the colouring properties were different this would be so, and when they were similar the curves probably would be similar. In conclusion he thanked the Society for the kind manner in which his paper had been received.

## THE NATURAL DIMINUTION OF THE DISSOLVED ORGANIC MATTER IN THE WATER OF RIVERS.

BY W. C. YOUNG, F.I.C., F.C.S.

*Consulting Chemist to the Lee Conservancy.*

THERE is a wide difference of opinion amongst chemists who have devoted attention to the subject, as to whether the organic matter in solution in the water of a river is diminished by the operation of natural processes during its flow. Some authorities hold that the diminution, from any cause, is extremely minute, whilst others contend that natural oxidation destroys the bulk of the organic matter in solution in the course of a few miles' flow.

Dr. P. Frankland, in a paper on the "Self-Purification of Rivers," read before the Congress of Hygiene, held in London in August 1891, states, as a result of experiments he had made upon the Thames and other rivers, that there was practically no loss of organic matter in solution during the flow of a river. He states, as his opinion, that the

improved appearance of a river, which is always noticeable some distance below the spot at which it receives pollution by sewage, is due to sedimentation, and that oxidation has little or no influence in the improvement. I presume he means by this that the matters in suspension have been deposited on the bed of the river, and that the improvement consists merely in the water having become clearer and brighter.

Perhaps the most marked features in a sewage-polluted stream are its inky colour and offensive odour, the former due to iron sulphide and the latter to sulphuretted hydrogen. There can be no doubt that to a very great extent these are oxidised, and the improvement in colour and odour may therefore be attributed to oxidation alone.

In the spring of 1891 and again in 1892, I had occasion to make a series of analyses of samples of water from the River Lee from its source, above and below each tributary,

down to the intake of the East London Waterworks Company at Ponder's End. The investigations were originally made for the purpose of ascertaining what effect the mixture of water from the tributary streams had upon the composition of the water in the main river but the results also show the variation in the amount of dissolved organic matter in the water after flowing a considerable distance, and should throw some light upon the "oxidation" question. The samples, on each occasion, were taken under the most favourable circumstances, the weather having been fine and dry for two or three weeks previously, so that surface water was entirely absent from the streams and the water therefore in its normal condition. The total dissolved organic matter in each sample was determined by the process described in my paper read before the Society in November 1891.

The results are stated in the following table:—

TABLE No. 1.  
Grains per Gallon.

No.	Source of Sample.	Distance from Point above.	Date.	Total Dissolved Organic Matter.	Date.	Total Dissolved Organic Matter.
1	Source of River Lee.....	—	1891, 14 April	0.511	1892, 6 April	0.569
2	100 yards above River Murrum .....	23 miles	"	0.633	"	0.420
3	" " Beane .....	1 "	21 April	0.672	7 "	0.329
4	Intake, New River Company .....	1 "	"	0.441	"	0.308
5	100 yards above River Ash .....	3 "	"	0.529	8 "	0.497
6	" " Stort .....	1 "	"	0.623	"	0.394
7	100 yards below Waltham Abbey Sewage Farm.	6½ "	"	0.685	9 "	0.490
8	100 yards below Small Arms Factory Sewage Farm.	1½ "	"	0.490	"	0.418
9	Intake, East London Water Company, Ponder's End.	2½ "	"	0.483	"	0.385

The results show, especially in the 1892 samples, that the river, when free from flood water, contains very little organic matter in solution. In the 1891 samples the maximum amount was found in No. 2 (100 yards above the junction with the Murrum), and the minimum in No. 4 (the intake of the New River Company), the difference being 0.252 grain. This diminution appears to be due to the admixture of the water of the River Beane (which joins the Lee between these two points) with the main river, as samples taken at the same time from the Beane and 100 yards below its junction with the Lee, gave 0.252 and 0.469 grain dissolved organic matter per gallon respectively. In the 1892 samples the maximum found was in No. 1 (taken at the source of the River Lee), and the minimum in No. 4 (the intake of the New River Company), the difference being 0.252 grain, the same as in the 1891 samples.

As it is evident that the river receives small additions of organic matter at various points in its course, and is undoubtedly diluted occasionally by spring and river water of greater purity than its own, it is almost impossible to draw any definite conclusion as to the rate at which the dissolved organic matter diminishes, but it is evident, from these results, that there is a tendency toward diminution, although the action appears to be very slow.

I may here mention that the natural spring water in this locality contains an appreciable quantity of dissolved organic matter, the samples from the springs at the source of the Lee in Leagrave Marshes, Bedfordshire, giving rather more than  $\frac{1}{2}$  grain per gallon, and samples taken in April 1891 and 1892 from Chadwell Spring, the New River Head, giving 0.693 and 0.42 grain per gallon respectively; therefore such water mixing with the main river can have but little effect in lessening the proportion of dissolved organic matter.

After the water passes Keid's Weir, Ponder's End, which is the intake of the East London Water Company, there is absolutely no chance of increase of dissolved organic matter

by the addition of polluted water, or of decrease of organic matter by dilution with purer water, so that from this point to the time it reaches the consumer it is exposed to the action of natural processes, and a comparison of the results obtained from the water taken from the East London Water Company's mains with those from the water at the intake, would show the degree of purification effected in this way.

I am informed by the engineer to the East London Water Company that a period of from 18 to 21 days would elapse before the water which has passed Keid's Weir would be delivered at the offices of the Board of Works for the Poplar District, where my laboratory is situated. Accordingly, in order to obtain results fairly comparable with those given by the sample taken at Keid's Weir on April 9, 1892 (No. 9, Table I.), which contained 0.385 grain per gallon, I determined the total dissolved organic matter in the water supplied to my laboratory (taken direct from the main) daily from the 26th April to the 5th May 1892.

The results are given in the following table:—

TABLE No. 11.  
Grains per Gallon.

No.	Total Dissolved Organic Matter.
No. 1 .....	0.28
" 2 .....	0.21
" 3 .....	0.21
" 4 .....	0.21
" 5 .....	0.308
" 6 .....	0.21
" 7 .....	0.371
" 8 .....	0.28
" 9 .....	0.3
Mean .....	0.264

Taking the mean of these results, and comparing it with the 1892 sample from the Water Company's intake, it will

be seen that after the water has travelled a distance of four miles to the reservoirs, been exposed to the air in the reservoirs for about 20 days, flown thence one mile through an open stream to the filters, and passed through about six miles of mains, the organic matter is reduced from 0.385 to 0.261 grain per gallon, or 31 per cent.

These experiments having been made upon water containing only a very minute proportion of dissolved organic matter, I thought it desirable to extend the investigation to highly polluted water, such as sewage effluents, in order to ascertain whether the rate of decrease of dissolved organic matter was the same or not. Accordingly two samples of such water, which had been standing in stoppered bottles in my laboratory for about six months, were placed in open vessels, covered with paper to exclude dust, and frequently shaken so as to renew the supply of dissolved oxygen. At the commencement sample No. 1 contained 4.9 and No. 2 3.6 grains dissolved organic matter per gallon. After 14 days a bright green microscopic growth developed in No. 1, which rapidly increased in amount until, after 42 days, the water was of an intense green colour. It was then filtered through asbestos, the finest filter-paper being useless for the purpose, and the dissolved organic matter determined. This was found to have dropped to 1.7 grains per gallon. The filtered water was again set aside, frequently agitated, and after a further period of 30 days (during which a slight additional growth developed) was found to contain 1.1 grains per gallon.

In the case of sample No. 2, which at the commencement contained 3.6 grains dissolved organic matter per gallon, only a faint appearance of green colour was observed after 42 days, and the organic matter in solution then amounted to 3.5 grains per gallon, or practically the same as at the commencement. Shortly after, the green growth developed rapidly and in the course of week amounted to nearly as much as in No. 1. After a further period of 30 days the dissolved organic matter had fallen to 2.2 grains per gallon.

The results of these experiments are very interesting, and show clearly that oxidation has little, if any, influence in removing the dissolved organic matter. In both experiments the water was well shaken three or four times every day for a period of six weeks, and in No. 2 there had been practically no diminution. In No. 1, after two weeks, a vigorous growth of microscopic vegetation had commenced, and after six weeks (when the growth appeared to have reached its maximum) the organic matter was found to have dropped from 4.9 to 1.7 grains per gallon. After a further period of 30 days (the green growth having been previously separated) during which the water was well shaken every day as before, a very slight additional growth occurred and the organic matter amounted to 1.1 grains per gallon. In No. 2 oxidation had no effect after six weeks, but the growth of vegetation brought down the organic matter from 3.5 to 2.2 grains per gallon in four weeks.

Having a number of samples of originally very foul sewage effluents which had been filtered and set aside for a very long period in stoppered bottles nearly filled, I thought it would be interesting to know whether any alteration in the quantity of dissolved organic matter present had taken place. In all the samples the dissolved organic matter originally present was known, and in two the amount contained after keeping a few weeks had also been ascertained. The dissolved organic matter in each was therefore determined, the results obtained being stated in the following table:—

TABLE No. III.

DISSOLVED ORGANIC MATTER IN SAMPLES OF SEWAGE EFFLUENT.

Grains per Gallon.

	1.	2.	3.	4.
At startline .....	13.6	24.0	9.6	15.7
After 6 weeks .....	..	..	..	3.8
" 7 " .....	..	..	3.0	..
" 18 months .....	5.6	4.5	2.7	3.1

These samples had all been kept in a badly lighted place, and the bottles being nearly full, and never opened except for the purposes of these experiments, it is evident that atmospheric oxidation had taken no part in the reduction of the organic matter. In each case, however, a considerable growth of fungus had developed; therefore, there can be little doubt that it was to this that the purification was due.

As it seemed probable that the organic matter represented by the fungus would account for the amount abstracted from the samples, I collected the insoluble matter from each sample and determined its amount. After deducting the ash the quantity in each sample was as follows:—

Grains per Gallon.

1.	2.	3.	4.
0.4	2.1	1.1	2.5

It is evident from these results that the lost organic matter cannot be accounted for in this way, but the experiments were necessarily very rough and far from conclusive, as it was impossible to remove the whole of the deposit from the bottom and sides of the bottles. Sewage fungus is also extremely unstable, decomposes rapidly on exposure to the air, and when disturbed has the property, apparently, of returning to the water much of the organic matter it has previously absorbed from it. In illustration of this latter property, I may mention an experiment I made some years ago. A quantity of fresh sewage fungus, after draining, was immersed in distilled water, shaken, and set aside over night. In the morning the whole was thrown on to a filter. The filtrate, which was of a greenish-yellow colour and had a most offensive odour exactly resembling rotten green vegetables, was acid, and slowly deposited a reddish-brown precipitate, which contained ferric oxide. After the filtrate had ceased to deposit the brown matter above referred to, the clear liquid was analysed with the following result:—

	Grains per Gallon.
Total solid residue dried at 220° F.....	149.0
Loss on ignition .....	65.0
Chlorine as chlorides .....	7.0
Free ammonia .....	22.34
Albuminoid ammonia .....	0.647
Organic carbon.....	13.58
" nitrogen.....	27.16

The albuminoid ammonia and the organic carbon and nitrogen results are all undoubtedly too low, as it was evident by the strong odour given off during the distillation and evaporation respectively that a considerable quantity of volatile organic matter was lost. I regret that, at the time this experiment was made, my method of determining the organic matter in solution was not devised, as it would have been extremely useful.

These results afford conclusive evidence that sewage fungus, when disturbed or on decomposition, gives up to the water in which it is immersed a large proportion of soluble organic matter and also much soluble inorganic matter; in fact, such water upon being subjected to the usual course of analysis gives every indication of sewage pollution.

With a view to ascertaining the effect produced upon the amount of soluble organic matter in crude sewage by the growth of sewage fungus, I took two portions of crude sewage, which contained 17.5 grains of dissolved organic matter per gallon; to one I added 7 grains and to the other 14 grains of sulphate of alumina per gallon. After vigorous shaking, the liquids were set aside for four hours, by which time the suspended matter had completely subsided, leaving a clear liquid above. The clear liquid was then siphoned off, and the dissolved organic matter determined in each. The result showed 15.6 grains per gallon in that to which 7 grains of sulphate of alumina had been added, and 13.5 grains per gallon in the other, where 14 grains of sulphate of alumina had been employed.



I may remark, in passing, that this experiment shows that hydrate of alumina precipitated from sulphate does not possess, to any appreciable extent, the property so often claimed for it of uniting with the dissolved organic matter in sewage and so rendering it insoluble; and further, as I shall subsequently show, does not prevent the growth of sewage fungus in the clarified liquid.

The clear sewage waters obtained as above described were then set aside in open vessels, covered to exclude dust. After three days, during which the water became increasingly turbid, a growth of fungus commenced on the surface, ultimately completely covering it. In the course of a few days it had extended from beneath, in long filaments and festoons, nearly to the bottom of the vessel. After three weeks the fungus became detached from the surface of the water, fell to the bottom, and gave slight signs of decomposition. The clear liquid was then siphoned off, care being taken not to disturb the fungus, and the dissolved organic matter determined in each. The result showed 6·8 grains per gallon in No. 1 and 4·6 grains per gallon in No. 2.

The fungus at this stage was collected and weighed. After deducting the ash, the amount found was equal to 2·5 grains of organic matter per gallon in each case.

The clear liquids were then set aside in open vessels for a further period of five weeks and frequently shaken every day. During this period a minute quantity of a colourless microscopic growth consisting of simple cells developed, but there was no further appearance of fungus, and at the end a green growth commenced similar in every respect to that already referred to. The dissolved organic matter was then determined and amounted to 5·1 grains per gallon in No. 1 and 3·9 grains per gallon in No. 2.

These results prove conclusively that the development of sewage fungus has a remarkable effect in removing dissolved organic matter from sewage water, as in the case of Experiment No. 1, 57 per cent., and in Experiment No. 2, 66 per cent., was removed by its agency in three weeks. They also confirm the results of experiments previously alluded to as regards the effect of atmospheric oxidation, as, assuming that the whole of the loss was due to that cause (which is extremely doubtful), in the case of Experiment No. 1 only 25 per cent., and in Experiment No. 2 only 15 per cent., of the dissolved organic matter was removed in five weeks.

The results of these analyses and experiments compel me to come to the following conclusions, viz. :—

1st. The removal of dissolved organic matter from river water by natural means is extremely slow.

2nd. The principal agent in the removal of dissolved organic matter from river water is the growth of vegetation.

And 3rd. That atmospheric oxidation has little or no effect in removing dissolved organic matter from river water.

#### DISCUSSION.

The CHAIRMAN said this paper was extremely interesting, and was evidently the result of a very large amount of careful and patient work. The question of the removal of organic matter was very complicated and perplexing. It seemed quite evident that a polluted water might contain a quantity of dissolved oxygen in contact with and abundantly sufficient to completely destroy all the organic matter for an almost indefinite period without any change whatever taking place. If that water were filtered or passed through or over any very large surface, such as sand, decomposition was set up, and the organic matter was destroyed. It was a matter rather for question whether even the action of this kind of fungus in the water was not possibly in some way one of surface.

Mr. D. HOWARD said he never could quite fall in with the view of those who maintained that no length of time in a river could remove any organic matter from it, because it was the case where did the water-cresses come from. This was putting the matter crudely, but the life history of water-cresses threw some light on that of organisms much

smaller than water-cresses and of greater importance. Those who knew the New River from Bedfordshire down to London would know that there were vast beds of water-cress; the best were not on the Lee, but on tributaries in which there were certain elements which were well kept out of the Lee by the water-cress. It was self-evident that these water-cresses came from somewhere, and if they were taken away there was so much less organic matter in the water; if they were not picked they decayed and went back again into solution like the sewage fungus. He remembered a very hot discussion in that room on one occasion, whether it was possible for oxygen and shaking to remove organic matter from water, and it was fairly proved that it was not, but the question which the development of modern study now naturally suggests as to the action of organic life was left out of consideration. Even the mere question of filters was complicated by possible organisms on the filters. He remembered reading a foreign investigation on the effects of filtration, which certainly appeared to show that there were organisms attaching to the grains of sand which had a good deal to do with the purification of water by filtration. There was one question that he should like to ask with regard to the extent of pollution. He was under the impression that a good deal of chalk water was pumped in below Ponder's End lock, between there and the filter beds at Chingford Mills, and he should like to know if that were so.

Dr. DARRÉ thought by this time everybody had agreed that direct oxidation had nothing to do with the purification of a river. It was entirely due to organisms; under which term he, of course, understood all living things, vegetable or animal, microscopic or otherwise, to be included. When he first stated that proposition eight or ten years ago he was laughed at, but he was certainly under the impression that it was now entirely accepted, for sterilised water had no power of oxidation, but if they allowed life to exist it oxidised considerably. The River Thames gave a splendid example of immense pollution and corresponding purification. During the Royal Commission of 1883, Mr. Latham calculated that there was between Barking and Greenhithe as much as 17 days' London sewage in the river. The river was at that time frequently analysed, and it was possible to calculate the actual amount of sewage in it; this was less than two days; in other words, nearly nine-tenths of the sewage discharged into it had been destroyed between Barking and Greenhithe. About that time he and others analysed the water hundreds of times, and to his great astonishment he found that the river was always purer in summer than in winter, although it looked fouler there was less organic matter, simply because there was more life in the warm summer than in winter. In winter there was much more fresh water in the river, and only the same amount of sewage, and the river ought therefore to be purer in winter than in summer, but the reverse was the case. Thus in January, March, and May 1892 the average composition of the river between Greenwich and Erith at high water in terms of albuminoid ammonia was 0·030 grain per gallon; at low water, 0·025; the mean being 0·027. In July, August, and September it had sunk to a mean of 0·021. The oxygen absorbed, taking the mean of 100 or 200 analyses, was 0·216 in the cold months, 0·18 in the warmer months, again showing that, measured by the oxygen absorbed, the river was purer in summer than in winter. There could not be any doubt now that the purification was entirely due to the action of organisms, but it depended on the kind and number of organisms. One might make hundreds of experiments, but unless one knew the number and nature of the organisms present they were valueless. Temperature also was of importance, as warmth favoured the growth of organisms. There was an immense amount of purification constantly going on in the River Thames, otherwise it would really be in the horrible condition in which fancy painted it sometimes; but, in fact, notwithstanding the enormous amount of sewage discharged into it, the river was practically pure 10 or so miles lower down.

Mr. W. C. YOUNG, in reply, said the chalk well-water about which Mr. Howard asked was not pumped into the river,

but direct into the local mains, and did not reach the reservoirs at all. With regard to Dr. Dupré's remarks, he must say that he did not think any investigation had been made on the lines on which he had made these. All other investigations had figures obtained by the albuminoid process, or by Frankland's organic carbon and nitrogen process; these figures did not represent definite amounts of organic matter. He thought he should be able to show that that organic matter contained varying proportions of carbon, and therefore by Frankland's method they would not get so reliable a result as working in the manner he had done. He quite agreed with Dr. Dupré that the condition of the river water was, as regards the quantity of dissolved organic matter, worse in the winter than in the summer, although it might appear to be better. The water supplied by the East London Waterworks Company contained a little over 1 grain of organic matter per gallon last month, whereas in the summer it dropped to below 0.2 of a grain. He did not think Dr. Dupré had given sufficient credit to vegetation, or rather had made no distinction between vegetation and the action of bacteria in removing the dissolved organic matter. The experiments recorded in the paper, especially those in which a bright green algae developed, showed clearly that vegetation was a very potent power in removing dissolved organic matter. Many experiments had been made which tended to show that crude sewage contained no organisms or microbes whatever; that there was something there which killed them. Dr. Percy Frankland showed, in a paper read some years ago, that by precipitating alumina in water containing an enormous number of microbes, he practically sterilised it. In these experiments that had been done, and the crude sewage matter after clarification, he presumed, would contain no bacteria. It contained certainly 9 to 15 grains of organic matter per gallon, and that was almost entirely removed by the growth of sewage fungi and the subsequent growth of vegetable matter.

The CHAIRMAN said that Mr. Young's remarks on Mr. Lovibond's paper made him think of the evidence there given, that the organic matter in water was of very varying character. If a way could be found of ascertaining, not only how much was present, but what it was, it would add very greatly to their knowledge. Whether they attempted to determine the total weight of organic matter, or of carbon in it, or of albuminoid ammonia obtained from it, the question of the variability of its nature remained.

#### NOTE ON AN IMPROVED SPECIFIC GRAVITY BOTTLE.

BY HENRY LOUIS, A.R.S.M., F.I.C., F.G.S., M.A.I.M.E., ETC.

DURING the course of an investigation which necessitated the determination of the specific gravities of a series of pulverulent substances of high density, I found that the ordinary type of bottle was in so far defective that it is impossible to determine its weight, when filled with water under ordinary circumstances, with absolute accuracy. It is obvious that any errors in this weighing will affect the determination of the specific gravities of very heavy substances proportionately more than in the case of comparatively light bodies.

I found that the irregular results are due to the very rapid evaporation that took place from the capillary tube within the atmosphere of the balance case when this was, as usual, kept dry by means of a vessel containing sulphuric acid or calcium chloride; even when the latter was withdrawn and a beaker of water substituted, the evaporation, although it diminished, by no means ceased.

It would seem that the excessively thin film of water, which forms the margin of the meniscus in the capillary tube through the stopper, evaporates very rapidly, and that this evaporation is supported by the capillary action of the

tube until the latter is entirely emptied. The action appears to be independent of small variations of temperature.

Thus in one experiment, the full bottle, the approximate weight of which was known, was weighed as rapidly as possible, and then again after successive intervals of time, the temperature being recorded at the same time.

The results are here given in tabular form:—

	Grains.	Temperature.
		° C.
1. First weighing .....	1328.000	16.9
2. After 5 minutes .....	1328.940	16.9
3. " 20 " .....	1328.899	17.0
4. " 30 " .....	1328.850	17.1
5. " 45 " .....	1328.795	17.4
6. " 60 " .....	1328.755	17.6

These results are also shown graphically in the annexed diagram, Fig. 1, from which it will be seen that the rate of loss, after the first five minutes, is practically uniform.

Fig. 1.

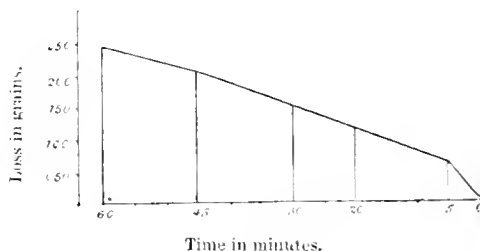
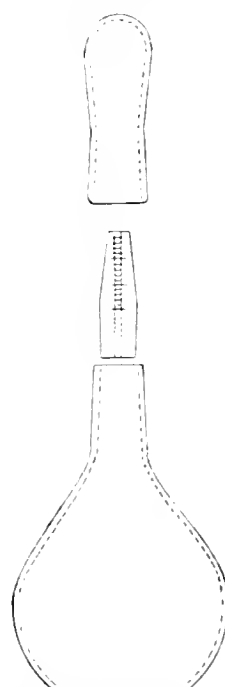


Fig. 2.



IMPROVED SPECIFIC GRAVITY BOTTLE.

In the course of an hour the loss amounted to 0.245 grains, the level of the meniscus in the capillary tube

having been meanwhile lowered to the extent of 0.65 in. It is quite evident that such considerable loss of weight cannot be neglected in accurate experiments, seeing how much time an accurate weighing takes; in fact the loss of weight is so rapid that it is scarcely possible to weigh the bottle exactly at all on a sensitive balance.

In order to do away with this source of error, I have had a specific gravity bottle made, which differs from the ordinary type only in that the stopper is longer than usual and is graduated, whilst a light glass cap is ground to fit accurately over the neck of the bottle, as shown in the accompanying figure (Fig. 2).

The bottle is filled in the usual way; the neck and stopper are then rapidly wiped dry, the cap put on, and the body of the bottle then dried. When weighing the bottle, any variation in the height of the column of fluid in the capillary tube must be read off on the graduations, which may be to any convenient scale; mine are  $\frac{1}{10}$  in. apart, corresponding to 0.01 grains of water. It is an easy matter to calibrate this tube and thus to apply the necessary corrections. The capped bottle can be left for any length of time without any loss of weight taking place. I have noticed, however, that, after standing for 21 hours, considerable evaporation had taken place from the capillary tube, the water being condensed in comparatively large drops on the inner surface of the glass cap. This does not matter, provided that the temperature of the water and the level of the meniscus are read at once as soon as the bottle is ready for weighing; the weighing itself can be performed at any time thereafter.

and each succeeding one less of the nitric acid, until from the overflow of the last one the bisulphate of soda flows away without any nitric acid.

I may point out that while the ordinary process of making nitric acid is one of fractional distillation by time, mine is fractional distillation by space.

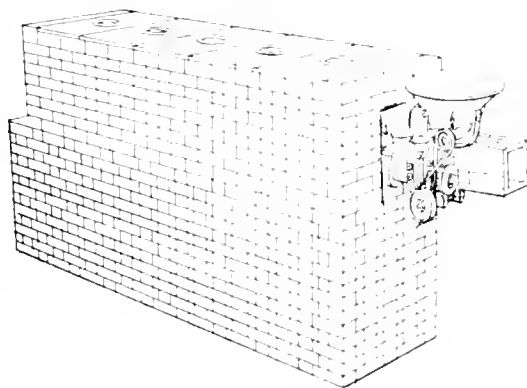
Instead of the operation being always at the same point of space, but differing by the successive points of time, I arrange for the differences to take place at different points of space, and these differences exist during one and the same points of time.

Experience has shown that there may be considerable variation made both in the proportion of sulphuric acid used and also in its strength, enabling one to obtain the nitric acid from the different still-heads of varying strengths and varying proportions of acid of these varying densities. This is useful to those makers who require different acids for differing trade purposes.

It is also quite possible to produce the full product of nitric acid of the full gravity of 1.500.

With regard to the saving effected by my system, it has been found to exceed my expectations.

I now save more than two-thirds in fuel, also save four-fifths of the condensing plant.



## NOTE ON AN APPARATUS FOR THE CONTINUOUS PRODUCTION OF NITRIC ACID.

BY MAXWELL PRENTICE.

By the kind invitation of the Committee I have the honour to exhibit a model of the apparatus designed to produce nitric acid in a continuous manner.

As long ago as 1878 I carried to a very satisfactory issue experiments in the continuous distillation or rectification of sulphuric acid. The system of platinum pans which I invented was soon adopted by the largest makers of oil of vitriol, both in Europe and America, and the Prentice pan holds its own, in my opinion, to this day.

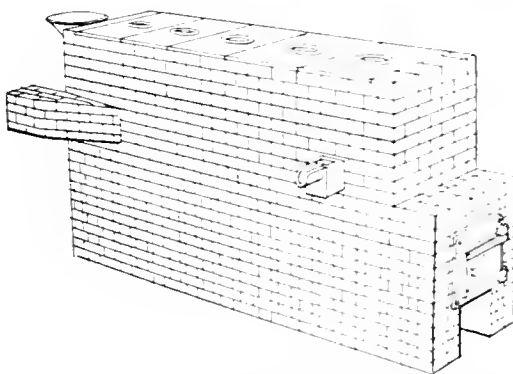
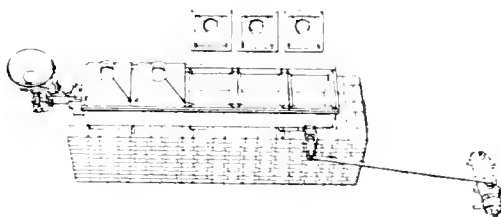
The great saving in fuel and general convenience of a continuous process is now so well recognised that you will not be surprised to learn that soon after patenting my platinum pans for the concentration of sulphuric acid, I began experiments with the object of discovering some method by which nitric acid could be produced without the loss caused by the alternate heating and cooling which occur in the ordinary process.

The difficulties seemed, however, insurmountable, and it was not until 1892 that I again took up this question, and discovered important points which enabled me at last to solve the problem.

The model before you is a very exact copy of the apparatus which has been working satisfactorily at Stowmarket for some time.

The nitrate of soda is placed in weighed quantities into the hopper, whence it passes (in the same manner as grain runs in a flour mill) to the feeder. This feeder is a miniature horizontal pug-mill, which receives the streams of acid and of nitrate, and after thoroughly mixing them delivers them into the still, where, under the influence of heat, they rapidly become a homogeneous liquid, from which nitric acid continuously distils.

The still is divided into compartments or chambers in such a manner that the fluid may pass continuously from one to another. The nitric acid being continuously separated by distillation, the contents of each division vary—the first containing the full proportion of nitric acid



One of my stills, capable of producing about four tons of nitric acid per week, weighs less than two tons; whereas (to produce the same quantity) two stills weighing five tons each were formerly used.

This saving of four-fifths of the weight of iron is accompanied by a corresponding saving in the cost of masonry.

There is also reason to believe that the life of my new still will be 10 times greater than that of any previous still, for the destruction of the stills has been due to the alternate heating and cooling, with the consequent condensation of acid vapours within the still. This is, of course, entirely obviated by my continuous process.

I hope that my work in improving the process of production of nitric acid may be as useful to the makers of nitric acid as my improvements in the production of oil of vitriol have been to the makers of sulphuric acid.

#### DISCUSSION.

MR. T. TYLER asked what was the relative life of the body of the still and the cast-iron top, and what was the minimum strength of acid Mr. Prentice found possible to use with the nitrate in order to secure sufficient fluidity for the outflow sulphate of soda with the smallest possible excess of acid, not bisulphate. Those questions were of great importance to those who were not superphosphate makers. There was one great advantage about the 5-ton still, that it lasted a long time. He had known a cast-iron nitric acid pot to last 20 years, and it would be interesting to know if the life of this still could be measured by any comparison with that. Mr. Prentice had committed himself to the statement that this still was 10 times as enduring as the 5-ton still which he formerly employed, so that if the 5-ton still would last 20 years this would last 200.

MR. MANNING PRENTICE said, so far the still in operation did not appear to have deteriorated at all, either the still itself or the top, so that experience was still lacking as to the relative life of one part of the still or another. With regard to the statement that this still would last 10 times as long as the other, he thought it was true, but he did not quite see how he was to demonstrate it. It was absolutely impossible to say how long one of these stills would last, but he said, advisedly, the place where former stills wore out was where the nitric acid condensed upon the cooler surface and trickled down. In his still there was no alternate heating and cooling, and therefore no condensed nitric acid trickling down. If the wear of the still were caused entirely by trickling nitric acid, and there were none, the still must last for ever. With regard to the percentage of acid used it was very uncertain what the percentage ordinarily used was, for, as far as he could gather, it differed much with different makers. With his still they generally used about 12 parts by weight of acid to 10 of nitrate of soda, but the question of the weight of acid would depend on a variety of considerations. In some works there was considerable advantage in having a large excess of acid. In some places a certain amount of weak nitric acid might be used mixed with the oil of vitriol.

MR. T. TYLER observed that a good deal depended on the strength of the acid used, whether it was chamber, brown, or concentrated acid. He had seen this apparatus working and was pleased with it, and therefore he hoped Mr. Prentice would give as much information as possible. If Mr. Prentice could say that 10 parts of practically dry nitrate of soda mixed with 12 parts of sulphuric acid of a definite strength, saying what that strength was, produced a fluid outflow of low acidity, and what the acidity of that outflow was, something definite for guidance would be gained.

MR. MANNING PRENTICE said by 12 parts of sulphuric acid he meant 12 parts of oil of vitriol. With regard to the acidity of the resulting eake he had not the figures before him. The fact was he had not written a paper on this subject, but this model had come to London for the purpose of being sent abroad, and it occurred to him that a sight of it might interest some members of the Society, and he had therefore brought it forward, but he was not prepared to give the exact figures as to nitric acid making. They were making, however, good nitric acid of full 1500° sp. gr., which was running in a continuous stream of a pale straw colour. For makers who wanted nitric acid of a lower gravity there

was no necessity to use oil of vitriol, a weaker acid could be used. There was no doubt that on a large scale nitric acid could be made very cheaply. He could imagine a constant stream of acid running from a Glover tower through this still, a constant stream of nitric acid running out from condensers, bi-sulphate flowing continuously from the exit, and superphosphate being wheeled away, so that the whole matter became wonderfully simple.

*Meeting held Monday, April 16th, 1894.*

MR. WM. THORP IN THE CHAIR.

#### MODIFICATION OF FERRIC CHLORIDE DISTILLATION PROCESS FOR ESTIMATION OF ARSENIC IN COPPER.

BY F. PLATTEN.

THE accurate estimation of arsenic in copper has become of late years a matter of great importance to manufacturers, on account of the influence exerted by this impurity on some of the mechanical and other properties of the metal.

With the great advance in electrical engineering has sprung up a demand for copper, with the maximum conducting power 100 per cent. is now stipulated for in the Post Office specification relating to wire, and as the presence of mere traces of arsenic is found to lower the conductivity to a serious extent (see *Electrolytic Separation of Metals*, G. Gore, p. 27), it is an absolute necessity to be able to detect and estimate extremely small quantities with certainty.

The question of how much arsenic may be present in copper without impairing its suitability for rolling purposes, does not appear to be yet settled, but, on the authority of an expert smelter, it may be stated that copper sheets have rolled well when containing arsenic to such an extent that the rolls were whitened by the fumes; unfortunately, however, no analysis is available.

In the manufacture of copper plates for locomotive fire-boxes, it has been the custom for some years for engineers to limit the arsenic to a maximum of 0.15 per cent., but this regulation was apparently ill-advised, for recent experience has shown that even up to 0.50 per cent. of arsenic has not proved in any way detrimental to the working properties of copper for this purpose; in fact, the reverse is now supposed to be the case, and the limit is accordingly being relaxed, but while it was in force the variation in the analyses of different chemists gave rise to endless trouble.

Various methods have been proposed for estimating small quantities of arsenic in copper, and of these the one in use at the Tharsis Works at Oldbury seems to be most generally adopted.

It consists of dissolving the metal in nitric acid, adding ammonia in excess, and saturating with sulphuretted hydrogen.

The ammonium sulphide formed, precipitates the copper as sulphide, while the arsenic is retained in solution.

The cupric sulphide is then filtered off, washed with dilute sulphide of ammonium, and to the filtrate containing the arsenic is added hydrochloric acid in excess; this decomposes the ammonium sulphide, and arsenic trisulphide, accompanied with much free sulphur, is thrown down.

The precipitate, after being filtered off, is oxidised with fuming nitric acid and the arsenic acid precipitated with magnesia mixture and weighed on a tared filter after drying at 95–100° C.

There is undoubtedly a tendency for cupric sulphide to retain arsenic even when precipitated in an ammoniacal solution, but with ferrous sulphide the case is different, and the following method takes advantage of this point, and is

said to be very accurate (see Crookes' Select Methods in Chemical Analysis, p. 439), but it requires special precautions and great care.

The metal is dissolved in nitric acid and a small quantity of ferric nitrate added, the solution is then nearly neutralised with sodic hydrate and excess of sodium acetate added.

The solution is then heated to boiling and filtered as rapidly as possible, the precipitated ferric acetate dissolved in hydrochloric acid, ammonia added in excess, and sulphuretted hydrogen passed to saturation, and from this point the process is identical with the one already described.

The great danger is the precipitation of a basic acetate of copper with the iron, when there is nothing for it but to throw out the solution and commence again.

It is said to be possible, after precipitating arsenic trisulphide and cupric sulphide in an acid solution, to render the mixture free from arsenic by digestion with yellow sodium sulphide, but even if it were so the method would be extremely laborious, as it would be indispensable to treat the precipitate four or five times.

A method suggested by Fresenius (see Quant. Analysis, p. 469) is to pass dry chlorine gas over the finely-divided metal, volatilising the chloride of arsenic; but it is not practised, principally, no doubt, on account of the objections to the use of chlorine in the laboratory.

Generally speaking, the amount of arsenic present in the commercial copper is small, and it is on this account that a fair quantity of the sample—not less than 10 grms.—must be taken for analysis, and the manipulation of this quantity in processes which involve precipitation and washing is a serious matter. A method which is fast superseding all others is known as the ferric chloride distillation process, which depends on the volatility of arsenious chloride.

A very convenient way of conducting it, is to take 20 grms. of ignited ferric oxide, dissolve in 150 cc. pure hydrochloric acid, and transfer to a Wurtz flask connected with a condenser, and distil over about 50 cc. of hydrochloric acid.

If this distillate gives no trace of arsenic on treatment with sulphuretted hydrogen, it may be taken that the reagents are free from arsenic, and the copper to be analysed may be introduced into the flask.

10 grms., preferably in the form of drillings, are taken and the distillation carried on till about 150 cc. of the liquid has distilled over; this will as a rule contain the whole of the arsenic, but it is best to change the receiver, add more hydrochloric acid, and test a further distillate with sulphuretted hydrogen.

The arsenic is then precipitated with sulphuretted hydrogen, the arsenic sulphide filtered off, washed, dried, and then treated with carbon disulphide, and finally weighed on a tared filter after drying at 100°.

This method was introduced by Dr. John Clark in a paper on the estimation of arsenic in pyrites (see Journal of the Society of Chemical Industry, May 1887), and the results then published were of a highly satisfactory character.

He recommends, in the case of large quantities of arsenic, direct titration of the distillate with iodine solution, but when inconsiderable, the precipitation with sulphuretted hydrogen as stated above.

The percentage of arsenic in commercial copper is rarely, if ever, high enough to admit of directly titrating the distillate, so that up to the present the usual course has been to weigh the arsenic sulphide.

This necessitates the use of tared filters, which is generally admitted to be unsatisfactory; certainly a margin of a milligram is the minimum that must be allowed, and this is frequently insufficient, and, when the prolonged washing of the precipitate and subsequent treatment with carbon bisulphide is taken into consideration, it will be seen that discrepancies are almost certain to occur.

In the present communication an account will be given of a modification touching on this point, whereby it is believed that a considerable saving in time must result to the many chemists who are using this deservedly popular method.

Now arsenious sulphide is commonly supposed to be unacted on by water, and it may be interesting to quote Fresenius on the subject, as follows:—

" . . . . . When boiled with water or left for several days in contact with that fluid, it ( $As_2S_3$ ) undergoes a very trifling decomposition—a trace of arsenious acid dissolves in the water and a minute proportion of sulphuretted hydrogen is disengaged; this, however, does not in the least interfere with the washing of the precipitate" (obtained in the course of a quantitative analysis).

It is presumed this refers to cold water, although the point is not quite clear—certainly it is unsafe to wash in hot water—for appreciable quantities of arsenious sulphide are frequently found to appear in the filtrate on standing, and it was the observation of this fact which suggested a careful inquiry into the extent of the action of water on the sulphide.

It is undoubtedly greater than is generally supposed, and instead of weighing the small quantities of arsenious sulphide obtained by the distillation process, it is found to be far preferable to boil the sulphide in water, and, when dissolved, titrate the solution of arsenious acid with centinormal iodine, and excellent results have been obtained in a great number of analyses. After transferring the sulphide to an ordinary flask, no further attention is required; it may be placed on an iron plate and left to boil, and any number can be dealt with at the same time.

It is usually found that solution is effected by boiling in 400 to 500 cc. of water in one to two hours, but it is best to boil, for a few minutes after the last trace of colour has gone, to thoroughly expel sulphuretted hydrogen.

It is interesting to note that commercial sulphide of arsenic is said to be always contaminated with arsenious acid (see Taylor on Poisons in Relation to Medical Jurisprudence and Medicine, 1875 edition, pp. 354—356), but the test by which this conclusion is arrived at is evidently misleading, which reads: " . . . . . the powdered sulphides yield a solution of arsenious acid on boiling with water acidulated with hydrochloric acid, and this sometimes amounts to as much as 30 per cent. . . . . " It will be seen at once that if only a small quantity of arsenious sulphide were taken in such an analysis, a much higher result might be easily obtained, inasmuch as the whole of the sulphide might be converted by boiling into the soluble arsenious acid. Apparently the only reference published is in a method devised by MM. Clermont and Frommel for the separation of arsenic, antimony, and tin when present as sulphides, which will be found in Comptes Rendus, No. 13, April 1878, and from which the following is extracted.

The mixture of sulphides is to be washed with the greatest care, as the minutest trace of hydrochloric acid would occasion a loss through the volatilisation of arsenious chloride; the mixture is then washed into a flask and boiled with pure water, a current of air materially aiding the solution.

Unless the quantity of arsenic exceeds one-fifth of a gm., the dissociation of the sulphide is complete after distilling 500 to 600 cc. water, and on filtering the whole of the arsenic is found in the filtrate as arsenious acid.

Before relying on this reaction and adopting it permanently, the following experiments were made in the metallurgical laboratory of the Mason College under the supervision of Mr. T. Turner.

Two separate analyses of a sample of copper sheet were made by the distillation process, and the arsenious sulphide carefully washed, treated with carbon bisulphide, and weighed, the results being—

	Per Cent.
I. . . . .	0.707
II. . . . .	0.719

average 0.713, a variation of 0.012 gm., or 1.6 per cent

To ascertain if the whole of the arsenic is expelled in this process, a solution of arsenious acid was carefully made up, and varying quantities added to the distilling flask, with the usual quantity of the purest electro-deposited copper,

from which it was impossible to get a weighable quantity of arsenious sulphide. The results were as follow:—

	Arsenic introduced.	Found.	Excess.
	Mgms.	Mgms.	Mgms.
1	9.048	9.75	0.7
2	22.62	25.42	2.8
3	46.79	47.98	1.19

which results are fairly satisfactory considering the small amounts dealt with and the liability to error in using tared filters. It may be noted that there is an excess in each case, which may be accounted for in a measure by the fact that the electro deposited copper contained arsenic, but not in weighable quantity. To test if there is any loss on boiling the sulphide the following experiments were carried out. A carefully prepared solution of arsenious acid in hydrochloric acid was made and various quantities treated with sulphuretted hydrogen, filtered, and the precipitate washed into a 30-ounce flask, filled up with pure water, boiled, and, when completely dissolved and colourless, titrated with centinormal iodine standardised with the arsenious acid solution. The following are the results:—

Arsenic introduced.	Found.	
Mgms.	Mgms.	Mgms.
3.74	3.78	0.04 excess.
7.48	7.36	0.12 loss.
18.81	18.57	0.23 "

which will no doubt be considered conclusive. Finally, the arsenical copper, which gave 0.713 per cent. arsenic by distillation and weighing the sulphide, was again determined by solution and titration of the sulphide, the figures in two separate analyses being—

$$\left. \begin{array}{l} 0.683 \\ 0.675 \end{array} \right\} \text{average } 0.679 = 0.034 \text{ per cent. less.}$$

It is seen that the result by iodine method in this case is somewhat lower, but this hardly detracts from the value of the experiment, for it is invariably found with impure coppers, with which this sample must be classed, that there is a dark insoluble residue left on boiling the sulphuretted hydrogen precipitate.

These residues have been tested in several cases and have been found to contain antimony, so that when the arsenious sulphide is weighed this antimony precipitate would be included and appreciate the result.

In conclusion, it is not considered that sufficient data have been given to claim for this modification any superiority except in point of saving time. It will readily be seen, however, that if the weighing of filters (in itself a most laborious task), careful washing with carbon bisulphide, and finally weighing several times, can be dispensed with in favour of boiling the precipitate and titrating, hours of labour may be saved, and the saving will be in direct proportion to the number of estimations on hand.

It should be mentioned that the freshly precipitated sulphide is more easily dissolved than if left to stand, and that it is quite unnecessary to wash the precipitate free from hydrochloric acid.

MM. Clermont and Frommel state that the minutest trace of this acid will cause a loss, but this is certainly not the case. In addition to the foregoing experiments, in which no washing was done, a special test was made to determine this point by boiling a precipitate transferred from the filter-paper without washing at all for several hours in a Wurtz flask connected with a condenser. The distillate gave no trace of arsenious, and it may be safely assumed that even were arsenious chloride formed it would be instantly decomposed by the excess of water present.

## DISCUSSION.

The CHAIRMAN said Mr. Platten seemed to have hit on one of those apparently simple things which made a great difference in the amount of labour involved. The chemistry of his modification would be patent to all.

Mr. SUTHERLAND said he had to use the process of distillation by means of ferric chloride very frequently in estimating the arsenic in commercial copper, and he had found the process extremely reliable. He had not found any great delay involved in the case of the chloride being titrated direct with iodine; he always found the results very satisfactory. The sulphide could be treated with nitric acid and titrated with uranium acetate, the results being likewise accurate. A very suitable method for estimating arsenic gravimetrically had been suggested to him some years ago by Mr. Blount, viz., dissolving the sulphide in ammonia, evaporating down, and then weighing. He had himself used this method frequently, and found the results came out very accurately. He should like to ask Mr. Platten if he had compared the results of these processes with those he had obtained in the one he had now described.

Mr. PLATTEN said the only objection he could see to the ammonia process was that ammonia dissolved appreciable traces of sulphur; and very frequently sulphide of arsenic, from some cause or other (as a rule impurities in the ferric chloride or perhaps in the acid), when precipitated, was contaminated with sulphur, which would be dissolved in the ammonia, and so increase the result.

## THE TREATMENT OF GOLD ORE AT THE WITWATERSRANDT (TRANSVAAL) GOLD-FIELDS.

BY HENRY DE MOSENTHAL, F.I.C., F.C.S.

I HAVE lately returned from the Transvaal, where I spent some months. Whilst there I visited a number of mines, and, thanks to some of the leading mining engineers and chemists, who gave me, in the kindest manner possible, the information I sought for, I was able to investigate the methods employed for the winning and recovery of gold in the Witwatersrandt district.

As I have been unable to find any recorded general account of the methods employed, and the difficulties to be overcome, I have ventured to think that a description of my experiences and observations may not be without interest to the members of the Society.

I wish to record here my thanks to Mr. Hennen Jennings, one of the leading mining experts, who showed me some of the plant recently erected under his superintendence; to Mr. William Bertel, the senior chemist at the well-known Robinson mine, who gave me the benefit of his experience, and devoted considerable time to preparing notes for my guidance and samples for my collection; and to Mr. Charles Butters, who kindly allowed me to visit the works of the Central Ore Reduction Company.

I do not presume to discuss the merits of the various methods employed; my object is rather to give an account of what I saw, and record the information I have been able to gather.

Johannesburg, the great centre of the gold-mining industry, is a town of some 30,000 white inhabitants, and on seeing it no one would believe that it is but six years old. There are mines on the very outskirts of the town, and on both sides they extend, mostly in single file, along the main reef. Their appearance is very similar. A head-gear and hauling machinery, situate sometimes at a distance from, sometimes in close proximity to the mill; cyanide works, erected, or in course of erection, near the mill; dams or reservoirs in which the rain-water, and the

water pumped out of the mine, is collected; and settling-dams for the tailings and slimes, are what you see everywhere.

The general impression is certainly most striking. About 80 mines (including the deep levels), one next to the other, the majority having extensive machinery and plant, most of which has been brought there by ox-waggon before the railway line was made. A gigantic industry sprang up in a wilderness nearly 1,000 miles from the principal seaport. The difficulties that had to be overcome were numberless.

The railway has now created greater facilities, but still labour is very dear, and the price of all commodities remains excessive. According to recent statistics published by the Johannesburg Chamber of Mines, 3,200 white and 21,000 coloured workmen were employed at the mines at wages averaging 16s. to 18s. a day for the former, and 2s. to 3s. a day for the latter.

These remarkable goldfields are situated on a high plateau at one of the highest points of South Africa, which entire country rises terrace-like from the sea. The gold-bearing district has the shape of a shallow basin bordered to the north by a sandstone ridge, which forms the watershed between the Orange River, which flows into the Atlantic and the Limpopo River, which flows into the Indian Ocean; and to the south by hills of Trap Rock. A large number of reef-groups have been found in that area, the chief group being the so-called main reef series, which includes the north reef, main reef, main reef leader, middle reef, and the south reef; which, together, cover an extent of nearly 40 miles from east to west; the other groups of reefs being found chiefly to the south, and parallel to it.

Johannesburg is situated close to the main reef, 4,772 feet above the level of the sea, whilst other points on the main reef attain an elevation of 5,500 feet. The whole area occupied by the mines of the Witwatersrandt district is about the size of London, including the suburban districts.

Within this area coal has been discovered, and in a country like the Transvaal, where wood is scarce, this discovery of coal has alone made it possible to develop the gold industry in so marvellous a manner.

The coal has about 60 per cent. of the caloric value of English coal, and was selling in December last at 22s. per ton, delivered at the gold mines. The price would be lower were it not that the railway company will not carry coal in bulk. A sack, which contains 2 cwt., lasts only three journeys, and costs 9d. at Johannesburg; so that the compulsory use of sacks increases the cost of coal by 2s. 6d. per ton.

This local coal, in consequence of the large percentage of ash, makes very inferior coke, so that it cannot be used for assaying or refining. For these purposes English coke must be used, which was selling at 14l. per ton.

As to the occurrence of gold, some believe that this flat basin was once a lake, and that the gold was deposited there. Of course this is in dispute, and numberless other theories have been started.

I may mention here that a large section of the Boers—and I understand that Mr. Christian Joubert, the Minister of Mines, is of the number—firmly believe that there was no gold in the ground as long as the country was under British rule, but that after the successful war of independence God placed the gold into the earth to reward His faithful people.

The formation is so different from that in which gold is known to occur in other parts of the world that geologists and mining experts would not believe at first that the gold would hold out to any depth. It does not occur in quartz, but in a conglomerate or pudding stone, which is locally termed "banket," this being the Dutch name for the sweet-meat we call "almond rock." The banket consists of pebbles held together by a silicious cement, which contains crystals of iron pyrites. Above the permanent water-line the pyrites in the banket cement has become oxidised through the action of air and water.

Thus the rock is more oxidised at the surface and less so as you descend; the greater the depth the more pyritic the rock.

The oxidised rock, which in mining language is termed "free milling" ore, as against pyritic or "refractory" ore, is very friable.

The gold, which is so fine as to be but rarely visible even with considerable magnification, occurs almost exclusively in the cement which holds the pebbles together; sometimes it has been found in the pebbles themselves, but then only in cracks in the quartz.

In the pyritic ore the gold is finely distributed among the iron pyrites.

I possess a number of thin sections prepared for the microscope, both of the oxidised and the pyritic rock, two of which are represented in the annexed microphotographs. The quartz pebbles polarise in the well-known manner, whilst the cement which holds them together, being amorphous, does not. In the pyritic ore the crystals of iron pyrites can be seen embedded in the cement. The cement consists chiefly of finely-divided particles of amorphous silica. An analysis of the oxidised cement showed it to consist of:—

Silica .....	87.4
Oxide of iron .....	10.8
Water .....	1.8
Lime .....	Traces.
Magnesia .....	"
Alumina .....	"
	100.0

I have some particles of gold extracted from rich pyritic ore by means of nitric acid, and I have examined this gold microscopically. I find that it consists of very fine laminae or specks. When viewed with transmitted light some of these laminae show the well-known green colour of translucent gold leaf, only darker, due possibly to the presence of silver, which may also account for some appearing bluish; some, again, are of a bright red, which has been observed to be frequently the colour of finely-divided gold; the tints vary considerably. Viewed with reflected light they nearly all appear yellow, orange, or red, and have a metallic lustre. Some of the specks show crystalline structure, but in most I have been unable to detect any trace of crystallisation.

Some of the gold is associated with sulphides of the base metals, and these auriferous sulphides are spoken of as sulphurets.

The fine distribution of the gold and the presence of sulphurets are generally held to account for the difficulties which have been encountered in the extraction, and I shall have to refer to this as we review the various processes.

The reef, which consists of banket, as I have said, is bordered by a kind of sandstone, the hanging wall and foot-wall being the same all through the main reef. Some of the other reefs have a more shaly foot-wall, and in some mines the foot-wall and hanging wall, are quartzose sandstone.

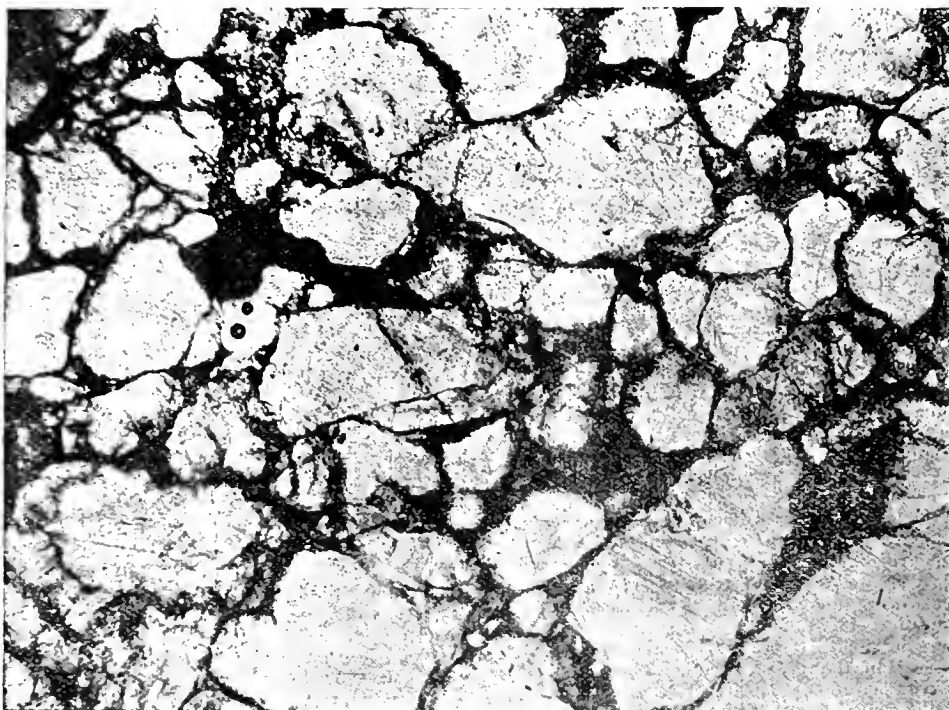
None of the mines at present worked are deep, most of the workings being about 300 or 400 feet below the surface. In the so-called deep level mines recently tested—with the exception of the Randt Victoria Deep Level, which has a borehole 2,400 feet deep—the reef has been struck at less than 1,500 feet.

The reef in some of the mines which have been worked hitherto descending at an angle of about 45°, it was thought that deep borings further on would strike the reef again. This assumption proving correct a large number of deep-level workings are now being opened up. The reports of competent mining experts go to show that in these deep levels the reef continues of the same nature as above, and bears about the same average quantity of gold. Shafts have been sunk in several of these deep-level mines and large batteries are to be erected to treat the ore.

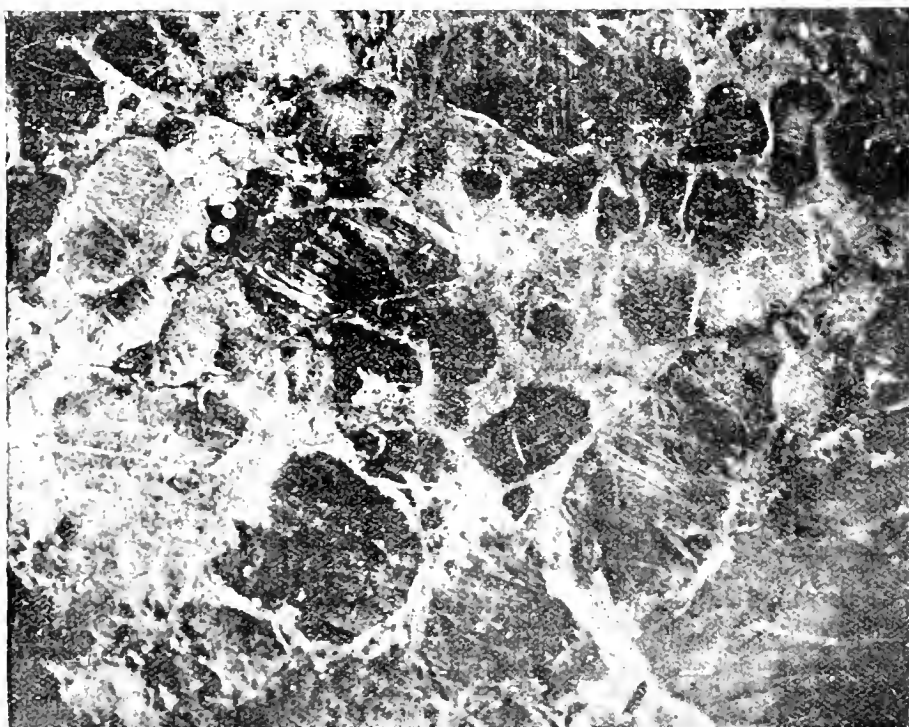
I shall not enter into a description of the underground working, my object being to describe what is done with the ore after it has been obtained and drawn up out of the mine by strong hauling-gear to the top of the head gear.

If the stonebreaker is in the mill the ore is at once taken there, but in most cases the stone-breaker is placed in the head-gear, and this is naturally more economical in a flat country.

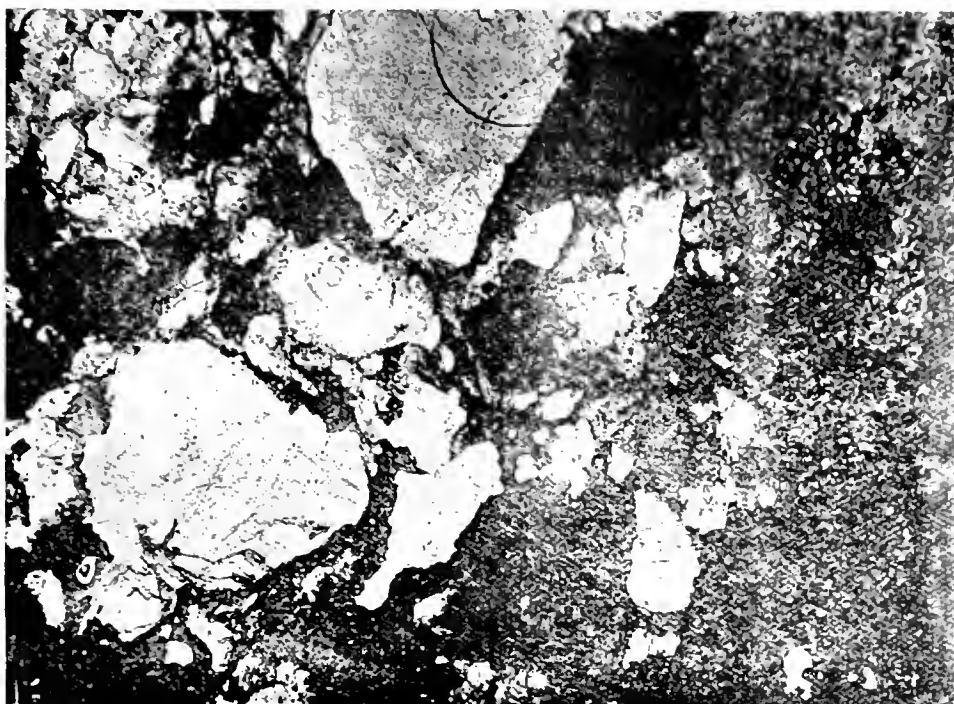




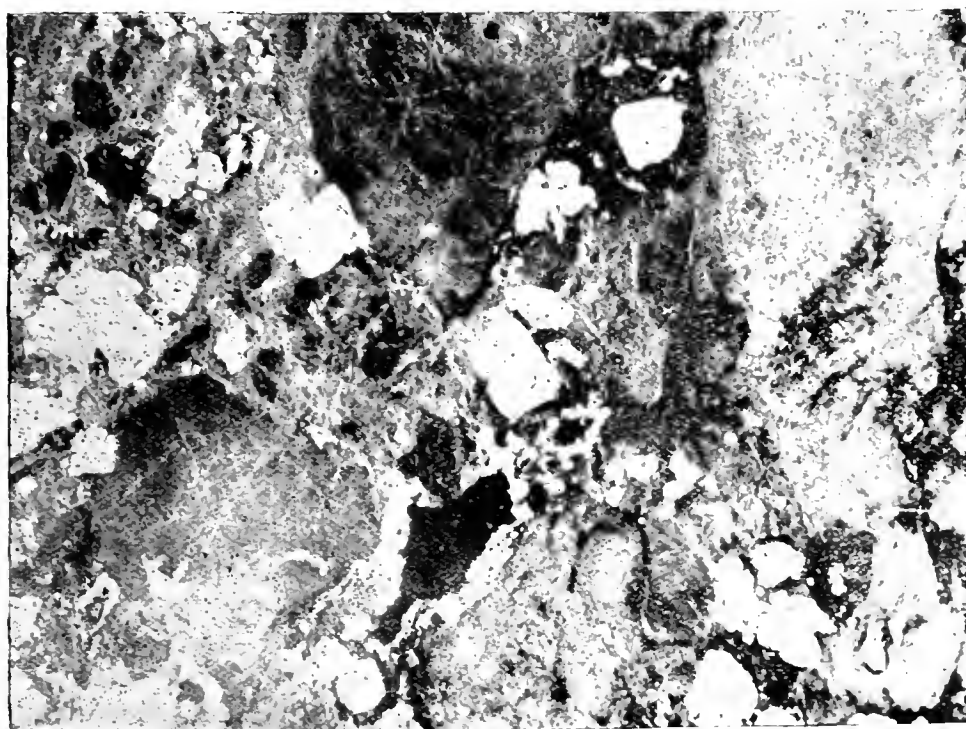
Section of oxidised basket — 50 photographed with transmitted light.



The same section of oxidised basket — 50 photographed with reflected light.

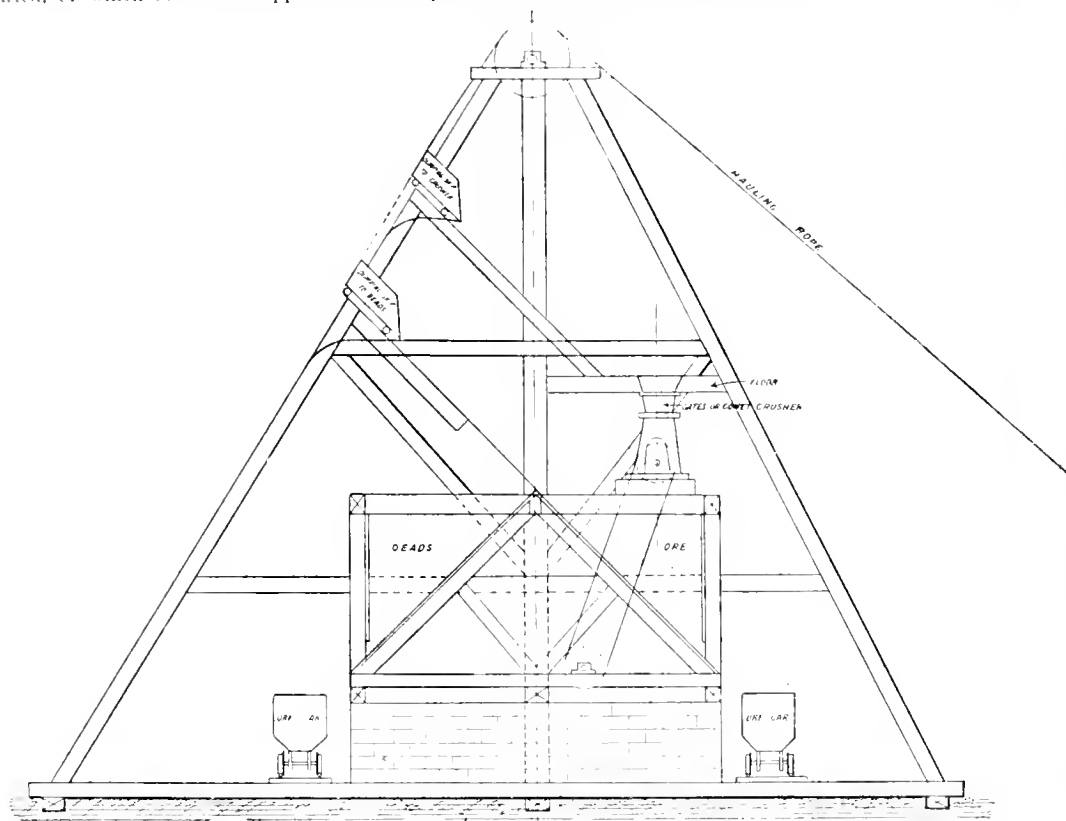


Section of pyritic banket - 50 photographed with transmittant light.

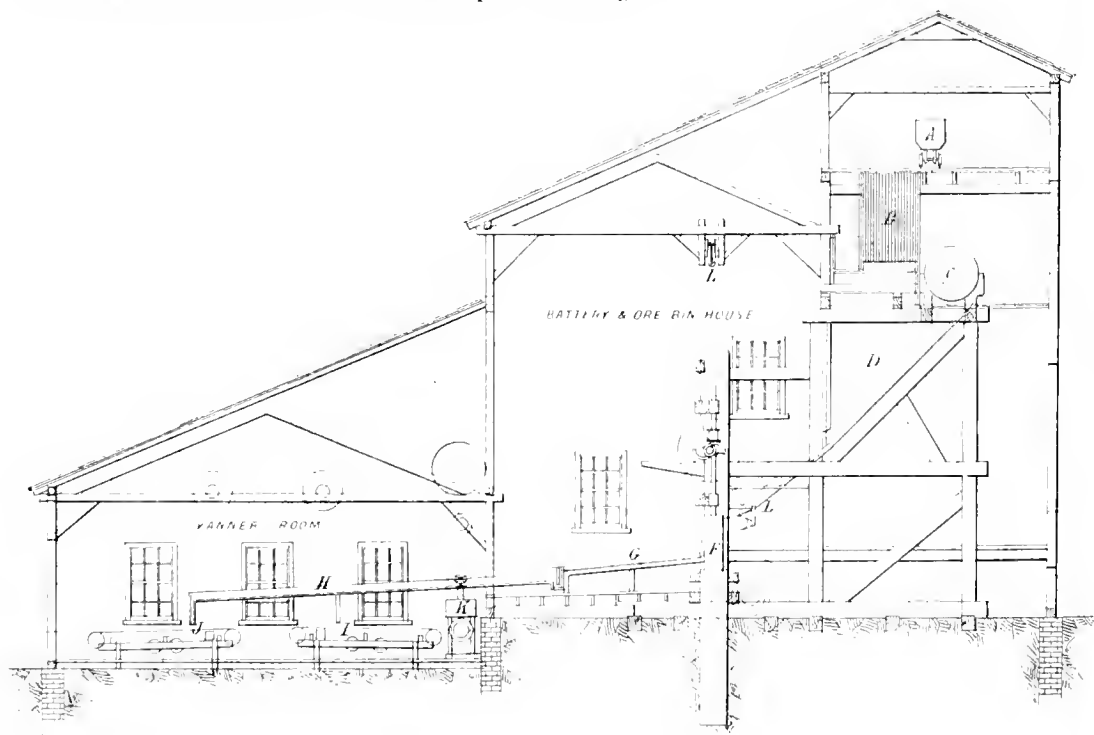


The same section of pyritic banket - 50 photographed with reflected light.

In most of the mines which have been more recently started, or which have been supplied with new plant, the head-gear contains a Gates or comet crusher, and a sorting floor as here shown.



When the ore reaches the top of the head-gear the skip in which it is contained is tilted—in the more recent plants this is done automatically—and the ore falls on to the sorting floor.



On this sorting floor the ore is sprinkled with water, which renders it easier to distinguish the country rock from the auriferous banket, and the former is as far as possible thrown out by hand, the latter being thrown into the crusher.

The Gates or comet crusher, now greatly in favour on the Randt, consists of a spindle which oscillates eccentrically.

A description and drawing of this crusher will be found in Eissler's "Metallurgy of Gold."

Most of the machinery in the mills and head-gear at the Transvaal mines have been supplied by the Sandycroft Foundry Company, and by the well-known firm of Fraser and Chalmers.

The diagram at the bottom of previous page shows a plan of a mill with Grizzly B, and the rock-breaker C within the building. It is to this drawing that the capital letters in the text refer.

In the mines which have not got the new plant above described the well-known Blake stone-breaker is used together with the Grizzly, which consists of iron bars placed 2 inches apart, so that the small pieces fall straight into the ore bin and not into the crusher. This Grizzly and Blake crusher are more frequently to be found in the head-gears than in the mill itself, as above mentioned.

From the ore bins in the head-gear the crushed ore is brought to the ore bins in the mill, and passes into the automatic feeders E, and thence gradually into the mortar F. The mortar is that portion of the mill which contains the stamps. Five stamps act in one mortar.

A mortar of the more recent kind with internal amalgamation plates is here shown:—

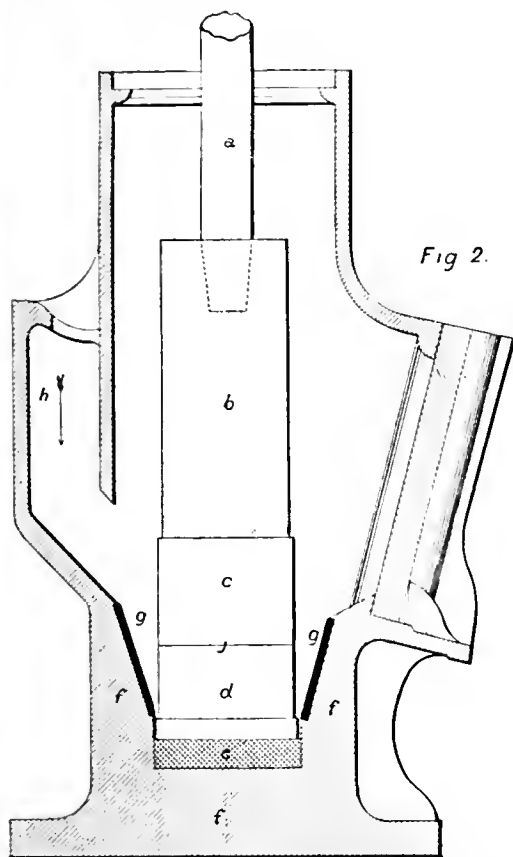


Fig. 2.

a Stem (wrought iron). b Head (cast steel). c Shoe (forced steel).  
d Die (forged steel). e Steel liner. f Mortar (cast iron).  
g Copper plate for internal amalgamation. h Ore feed. i Screen.  
Drop of stamp at j = 9 inches.

Some mortars have the inside amalgamation plates placed somewhat higher up than shown in the drawing, because they are then less liable to be damaged by the strain of the falling stamp.

The ore is kept wet by a constant supply of water. The copper plates *g g* are covered with mercury in the same way as the external plates or aprons G, of which we shall have to speak presently. Mercury is dropped into the mortar. The quantity of mercury thus dropped in is regulated according to the appearance of the mercury on the apron, which must never become too fluid.

This mercury combines (amalgamates) with some of the gold, and some of the gold is caught on the internal amalgamating plates. The crushed ore or pulp and some of this internally-formed amalgam, as well as the excess mercury, is discharged through the screen which is fixed in the front portion of the mortar. The stamps weigh from 900 to 1,200 lb., each the heavier ones being the more recent. They are raised by cams and allowed to drop. In most of the mills the stamps drop on an average 92 times in a minute. The latest style of stamp crushes  $4\frac{1}{2}$  to 5 tons in the 24 hours when a 900-mesh screen is used (900 holes to the square inch or 30 to the linear inch). Some mills use coarser, others somewhat finer screens, but in most cases 900-mesh screens are used. In December 1893 the average for 52 mills with a total of 2,175 stamps was 3.65 tons per stamp head per diem. The quantity discharged is fairly even and the pulp flows in a thin film over the amalgam plates G which form the apron, and here a considerable portion of the gold is caught. As the dies wear a chuck-block of a less height is put underneath the screen in order to regulate the discharge.

The photographic view on next page shows the new battery of the Robinson Company. The screen has been removed from the mortar to the right, so that the heads of the stamps with the shoes and dies can be seen.

The amalgam plates are made of very porous copper, which is thoroughly cleaned and then coated with mercury, which forms on the lower surface an amalgam with the copper. The mercury must not form drops, and the upper surface must be kept bright. Some use a little potassium cyanide solution for preparing the plate before the mercury is put on it. The gold being heavy falls on to the mercury, as also does the amalgam which has been formed in the mortar and is there retained. It has been the experience of amalgamators that the coarser and purer the gold the more remains on the plates. It has also been found that the chief amalgamation takes place on the plates within the mortar, and on the first 3 feet of the aprons, which are usually 6 feet wide by 12 feet long. The surface of the mercury has to be kept clean, as above observed, otherwise the gold will not amalgamate. The incline given to the plate, which determines, of course, the velocity of the flow of water, is also a question which is ascertained by experience. If the plate is not sufficiently inclined a great deal of sand and other impurities collect thereon, whilst if it is too much inclined the water carries away too much gold. In some mills, as many as three plates are placed the one after the other in cascade fashion, as it was thought that the drop from the one to the other rendered it possible to collect more gold, but single plates have found more favour, and some of the mills that formerly had three plates have now only one. With very fine gold the quantity carried away by the water is naturally greater. In fact we shall see that the degree to which the gold in finely-divided particles will remain in suspension in water is most remarkable.

This "float gold," as it is termed, and the sulphurets, do not amalgamate on the plates, neither does the gold which is held between the particles of iron pyrites.

I am informed that in most of the mines in the United States, Venezuela, and Australia, the gold is comparatively coarse and pure, and about 75 per cent., in some cases even 80 per cent., thereof is caught on the plates. At the Witwatersrandt, however, only about 55 per cent. to 60 per cent., and frequently less, remains on the plates. I have also been told of some few mines in other parts of the world where the yield on the plates was less still, but 60 per cent. is generally considered a very low percentage, especially for ores averaging under one ounce to the ton.

I understand that in some mines in other countries the plates are continually cleaned by means of india-rubber

stamps without stopping the mill. On the Witwatersrand the stamps have to be stopped for cleaning the mercury, which is done twice a day.

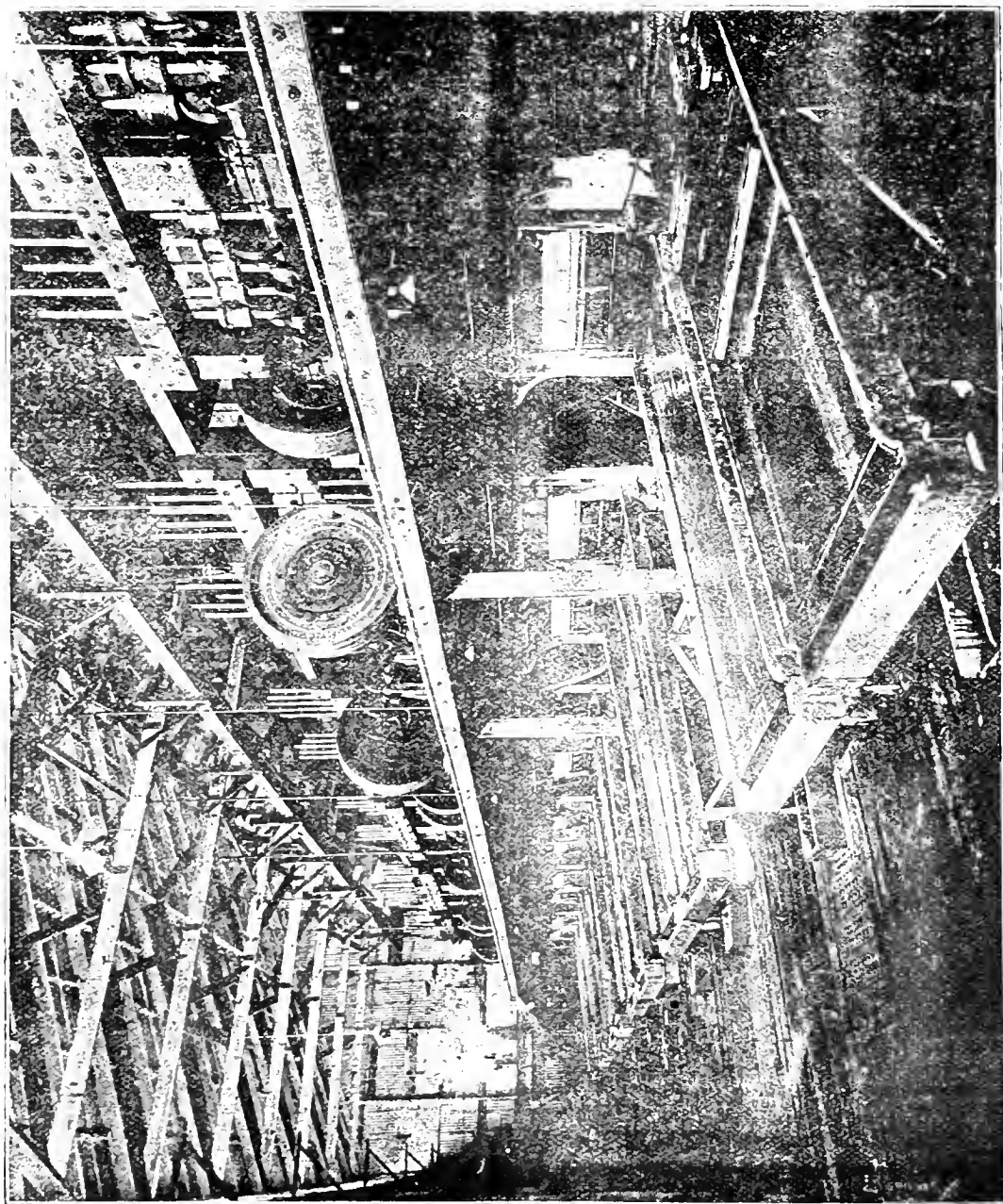
A clever amalgamator, who understands how to prepare and attend to the plates, and how to subsequently treat the amalgam properly, is a man much sought after.

The quantity of mercury lost depends to some extent on the care bestowed on the treatment, but chiefly on the nature of the ore. With heavily sulphuretted ores the loss is much higher than with ordinary quartz.

Thus, with pyritic blanket, in spite of all care, the loss of mercury is high, it being about three-quarters of an ounce to the ton of ore treated, whilst I understand that in California the loss of quicksilver is reckoned at 1 lb. to 32 tons of ore crushed, or half an ounce per ton.

A small part of the loss is usually accounted for by carelessness, and there is always some loss in the hereinafter described cleaning and retorting of the amalgam; but the larger portion of the loss is usually attributed to the so-called "flouring" of the mercury on the plates, which causes some of it to be carried away by the flowing water.

As most of the mines on the Randt are milling highly pyritic blanket, the loss on an average seems to be very close to an ounce a ton. The total amount of ore crushed in 1893 was, in round figures, 2,200,000 tons, which represent approximately a loss of 140,000 lb. of mercury. The mercury is sold in iron bottles containing from 72 lb. to 75 lb., and these bottles are sent out to South Africa. The present price is 5*l.* 10*s.* per bottle, so that the annual consumption of mercury on the Randt can now be put down at about



THE NEW BATTERY AT THE ROBINSON MINE.



2,000 bottles of a value of 11,000*l.*, without taking into account the mercury required for starting new mills.

The gold amalgam which has formed on the plates is collected and cleaned in a specially designed clean-up pan, or else ground in iron hand pans with iron balls, and sometimes in revolving barrels.

The degree to which this grinding operation is carried out is a matter which requires experience.

When sufficiently ground, the amalgam is flooded, that is to say, it is thrown into a quantity of mercury, in which it partly dissolves and partly falls to the bottom, whilst the impurities remain on the top and are skimmed off.

These impurities are collected and mixed with the concentrates, of which we shall have to speak presently.

I have examined some of these impurities and find that they consist mostly of particles of pyrites and particles of silicious matter, both crystalline and amorphous.

The cleansed amalgam, with the excess of mercury, is placed in a press, the lower portion of which consists of a perforated plate, covered with filtering cloth, made of twill, such as is commonly used in filter-presses, and the excess of mercury is pressed off. The dry hard amalgam,

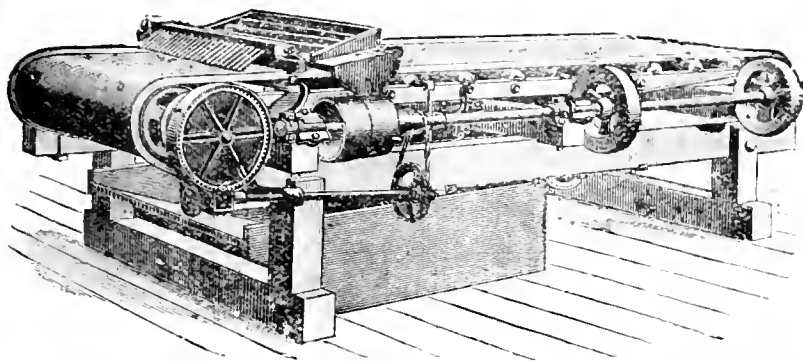
which has a silvery appearance, is then placed in a retort, which ends in a tube surrounded by water.

Heat is applied gradually to the retort, and this causes the mercury to volatilise. The mercury vapour passes through the tube, and is condensed and recovered, whilst the gold remains behind in the retort. This gold is then melted in a large plumbago crucible with a small quantity of flux, and the impurities are skimmed off. The pot, with its contents, is then lifted up, and the gold is poured into cast-iron moulds to form ingots of 1,000 ounces each.

This gold varies from 800 to 850 per mille in fineness. It contains some silver and a proportion of base metals.

The "pulp," as it runs off the copper plates, is conducted in most mills through wooden gutters or sluices, then on to concentrators. In some cases blankets are used, and some mills have Slinger tables, but in most cases Frue vanners, & J., are used, and this latter class of concentrator is becoming more and more general on the Rand.

In all these mechanical devices the principle is the same, viz., to separate by gravity the heavier particles from the lighter.



The Frue vanner consists of an endless inclined rubber belt of 4 feet wide by 12 feet long, supported by rollers and bordered on the sides by rubber flanges. The belt travels up the incline and around a lower drum, which dips into a water tank where the mineral is collected. In addition to the travel of the belt, the latter receives a steady shaking or settling motion from a crank shaft along one side, the shake being at right angles to the inclination and travel of the belt. Over this belt there is a distributor, the lower part of which is formed by a copper plate which is coated with mercury. The pulp enters from above through a 3-foot aperture in the distributor, and some of the free gold which has escaped amalgamation is here caught by amalgamation with mercury. This amalgam is collected and added to that taken from the inside plates and the apron of the batteries.

The rapid shaking motion of the vanner keeps the lighter portion of the pulp in suspension in the flowing water, the heavier portion, consisting largely of auriferous iron pyrites, adheres to the india-rubber, and is brought forward and washed off as the belt passes round into the water tank below, where the concentrates are collected. Water emanating from a row of small spouts washes back the lighter sands and particles which flow over the belt in the direction of the incline, therefore in the opposite direction to that taken by the concentrates, and they are conveyed through wooden sluices into the settling-dams. This pulp which flows off still represents the almost entire bulk of the ore crushed. The concentrates collected amount on an average to somewhat less than 3 per cent. of the total weight of ore crushed, and I understand that in other parts of the world where pyritic ore is treated the percentage of concentrates collected is about the same.

There is a considerable amount of discussion as to the importance of having the best mode of concentration. Most of the experts on the Randt consider careful concentration of the greatest importance, and the Chamber of Mines of Johannesburg decided to offer a prize—I believe it was

3,000*l.*—for the best concentrator. Some, again, consider concentration superfluous, and in some mines it is dispensed with, the pulp being treated differently, as I shall mention further on.

Most mines concentrate, and the Frue vanner system is the one at present most in favour.

The concentrates are very rich in gold, containing from 5 to 8 oz. to the ton. They are roasted and treated by chlorination.

Most of the mines sell their concentrates to one of the two chlorination works at the Randt, viz., the Robinson Company or the Randt Central Ore Reduction Company. Mr. Charles Butters designed both these works, started the former, and is now managing the latter, so that the methods employed are practically the same in both.

The mines sell their concentrates at 90 per cent. of the assay value, less 4*l.* per ton for treatment.

In treating concentrates by chlorination 95 per cent. of the gold is extracted, and in addition to this difference between 90 per cent. of the assay and the actual extraction there is a profit on the 4*l.* for treatment. What the chlorination treatment actually costs in the Transvaal it is difficult to say, as a good deal must depend upon the manner in which the general expenses are calculated, but I have heard the figure of 3*l.* mentioned as the average cost. Of course it would not be possible to treat any but rich material by this method, which requires nearly an ounce of gold per ton to cover the cost.

The concentrates are placed in reverberatory furnaces, with abundant admission of air. The consumption of fuel to effect the roasting is 60 per cent. of the weight of the concentrates.

It must be borne in mind that, as stated already, this would mean about 35 per cent. were English coal used, as the Transvaal coal makes so much more ash.

The percentage of iron pyrites ( $\text{FeS}_2$ ) in the concentrates varies considerably, but is seldom more than 35 per cent. or 40 per cent., so that the concentrate

rarely contain more than 20 per cent. to 25 per cent. of sulphur. I have, however, known of samples to run as high as 40 per cent. of sulphur.

The purpose of thus roasting the concentrates is to drive off the sulphur and any arsenic which may be present, to oxidise the iron pyrites to ferric oxide (red oxide of iron  $\text{Fe}_2\text{O}_3$ ), to disintegrate the dense cubic crystals of the pyrites, and to oxidise the other base metals.

At the Robinson mine common salt is, towards the close of the operation, thrown into the furnace to assist the oxidation and produce a preliminary chlorination. I understand that at the Randt Central Ore Reduction Company's Works this is dispensed with. Whether salt be used or not depends upon the temperature of the "roast" and the air supply.

In addition to the reverberatory furnaces, finishing hearths are used, as it is of the greatest importance to roast the ore dead, the smallest percentage of sulphur affecting the

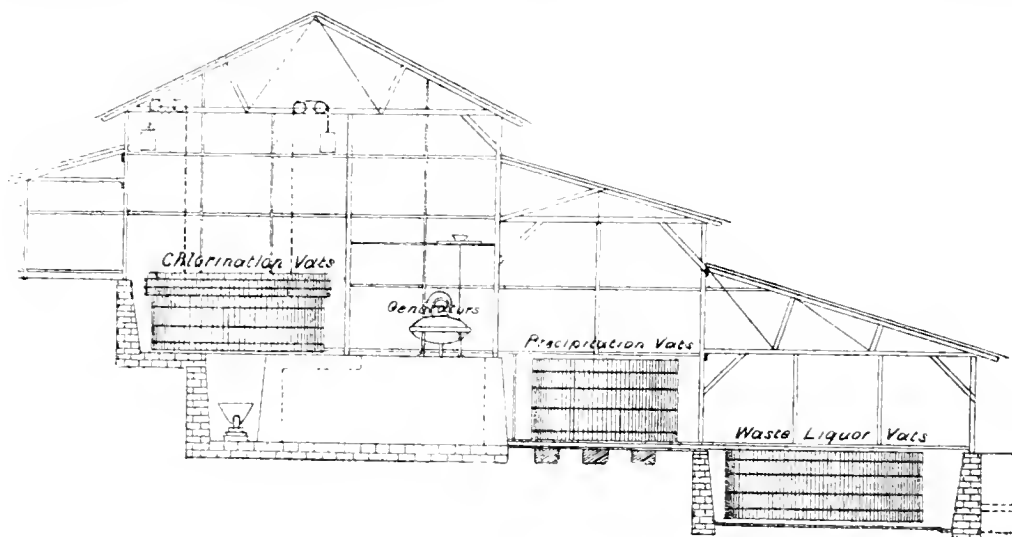
extraction considerably;  $\frac{1}{2}$  per cent. of free sulphur would make a difference of 15 to 20 per cent. in the yield. I am told that a bad roast can never be cured.

During the roasting dust accumulates in the flue boxes, and as this contains gold, sometimes in considerable quantities, the flues are periodically cleaned and the flue dust mixed into the next charge of concentrates.

The roasted concentrates are moistened. They ought only to contain 6 per cent. of moisture, but as a rule the moisture varies, according to the condition of the atmosphere, between 8 and 12 per cent.

Chlorine gas has no effect on the dry material; any excess above 6 per cent. of moisture is found to cause a waste of chlorine.

The roasted and moistened concentrates are placed in large wooden vats, termed chlorination vats, the bottom of which is fitted with a filter made of brick, pebbles, and sand.



These vats are closed with a lid rendered gas-tight by a water lute. Chlorine gas is introduced from below and allowed to act from 18 to 36 hours.

The chlorine used is produced at the works in lead-lined steam jacketed generators, in which dilute sulphuric acid, salt, and manganese dioxide are mixed; the chlorine thus produced contains about 6 per cent. of  $\text{HCl}$ .

When it is judged that the gas has been in contact with the ore long enough to convert all the gold into the chloride—18 to 36 hours—water, preferably warm, is introduced, and the chlorine gas is allowed to pass into another vessel. The auric chloride, being soluble in water, enters into solution and is washed, or, to use the technical term, "leached" out.

The solution is run into tanks (marked on the drawing as "precipitating tanks"). This solution contains, of course, the chloride of gold and small quantities of chloride of silver; this latter salt being but slightly soluble in water in the presence of other chlorides, most of it remains in the residue. In addition to this, we find in the solution copper, cobalt, alumina, lead, traces of iron, traces of arsenic, traces of lime, and, when salt is used, sulphate of soda.

The ore having been washed or leached several times is dug out and thrown away. These residues contain only 2 or 3 dwts. of gold to the ton.

The chlorination liquor contains, besides the above impurities, a good deal of excess chlorine and a considerable quantity of oxide of iron and silica, which escape through the filter into the solution.

Sulphuric acid is added to keep the basic salts in solution, and then ferrous sulphate ( $\text{FeSO}_4$ ) is added.

This ferrous sulphate is made at the works. At the Robinson mine it is made in a most peculiar but evidently very practical manner. The sulphuric acid has, until

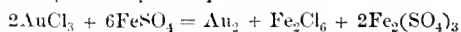
recently, been obtained from Europe in iron drums, which are perfectly valueless. Water is poured into a vat, then sulphuric acid is added, and the iron drums are thrown in and allowed to dissolve.

The ferrous sulphate thus obtained is, together with the excess of dilute sulphuric acid, run into the chlorination liquor.

I may here mention that for some time past sulphuric acid works have been erected in the Transvaal about 15 miles from Johannesburg, but the consumption is comparatively small, amounting to 500 tons per annum at the utmost, and hence a high price has to be charged, especially as the cost of labour and of all imported articles is very high. (Nitrate of soda, 24*l.* per ton.)

The cost of imported sulphuric acid was about 25*l.* per ton laid down at Johannesburg. Now there is a heavy duty on it so as to protect the Transvaal Sulphuric Acid Factory, which sells chamber acid at 20*l.* per ton. (They have no concentration plant.)

The action of the ferrous sulphate upon the dichloride of gold is explained by the equation—



The gold precipitated by the ferrous sulphate appears of a purplish colour, and when examined under the microscope it is found to be in cubic crystals. This gold is collected, calcined, and melted with borax and carbonate of soda. The gold falls to the bottom of the crucible and is, when cold, broken away from the slag and collected. When sufficient has been obtained it is melted over again and formed into ingots. This chlorination gold varies from 870 to 980 per mille in fineness. It contains a good deal of lead, some iron, and a little copper, with traces of arsenic, as, in spite of the addition of sulphuric acid, some of the arsenic



is precipitated with the gold. Although as fine as 980 1000, this chlorination gold is at times very brittle and very difficult to retine. Mr. Denker, the senior chemist at the Mint in Pretoria, mentioned some very curious points regarding the obstinacy of some of this gold, which remained brittle after the most careful retining.

The liquor from which the gold has been precipitated still contains some gold in suspension and in solution (1 to 2 dwt. per ton of liquor). This is due to the dissolving action of the ferric chloride ( $\text{FeCl}_3$ ) counterbalancing the precipitating action of the ferrous sulphate. To obtain the gold left in the liquor it is run into another tank and allowed to stand over scrap iron, which reduces the ferric chloride into ferrous chloride. The liquor, which was acid through the presence of some of the hydrochloric acid contained in the chlorine and the sulphuric acid, then becomes neutral through the action of the iron, and the gold, together with traces of copper and lead, as well as basic arseniate of iron, are precipitated. The residue is collected, and contains a considerable amount of gold, sometimes as much as 200 oz. of gold to the ton dry weight. Of course the quantity collected is very small.

At the Robinson works, in which about 10,000 tons of concentrates are treated annually, they collect about six tons of this residue in the year. The residue thus collected is mixed with the concentrates in the furnace and roasted with them, as is the case with the impurities collected from the amalgam, as already described.

In this chlorination process the quantity of ferrous sulphate used is greatly in excess of that which is required to bring about the reaction, chiefly because the excess of chlorine and the impurities in the liquor are very considerable. Several means of remedying this have been suggested, but, as yet, none have been adopted.

We will now return to the lighter portion of the "pulp," which we last saw floating off the concentrators into the settling dams. These are large reservoirs constructed like water reservoirs. In the first dam the heavier particles settle. These are called "tailings," and consist of very fine sand. The water flowing off these tailings carries with it the still lighter particles, which settle in the next dam. This very light material is termed "slimes," or more correctly "battery slimes." When dry these slimes present the appearance of impalpably fine dust in caked lumps. They are soapy to the touch, and are chiefly composed of the silica which holds the pebbles together. The tailings have a sand-like appearance.

The tailings amount as a rule to 60 per cent. of the weight of the original ore crushed, and contain from 5 to 7 dwt. of gold to the ton. It was, of course, indispensable to have a cheap process to make it possible to treat these large quantities of comparatively poor material; the MacArthur Forrester cyanide process has supplied the want, and it is by this that the tailings are treated. The slimes, which amount to about 40 per cent. of the weight of the total ore crushed, contain 1 to 5 dwt. of gold to the ton, and have not been treated up to the present, for reasons which I shall be better able to explain further on. They constitute absolute loss.

The majority of the mines have cyanide plant, or are erecting the same; cyanide plant in course of erection was, as I have already observed, a leading feature in the district when I was there.

The cyanide process consists roughly in bringing the ore into contact with a solution of cyanide of potassium, to get the gold into solution, and then precipitate it out of this solution by means of zinc shavings.

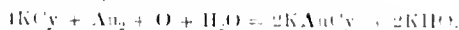
Mr. MacArthur, in a paper which he read before this Society (this Journal, 1890, 267—270), gave an account of the MacArthur Forrester process.

It was, I believe, at the Randt that this process was first introduced on a large scale. It was soon found nearly impossible to agitate large masses of material in the vats, as at first suggested, and percolation had to be resorted to.

Circulating pumps were introduced, and the liquor allowed to percolate several times through the ore, whereby the surface contact was greatly increased.

Messrs. Butters and Clennel published in the "Engineering and Mining Journal" for 1892 two papers entitled "The

Chemistry of the Cyanide Process" and "The Cyanide Process in South Africa," which were reprinted in the Journal of this Society (November 1892), and from these it can be gathered how the process was applied at the time, and the opinions which prevailed as to the chemistry. The equation there given for the reaction is:



The authors further express the opinion that no doubt the oxygen converts a portion of the cyanide into cyanate, which, being unstable, is converted into the carbonate of potash, carbonic acid, and free nitrogen.

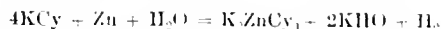
Messrs. MacArthur and Forrester recommend 75 per cent. cyanide, but a good deal of 90 per cent. was being used, and some mines were using 98 per cent.

The zinc replaces the gold in the auric potassic cyanide, according to the following equation:—



Metallic zinc alone precipitates gold very slowly if in dilute solution, but once gold is deposited even in traces on the zinc, this is converted into a galvanic couple—gold-zinc—and the galvanic action actuates the reaction with evolution of hydrogen.

What takes place can no doubt be expressed by the following equation:—



and this hydrolysis accounts for the fact that the quantity of zinc used is more than 100 times as much as is theoretically necessary in accordance with the first formula above given. The use of caustic soda, to which I shall refer hereafter, still further increases the consumption of zinc, because of the reaction—



The subsequent chemical reactions are excessively complicated, and of course the impurities contained in the solution play a considerable part.

It has been found that pure zinc will not precipitate the gold as readily as commercial zinc will, in consequence of the presence in the latter of a considerable percentage of lead, the presence of this third metal evidently increasing the galvanic action.

The very finely divided state of the gold in the Witwatersrandt gold ore, which is such a disadvantage in amalgamation, and which is the cause of the slimes running so high, is a considerable advantage as far as the cyanide process is concerned, because of course the infinite number of microscopic specks increases the amount of surface contact, and these minute particles are more readily dissolved than coarser gold would be.

The zinc is prepared in shavings, in order to increase the surface contact as much as possible. One pound of zinc, when cut up in the manner in which it is done at these mines, represents, I was told, an area of five square yards, or 2·31 acres to the ton. These zinc shavings can be closely packed, and then form a somewhat elastic bulk which is almost felt-like, and it is with this that boxes are filled, the zinc resting on sieves to enable the solution to rise up evenly through it, and also to afford means of easily removing the zinc from the deposited gold. There are several such boxes divided into compartments; those into which the strong solutions are led are termed "strong boxes"; the "weak boxes" being those in which the weak solutions are treated.

Although the arrangement may vary in detail, it is very much the same at all the mines.

The cyanide works consist of large tanks, as shown in the following drawing:—

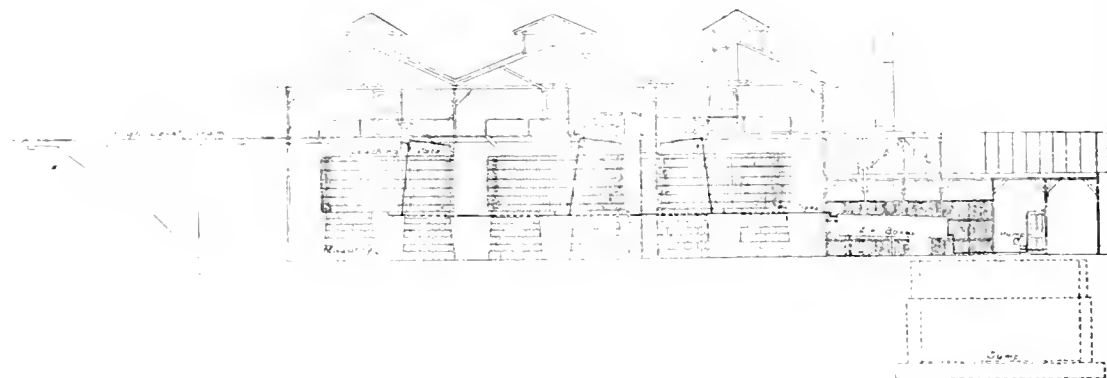
These tanks, in most of the works, hold from 70 to 100 tons of tailings, and in addition one-third as much solution; so that one of these tanks will hold, say, 75 tons of tailings, and 25 tons of solution. At some works the vats are very much larger. At the Langlaagte Estate Mine, the tanks hold 400 tons of tailings, and the mechanical arrangements are somewhat different to those herein described.

The tanks are lined with a filter consisting of wooden framework, on which canvas sacking and cocoanut matting are placed in superposed layers. This arrangement varies a little in the different works. Below this filter there are

the eduction pipes, and at the very bottom of the tank there is a large central discharge closed by a door conveniently clamped.

The reason for this latter arrangement is that the tailings after treatment cannot be dug out, as are the chlorination

residues, on account of the poisonous cyanide which still remains in them, and they have therefore to be taken out through this door, and dropped into trucks running on a tramway, which passes between the stone-work which supports the tanks.



At some of the mines the tanks are, by means of a circulating pump, connected with sumps or reservoirs: in some the circular pumps have been dispensed with.

At the mines at which circular pumps are used the solutions are pumped on to the tailings, and allowed to percolate and run into the sumps, care being constantly taken to test the strength of the liquor. As a rule a weak solution is first passed through the tailings and allowed to run on to the weak zinc boxes, then a strong solution is pumped on to the tailings and run into a sump, then another strong solution is pumped on, so that ultimately the strong solution and the weak solution has been allowed to remain in contact with the tailings three times for 12 to 18 hours each time, and then the solutions are run into the zinc boxes. After all the solutions have been run through, the tailings are thoroughly washed with plain water, which is also allowed to run over the weak zinc boxes, or run into the sumps to be used in making up liquor for a subsequent charge.

At the mines at which no circulating pumps are used a succession of strong and weak solutions is used, the same as above mentioned, and they are also left in contact for 12 to 18 hours, but there the solutions are at once run into the zinc boxes. It is held by some that it is not safe to pass the liquor more than once through the ore, as they believe it causes a loss of gold.

The strength of solution varies somewhat at the various works, but the general tendency is to reduce their strength, as it has been found that weak solutions give better results.

The strongest solution I have seen used is 0.6 per cent., in which case the weak solution used was 0.3 per cent. In other works 0.4 per cent. and then 0.2 per cent. were used, and I have even met with 0.3 per cent. as strong and 0.1 per cent. as weak solution.

The amount of cyanide required to dissolve 190 ounces troy of gold is theoretically 1.53 lb., but in practice it is found that it takes at the very least 300 lb. to extract 100 ozs. of gold out of 400 tons of tailings.

This would mean  $\frac{3}{4}$  lb. of cyanide per ton of tailings treated, but none of the mines are working as low as this. Besides, it has been found that the more pyritic the ore the more cyanide is necessary. I found that at some of the mines they were using 2 lb. of 90 per cent. cyanide per ton of tailings, whilst at others the quantity was considerably less. At the Robinson mine, as will be seen from the statement given further on, the average in 1893 was 1.16 lb. per ton of tailings treated. In other mines, again, I understand that the quantity sometimes exceeds 2 lb. The average is no doubt below that figure.

To arrive at an approximate idea of the quantity of cyanide used on the Randt, I believe that 2 lb. per ton can be taken. This would mean for the 1,200,000 tons of tailings treated in 1893 about 1,200 tons for last year.

The consumption has been steadily increasing as one mine after the other erected cyanide plant. On the 31st December last year there were only a few mines which had not completed their arrangements for cyaniding their tailings.

The following shows the quantity of ore crushed, and the quantity of tailings treated in each month of 1893:—

1893.	Tons of Ore Crushed.	Tons of Tailings Treated.
January .....	174,546	43,623.80
February .....	141,763	57,517
March .....	177,291	69,778
April .....	169,106	69,800
May .....	174,973	78,483
June .....	173,212	78,828
July .....	186,216	93,819
August .....	201,391	107,586
September .....	192,858	113,217
October .....	201,141	123,513
November .....	201,939	139,132
December .....	210,168	136,900
	2,263,794	1,112,196.80

If, therefore, instead of calculating the consumption of cyanide on the tailings treated in the whole of last year, the figure for December alone be taken, we find that the 136,900 tons of tailings represent, at the above average of 2 lb., about 120 tons of cyanide for the month, or at the rate of about 1,500 tons a year.

Bearing in mind that, as we have said, the tailings obtained represent 55 per cent. to 60 per cent. of the ore crushed, it will be seen on examining the relative proportions in the above two columns that in the beginning of the year, when a number of mines were not yet in a position to use the cyanide process, the quantity of tailings treated fell considerably below this percentage. The figures for the last two months of the year show that the quantity of tailings treated was more than 60 per cent. of the ore crushed.

This is due to the fact that a number of the companies have tailings which accumulated in the settling dams prior to the erection of their cyanide plant, and are now working off these old tailings.

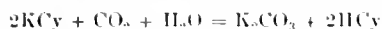
The chief reasons why the consumption of cyanide in the tanks is so enormously in excess of that which is theoretically necessary are :—

*Firstly.*—That the tailings contain finely divided pyrites. The action of the air on this in the settling tanks causes the tailings to become acid.

The action of ferric and ferrous sulphates upon cyanide (in acid tailings) is probably to form some of the Prussian blues.

This acidity has been remedied by using alkali; formerly lime-water was used; now, however, caustic soda seems to be preferred, most works using about  $\frac{1}{2}$  lb. of caustic soda to the ton of tailings. But although less than in acid solutions the pyrites present still consumes a good deal of cyanide, with formation of iron prussiates.

*Secondly.*—That carbonic acid is absorbed from the atmosphere and produces the following reaction—



whereby volatile prussic acid is formed

*Thirdly.*—That a good deal of cyanide remains mixed with the closely packed, to some extent, porous particles of ore in the tanks.

I may here remark that I was exceedingly struck by the large quantities of tailings which accumulate after treatment, and lie about in mounds all over the place. These still contain a good deal of cyanide, which, of course, is washed away by the rains, which are torrential in the rainy season, and a good deal gradually decomposes through the action of the air. There must, however, be a good deal of prussic acid formed, and this, one would think, might possibly do some harm; in addition to this the frequent high winds whirl the dust to a tremendous height, and are bound to carry a large quantity of this poisonous material into the town. Up to the present, however, no inconvenience has been felt from this.

In the works no ill-effects have been experienced from the prussic acid which escapes from the tanks, the odour of which is clearly perceptible, and with the exception of two kathirs, who, some considerable time ago, drank some of the water flowing from the cyanide works, I have been told that there has been no death due to cyanide. Occasionally a cow fancies the light brown liquid, and of course meets an untimely end.

A number of suggestions have been made and patents obtained for reducing the quantity of cyanide used, but I know of none (except the addition of caustic soda) which has, so far, been adopted.

When the cyanide liquors have left the tanks they run over the zinc boxes, as already mentioned. The zinc must be placed in the boxes in such a manner that, while not retarding the flow of the solution, it will cause the liquor to meet as much zinc surface as possible. The liquor runs through several successive boxes, but by far the largest amount of gold is precipitated in the first two. From the boxes the liquor runs into the snags, is there brought up to strength, and used over again.

With time the zinc all breaks down. The zinc boxes are emptied twice a month. Such zinc shavings as still remain are thoroughly washed with water, and the gold precipitate passes through the coarse retaining sieves on which the zinc shavings rest in the zinc boxes; these retain the zinc shavings which have not been decomposed. The heavier precipitate, or "gold slime" as it is called, remains on a filter placed beneath the sieve. At some mines the finer slime is filter pressed. All the slimes are then collected in basins and dried. These gold slimes contain a great deal of zinc and lead, also small quantities of silver, copper, traces of arsenic, alumina, and cobalt. In addition to this they contain potassic zinc ferrocyanide, zinc cyanide, zinc sulphide, ferrocyanide of iron, potassium carbonate, carbonate of lime, and in addition thereto ferric oxide and silica, some of which always escape from the tanks through the filters.

This material is calcined in order to oxidize the base metals and decompose the cyanides. The carbonic acid and ammonia formed escape.

The calcined oxidized material is melted with carbonate of soda, borax, and fluor spar, used in about the following proportions :—

	Parts.
Calcined slimes .....	82.0
Carbonate of soda .....	8.0
Borax .....	5.0
Fluor spar .....	4.0
	<hr/> 100.0

and a final covering of borax is given to the contents of the pot.

When thoroughly melted the contents are poured into conical moulds, the gold settling at the bottom. When cold, the gold is broken away from the slag, the pieces collected, and then melted again to be cast into ingots.

This gold runs from 750 to 780 fine. It contains a little silver, but its low quality is due chiefly to the presence of lead from the commercial zinc, and it contains zinc, copper, and other impurities in varying quantities.

Of course, the more thoroughly the calcining is done, the better the gold, but as in any case it has to pass through the refiners' hands before it is fit for use in the arts or at any mint, no special care is, as a rule, taken to improve this gold. It may be interesting to remark here that for the month of December 1893 the cyanide gold represented 37,451 oz. out of a total of 141,085, so that, approximately, 25 per cent. of the gold produced was cyanide gold.

The sumps are cleaned out from time to time and cleared of the clayey brown deposit which settles in them. This deposit is formed by the action of the potassic zinc cyanide formed in the boxes on potassio ferrous ferrocyanide in the presence of air and alkali.

Curious to say, this deposit is found to contain some gold.

The calculation of the cost of the cyanide process, as carried out on the Randt, must depend to a certain extent on the manner in which general expenses and depreciation are distributed over the various sections of the treatment, and the mines which work very large quantities must necessarily work cheaper than the smaller concerns.

I have found that generally about 6s. (exclusive of royalty) per ton of tailings is taken as the cost of cyaniding. This is borne out by the following statement of the cost of cyaniding contained in the report of the Robinson mine for 1893, which has been published in the March number of the South African Mining Journal :—

	Cost per Ton.			
	£	s.	d.	s.
Wages (whites and natives, including food) .....	3,496	11	8	1 2 81
General supplies (stores, assay material, &c.) .....	1,408	9	1	0 6 12
Fuel .....	1,204	19	9	0 5 24
Cyanide, 61,431 lb. = 1.16 lb. per ton. ....	5,563	0	2	2 0 19
Zinc, 12,521 lb. = 0.23 lb. per ton. ....	269	4	3	0 1 13
Contractor (filling and discharging vats) .....	4,325	12	2	1 6 81
Royalty .....	3,768	1	1	1 4 38
	19,036	18	2	7 2 68
Tons of tailings treated .....	55,230	tons (each 2000 lb.)		
Fine gold contained in above ..	21,180	oz.		
Fine gold extracted from above ..	13,872	oz.		
Extraction .....	68.7	per cent.		
Bullion returned .....	17,921	20 oz.		

One of the very largest mines, which is working about four times the quantity of tailings which the Robinson mine treated in 1893, claims, I understand, to be able to work the cyanide process for 4s. 9d. per ton, exclusive of royalty.

The owners of the MacArthur Forrest patent are, under their various agreements with the mines, receiving royalties which vary from  $7\frac{1}{2}$  to 10 per cent. of the value of the gold

obtained by the cyanide process. I have heard their present income from the Witwatersrandt estimated at from 75,000*l.* to 100,000*l.* per annum.

Some of the mines under their licence are bound for a certain period to take all their cyanide from the patentees, *i.e.*, The South African Gold Recovery Company.

It is quite natural that under these circumstances many are trying to find methods to supersede the MacArthur Forrester process, and inventors seem, till now, to have directed their attention chiefly to finding some other cheap method of precipitating the gold out of the cyanide solution.

The zinc method has the disadvantage of wasting a considerable amount of cyanide through the formation of the zinc salts, and the liquor, which is used over again, is when brought up to strength less effective than fresh liquor, through the presence of the zinc salts and impurities. On the other hand zinc is cheap, and it is easily separated from the gold.

I have seen the new process patented by Messrs. Siemens and Halske, whose representative, Dr. von Gerret, was working at the Randt Central Ore Reduction Company's works. They precipitate the gold out of the cyanide liquor by means of electricity, using iron as + electrode and thin lead foil as - electrode. The gold and silver is deposited on the lead, whilst the cyanide is deposited on the iron, and there forms Prussian blue. The iron is coated with a special material, which causes the Prussian blue to adhere to it instead of floating in a flocculent condition in the liquor, as it otherwise would. This Prussian blue is, by treatment with carbonate of potash, converted into cyanide.

The gold and silver are separated from the lead foil by cupellation.

It is claimed that the cost of this process is about the same as that now in use, with the advantage of the regained cyanide, in addition to which the liquor after treatment is purer than the liquor which has been through the zinc boxes.

Dr. von Gerret was working with 10 volts, and using about 10 amperes per ton of ore. I have since learned that any tension over 6 volts decomposes the cyanide to some extent. As far as I am aware the process has not yet been introduced at any of the mines.

Just as I was leaving, Mr. André arrived at Johannesburg, from the Gold und Silber Scheide Anstalt at Frankfurt, to try a new process, according to which the gold is precipitated from the cyanide liquor by means of aluminium.

I have mentioned that opinions are very divided as to the utility of concentrating, as, after all, only 3 per cent. of the ore is recovered; some therefore think that these concentrates are best left with the tailings and treated together with them by the cyanide process.

I have heard it stated, on the one hand, that when concentrates are treated by the cyanide process the extraction is practically nil, because the cyanide will not attack the gold which is finely distributed between the pyrites crystals; whilst others have told me that they have been able to obtain 70 and even 75 per cent. extraction.

Some mines are being worked without concentration, or by what is termed the direct method. I have seen the arrangement at one of the mines thus worked.

When the pulp flows off the amalgamating copper plates it runs through wooden gutters, but instead of falling on to concentrators it is led into a hydraulic separator—a kind of Spitzkasten—where the battery slimes are separated from the sand. The slimes are run to waste and the sands are directed into the tanks, and there treated with weak cyanide solution, *viz.*:—0.32 three times, in contact for 12 hours each time; 0.15 once, in contact for 12 hours; 0.08 once, in contact for 12 hours, and lastly the sands are washed out with water. Although the sands have not been exposed to the air to the same extent as the tailings which have settled in the dams they are found to be acid, and about  $\frac{1}{2}$  lb. of caustic soda per ton of sands treated is used.

Here, again, the slimes have to be separated out, and they represent about 40 per cent. of the ore crushed, and contain about 1 dwt. gold to the ton. The reason why these slimes cannot be left with the sands is that it is impossible to get the liquor to percolate, the slimes forming a mud which clogs the settling sands and stops the flow,

although in the more recent works vacuum pumps are used to assist percolation. The difficulty in the way of treating the slimes therefore, either with the sands or tailings or separately, is a mechanical one.

I have heard most varied statements as to the yield obtained by the cyanide treatment. I understand that the Gold Recovery (MacArthur Forrester) Company claim an average extraction of 70 per cent. Some have told me that they have obtained as much as 75 per cent., while others say they only get 60 per cent. Then, again, I have heard that the percentage of extraction is very unreliable, and that whilst it may be 70 per cent. one month it may fall to 50 per cent. the next. Of course these calculations depend upon the assays made, and it is very difficult to draw a reliable average sample, and the conditions vary in the different mines.

In the returns issued by the Chamber of Mines the average yield per ton is given, but not the assay value, so that no reliable figures are obtainable.

The direct method has the advantage that the ore is not handled as much as in the other, thus avoiding the loss which always occurs in handling large quantities. Moreover, the expensive chlorination process is avoided, and it is a matter of calculation whether the lesser yield is more than compensated by the difference in cost of treatment.

The total extraction at the mines from all sources is about 90 per cent. of the assay value. The average yield for 1893 was 14 dwt. to the ton of ore treated, which is equal to about 15  $\frac{1}{2}$  dwt. assay value.

I have endeavoured in the foregoing to describe what I have seen, and to lay before you the information I have gathered whilst in the Transvaal and since my return. There can be no doubt that a gigantic industry has sprung up in a very short time, and that all the elements are there for further development. But, as you will have seen, there is in addition to the question of mining, *i.e.*, beyond the judicious exploitation of the reefs, and economical underground working, a number of factors which, taken together, can only be described as an extensive chemical industry. Besides the mine properly so called, there are works which can compare to some of our large factories, and here, as in all other industries, large works must necessarily, through the distribution of expense over larger quantities, work cheaper than the smaller ones.

Management and technical skill must likewise contribute largely to the success of any one individual undertaking; and, quite apart from the value of the property, concerns may succeed or fail from other causes. This gold industry, although subject in many respects to the same vicissitudes as other industrial enterprises, has, however, over all others, the great advantage that there is an unlimited market, and a constant value for the finished product when obtained.

#### DISCUSSION.

The CHAIRMAN said Mr. de Mosenthal had succeeded in giving in the short space of an hour and a half a valuable summary of his observations, which must have been very carefully made, of a very important industry, one which a few years ago could have had no existence, because people would not have known then how to conduct these large operations, handling enormous masses of material to recover what was after all an exceedingly small percentage of a valuable constituent therein.

Mr. MACARTHUR said that it was now quite two years since he left the Randt and came home, and in that period there had been a tremendous development in the mechanical details of the processes used, and not only so, but the chemistry of the subject had been much more studied. What struck him as a chemist was that within the last few years chemistry had been taking its proper place in the production of gold. Hitherto the extraction of gold had been left rather unfairly in the hands of engineers. Very few chemists had drifted into that industry, but it was now being benefited by the chemical skill which was expended on it, and evidently this was only a beginning. As years passed on in the Randt and else-

where, he had no doubt they would be able to bring out the last 2 or 3 dwt. of the gold by the combined influence of chemical and engineering skill.

Mr. H. LOUIS, said that it was about seven years since he milled any stuff on the Randt, and it seemed to him that the industry had made most extraordinary strides since then. It really seemed almost incredible to him that these enormous works could have been raised up within the last few years. When he was there the only permanent building on the whole of this enormous goldfield was a little canvas canteen about 10 feet square. He slept with about 12 men under a waggon-tilt under blankets, and the canteen was the sole representative of the enormous buildings which had sprung up in 10 years. Then he saw nothing but the surface reef, the thoroughly oxidised reef as it was called, and he should like to ask a question with regard to that. He was rather inclined to differ from Mr. de Mosenthal if he understood him correctly to say that the various colours he saw through the microscope of the different kinds of gold were due to alloys with various metals. He had been working on this question for some time, and had come to the very strong opinion—he could not call it conviction—that gold was certainly dimorphous and probably polymorphous, and that these different colours were due to absolutely different forms of gold. The question was a most complicated one, and had a very serious bearing on the question of gold-milling. He proved to his own satisfaction, and he believed he was one of the first to find it out, that certain forms of gold, under certain conditions of deposition, were not attacked at all by mercury. It could be precipitated in such a form that it would not be attacked by mercury at all. Much of this Randt gold was highly crystalline, and there was some evidence to show that it was deposited *in situ*. It was not ordinary river gravel, but gravel into which the gold had percolated and been deposited there. The fact of the gold being thrown down from solution led him *a priori* to suppose it would be under conditions in which it would resist amalgamation, and that was exactly what took place. A certain proportion of the Randt gold was amalgamable, and a certain proportion was not, and he entertained a strong opinion that that was due to its polymorphous condition, and Mr. de Mosenthal's experience of the different colours of the gold seemed to him to point in that direction. He should like to ask if he understood the author correctly when he said that these so-called reefs maintained the same angle all the way down. His impression was, from the faint surface scratching which had only been carried out in his day, that the reef showed a tendency to flatten, as the subsequent borings seemed to prove. He had the idea that they had discovered the opposite rim of the basin, but that was a point he could not say he knew much about, as he was not there from the very beginning, but he should like some further information on that subject. After a good many years' gold-milling in many parts of the world, including America, he thought an extraction by amalgamation of 80 per cent. to 90 per cent. was thoroughly exceptional; he himself had never seen it, and he should say wherever they got 75 per cent. from milling gold in the ordinary run of stuff, they were doing remarkably good amalgamating; he knew very few instances where it was exceeded. He might also suggest that the excess of copper in the gold doubtless came from the copper plates themselves. It was his invariable experience that when one ran fresh plates one found more copper than after they had been running some time. In connection with that he would like to ask whether electro-plated plates were not being used on the Randt. In the States of America the plates were nearly all electro-silvered before amalgamation. There was an immense deal to be said on that subject which he could not enter into then. With regard to the cyanide process he should like to ask whether it was an actual fact that pure zinc would replace gold in a solution of auro-potassic cyanide, or was it a much more complicated question? His own impression was that, if you dissolved gold in a cyanide solution and put pure zinc in, you would not get any precipitation at all. He believed the thing was much more complicated than was thought. In conclusion, he could not help pointing out that the diagram of a 10-stamp

mill was misleading, inasmuch as it showed the tops of the dies 2 or 3 ins. above the string frames. Such a thing could not run for five minutes.

Mr. BOVERTON REDWOOD said he was ignorant both of the chemistry and the mechanical details of this subject, but he was sure he would be supported by everyone in moving a very cordial vote of thanks to Mr. de Mosenthal for his most interesting paper. He might say that he now fully understood for the first time how it was that with these complicated processes one might have exceedingly rich gold-bearing properties, which nevertheless would be pecuniarily unsuccessful. It was quite obvious that there was great room for mismanagement, and, on the contrary, opportunity for very considerable improvement, in organisation and in the processes of extraction through the application of chemical talent and engineering skill.

Mr. WATSON SMITH very heartily seconded the vote of thanks. In doing so he should like to call attention to a most interesting communication which had been sent to him by Dr. George Gore, F.R.S., of Birmingham (see page 428). In a series of experiments made recently by Dr. Gore on the action of finely-divided silica upon solutions of various salts, acids, and alkalis, this authority had specially experimented with cyanides of potassium and sodium. In the case of most of the substances in solution, Dr. Gore found that if the solution were shaken up with the silica (of course in comparatively large bulk compared to the weight of dissolved substance) and then allowed to settle, and the supernatant liquid subsequently examined, a considerable quantity of dissolved substance or salts would be found to have disappeared. If the supernatant liquor were poured off, and that left behind and which had soaked into the silica examined, it would be found to have gained in dissolved substance. Indeed, Dr. Gore suggested that finely-divided silica would prove a useful means for abstracting small quantities of iodine from weak solutions thereof, the sand being subsequently heated to set free and recover the iodine. That gave an idea of the extent to which this singular action went on. Turning to cyanides of potassium and sodium, he said the effects of silica upon weak solutions of cyanide of potassium indicated that the great loss of the latter substance in the commercial process of extracting gold and silver from powdered quartz was largely due to the "adhesion" of that salt to the silica. He remembered when, a few years ago, he, in conjunction with Mr. Graham Young, read a paper on experiments tried on the laboratory scale with the MacArthur-Forrest process, that Mr. MacArthur drew their attention in the discussion to the importance of washing the ground quartz with water after the cyanide treatment. Such a washing in good filter-presses was probably the means of overcoming the difficulty referred to in so interesting and striking a way by Dr. Gore. Nevertheless there was no doubt that were it not for this remarkable property of fine silica, mere filter-pressing would then represent what now filter-pressing plus a good deal of washing combined represented, or, in other words, a larger mass of extremely weak wash-liquors than would have been necessary had no such property existed in silica. Other inert solids in powder were also tested, but finely-divided silica exhibited this property of "adhesion" in the most marked degree.

Mr. SUTHERLAND said he should like to add a word if only to place on record his appreciation of what Mr. de Mosenthal had done. He had the pleasure of seeing this paper a few weeks ago, and was struck at his anxiety in endeavouring to bring before a meeting in one evening so large a subject, but he had done so most successfully, and had been able to focus it in a way which had greatly interested them all. It occurred to him that it would be a great advantage to the London Section if they had a few more popular papers of this kind upon certain subjects, as the members would then better appreciate special papers following afterwards on particular points, as he hoped they might in this case have separate papers hereafter throwing more light on the chlorination and cyanide processes. It was not many years ago since a Randt man arranged to use a portion of his laboratory for the purpose of

investigating a process for the extraction of gold from solution in cyanide of potassium. He was not quite so successful as he had hoped in depositing the gold from this solution, and after a few weeks asked for assistance, generously offering in return a share in the patent. It appeared that his process consisted in the addition of sulphuric acid, and he obtained a very large precipitate of gold, though after evaporating to dryness and fusing he did not obtain anything like the whole of his gold. It was not until that fused salt, after being dissolved and refiltered, was again evaporated and fused with nitrate of potassium that he got the whole of his gold out. He only mentioned that as a popular illustration of the difficulty of precipitating in the ordinary way.

MR. DE MOSENTHAL, in reply, said that Mr. MacArthur had made a reference to chemical skill and to the position which chemists now might occupy with regard to the gold-mining industry in South Africa. He had himself been struck by the secondary positions given to chemists there, and the fact that in South Africa, as in many countries, the so-called practical men were placed in charge. A man who had worked underground for many years and was no doubt very competent as to the best manner of exploiting a reef, was thought also the best man to conduct the works for the extraction of the gold. It was the same all over the world, and it would be many years before the prejudice would be entirely overcome. Nevertheless, he believed that the chemists on the Rand were making headway, and he was sure that the more they did so the better it would be for the industry.

In reply to Mr. Henry Louis, having regard to the large amount of material he had to deal with in a short space of time, it was quite impossible for him to go into details. As to the microscopic appearance of gold extracted from pyrites, he saw crystalline structure in some of the specks, while others were distinctly amorphous. He considered that the various colours shown in gold by transmitted light were due to a great extent to its physical condition, and that gold is polymorphous has been shown repeatedly. Some maintained, on the other hand, that the different appearances of gold were frequently due to the particles being coated with impurities, whilst others maintained that we were in presence of alloys, the gold containing minute traces of other metals. It was well known that if gold be dissolved in *aqua regia* and precipitated therefrom by sulphurous acid it appeared granular under the microscope, while the gold which had been separated from the mercury by treating gold amalgam with nitric acid, appeared as octohedral crystals. With regard to the high extraction of 80 per cent. over the plates, he had this information from someone who had worked practically in Venezuela and could only give it at second hand. As for the deep levels, time had only allowed him to give a very short description, and he was perfectly aware that it had been found that the reefs flattened somewhat in depth, as in the deep-level borings the reef had invariably been struck 100 feet or more nearer the surface than at first calculated. The substitution of zinc for the gold in the double salt ( $\text{KAuCl}_2$ ) when the precipitation first begins in the zinc boxes seemed excessively probable, and Mr. Louis could, after the meeting, see both the double salts, samples of which had been prepared for him by Mr. Bettel.

The CHAIRMAN said perhaps it was because he was a chemist, but he had a strong impression that an engineer's chemistry was not nearly so good as a chemist's engineering; and especially when a chemical process was concerned it was better to let the chemist be his own engineer. It was very important in these days that a chemist should be something of an engineer.

## Manchester Section.

CHEMICAL THEATRE, OWENS COLLEGE.

Chairman: Ivan Levinstein.

Vice-Chairman: Edw. Schunck.

Committee:

G. H. Bailey.	P. Hart.
F. H. Bowman.	J. M. Irving.
R. F. Carpenter.	E. Knecht.
G. E. Davis.	W. H. Perkin, jun.
C. Dreyfus.	Sir H. E. Roscoe, M.P.
H. Grimshaw.	C. Truby.

Hon. Local Secretary:

J. Carter Bell.

Rank House, The Cliff, Higher Broughton, Manchester.

The name in italics is that of a Member of Committee who retires at the end of the current session.

The following has been elected to fill the vacancy, and will take office in July next:—Committee: W. Thomson.

SESSION 1893-94.

May 4th:—

Dr. Carl Otto Weber. "On the Analysis of India-rubber Articles."

Dr. C. Dreyfus. "On Resist and Discharge Styles with Aniline Black."

Meeting held in the Chemical Club, on Friday,  
April 6th, 1894.

MR. IVAN LEVINSTEIN IN THE CHAIR.

### COMPARATIVE RESULTS OF SOME MODERN SYSTEMS OF SEWAGE TREATMENT.

BY W. NAYLOR, F.C.S.

SINCE the formation of county councils in the year 1888, the prevention of rivers' pollution has received a considerably increased amount of attention, due to the fact that the administration of the Rivers Pollution Prevention Acts is now in the hands of bodies not themselves in default. Moreover, the local authority having administration of the Act is not unduly influenced in cases of manufacturing pollution owing to the preponderance of representatives directly engaged in manufactures. The action of county councils, therefore, in this matter is distinctly towards broad uniformity. And this action, whether it be commendable or otherwise, has resulted in numerous schemes of sewage treatment and various descriptions of plant being placed upon the market for dealing with all kinds of noxious and polluting liquids.

I purpose to-night dealing with the question of sewage treatment only. Manufacturers, as men of business, I presume, may be safely left to manage their own affairs, although I would hardly venture to say the same concerning local authorities on this point. It certainly may appear a bold statement to make, but "by their fruits shall ye know them." In fact, it will probably be admitted that the members of local authorities are elected upon broader and indeed more honourable grounds than an intimate knowledge of sewage treatment, that they represent the ratepayers' interests generally, and that upon technical points they should consult specialists. Unfortunately my experience is that upon matters appertaining to the treatment of sewage specialists are very rarely consulted, and even when they are their advice is considered after, and a long way after, the question of £ s. d.

In the larger corporations the matter is left to a sewerage committee, not universally formed of the most influential and intellectual councillors. Of course, exceptions will at once occur to all of us here, but broadly speaking a sewerage committee is not a committee like the finance, library, and public works committees, and is often relegated to the "practical men" on the council. Now, to these practical men, so called, nothing in the world seems so simple as the proper treatment of sewage. Each one holds in his bosom a scheme destined to solve this complicated

problem and to save the impoverished ratepayers' money. But while this committee of practical men is in solemn conclave assembled, one of the kid-gloved councillors stealthily creeps in with a pamphlet, and from that time forward each in his turn arms himself with a pamphlet defying all comers, and a terrible war is waged on the various systems. It is just this state of things which prompts me to offer the following results, and to attempt roughly to delineate a sort of table from which perhaps a uniform line of action may be suggested. In each town, so far as my experience goes, the discussions on sewage treatment seem to be conducted on altogether different lines. Analyses are put forth copied from various printed pamphlets which I hesitate not to say are absolutely worthless. I lay the results to follow before you, therefore, holding a brief from no one. The samples were taken personally, and not after due notice, but on surprise visits.

At the outset I may take it that however divided the opinion may be regarding the treatment of organic matter in solution in domestic sewage, all suspended matter can be, and ought to be, eliminated by either filtration (in the case of broad irrigation), subsidence, or precipitation. The dissolved impurities or organic matters in solution must claim our common attention as objectionable. They are objected to on the following grounds:—

1. That they decompose, so causing a nuisance.
2. That they are liable to become the home at d breeding-place of disease-producing germs. It has been proved\* that the organic matter in sewage is *per se* harmless except in so far as it robs natural streams of dissolved oxygen, and this only to a very slight extent.†

The micro-organisms present, however, affect it in so much that they cause its decomposition‡ and on this fact certain schemes of sewage treatment mainly depend, viz., the sterilisation systems.

In making a table of the various systems, the plainest line of demarcation can be drawn between sterilisation systems and all others. While the sterilisation systems attack the organism in the open field, so to speak, with the object of killing it outright, all others aim at depriving it of food and so starving it out. In fact, the two systems may be compared to a pitched battle on the one hand and a siege on the other.

But to my mind it has always appeared useless to attack and kill organisms and yet not destroy their food, just as it would be useless to attack and destroy a garrison of soldiers and yet allow the supply of provisions to pass on to the enemy's reinforcements.

One of the earliest systems of sterilisation was the "Amines" system, in which sterilisation is claimed to be brought about by a gas termed aminol. It is said to be generated by trimethylamine and any alkali.

In practice the trimethylamine is added as a certain proportion of herring brine, and the alkali as caustic lime. The evolution of the aminol gas is in exceedingly small quantities, in fact imperceptible. It is claimed, however, that, small as it is, the quantity is quite sufficient to destroy all forms of germ life, and I do not for a moment intend to dispute this. Whether from a bacteriological point of view the effluent is dead or otherwise, I am not prepared to say, but this much is certain, that the organic matter in solution is not appreciably diminished. The effect of this treatment on the Wolverhampton sewage, so far as the organic matter in solution is concerned, is shown in the results given elsewhere, Nos. 1 and 2.

It is claimed by advocates of the sterilisation systems that the decomposition of the organic matter is arrested permanently, since the atmosphere contains only non-pathogenic germs, which exude organic matter as inert or inorganic. This argument, however, cannot stand, as pathogenic ferments can and have been discovered in natural waters.§ They have also been discovered in the atmosphere. As proof of this, one has only to place a vessel containing organic matter, such as beef tea, in the open atmosphere in summer weather, when it will soon begin to decompose and give off most disgusting smells.

Further, I will venture to say that there are very few rivers in England which do not receive crude sewage in some shape or form; and the only condition under which sterilisation systems can approach success is in cases where the effluent has but a short distance to run before being lost.

Another and more recent form of sterilisation system is the "Bacillite." In this case the sewage is intimately mixed with the vapour of a mixture of carbolic acid, cresylic acid, and pine-oil extract.

These three compounds are introduced by means of a small donkey pump into a boiler at high pressure; then as vapour they are introduced into the main outfall sewer by means of a battery of injectors. It is claimed that in the form of vapour the germicidal efficiency is increased 200-fold. Pine-oil extract, it is claimed, only acts as an oxidising agent.

Apart from the sterilisation, the effluent at Kettering, where half a million gallons per day are treated by this method, contained on the 27th of January 1894, 0.6 part per 100,000 albuminoid ammonia.

The "Hermite" process, I think, is the latest which claims to sterilise sewage. Here the sewage is mixed with a certain proportion of sea water, which has previously undergone electrolytic decomposition, and is therefore highly charged with oxides of chlorine, said to be powerful disinfectants. The experiments at Worthing are not yet completed, and it would be premature to say at present more than this, that a sample drawn after treatment gave 1.38 parts per 10,000 albuminoid ammonia, and this at any rate is a consideration which the inventor does not profess to concern himself with.

Dr. Kelly, medical officer of health for Worthing and combined sanitary district of West Sussex, after making a trial has reported: "Since there is no instantaneous decomposition of faecal matter and no sterilisation of sewage, I am of opinion that the process, as far as the late trials have gone, has failed to produce the results which are claimed for it by its inventor."

It must be admitted that the effect of a current of electricity passing through water containing organisms is to kill them, granting the current is of sufficient electro-motive force (about five volts), but its effects upon spores have not yet been demonstrated.

It has been proved that the—

	Volts,
<i>Bacillus tuberculis</i> is killed by an electro-motive force of 2.16	
<i>Bacterium lactis</i> " " " "	2.26
" <i>aceti</i> " " " "	3.24
" <i>allii</i> " " " "	3.3
<i>Bacillus subtilis</i> " " " "	2.72

Granting so much then for the electrical, so far as sterilisation is concerned, I can only refer to the Salford trials for the result of the treatment upon dissolved organic matter, for I am not aware that at any place it has been permanently adopted.

The albuminoid ammonia in the effluent was at times:—

0.25 part per 100,000.
0.17 " "
0.13 " "
0.11 " "
0.17 " "
0.10 " "
0.11 " "
0.11 " "
0.13 " "
0.092 " "

The results are after filtration through sand.

These figures need no comment, but speak for themselves as satisfactory; and as this is the first system mentioned as satisfactory, the next matter of importance is the cost. Various systems of treatment would occur to many people as capable of giving good effluents in which the cost is

\* Emmerich, "Zeitschrift für Biologie," Vol. XIV., p. 563.

† Rivers Pollution Commissioners' Report, 1868—1872.

‡ A. B. Griffiths, "Micro-organisms," pp. 87 and 40.

§ A. B. Griffiths, "Micro-organisms," pp. 45, 47, and 49.



prohibitive—distillation for instance. Now the cost of the electrical treatment at Salford was based upon:—

Estimated horse-power (labour and coal).....	£ 3,700
Consumption of iron.....	4,500
Depreciation and interest on capital.....	3,525
	<hr/> 11,725

This is apart from sludge-pressing (2,070*l.*).

It was the most expensive of the systems tried. One-third of the cost of treatment (exclusive of pressing, the cost of which differed very little from the next higher, the International) was for indicated horse-power, and this was based upon a run of 132½ hours. Now, high as this cost works out, it is probable that in lengthy practice it would work out higher.

The efficiency of the treatment, or, more properly, the extent of purification, depends upon the amount of current passed through the sewage. We may take it that in the electrolysis of sewage (as in anything else) the amount of chemical decomposition is directly proportional to the amount of passing current, and that a given quantity of sewage requires a given quantity of current for a given period of time.

This current has to be passed between the electrodes through the intervening sewage. This intervening sewage offers a certain amount of resistance, the resistance following Ohm's law,† in liquids as in solids, viz., "the current varies directly as the electro-motive force, and inversely as the resistance."

It follows, therefore, that the greater the distance between the plates, the less the current for a given electro-motive force, or the greater the distance between the plates, the higher must be the electro-motive force to cause an unvarying current. As the plates are oxidised away, and the distance between them is augmented, the speed of the dynamos must be increased, and the engines run quicker, and more coal and labour consumed to ensure the current in quantum suff.

At Salford the plates were ½ in. thick, and the intervening spaces ½ in. initially. Finally the spaces would be 1½ ins., and the initial electro-motive force to the final as 5:9; and the initial to the final cost of current would follow the same ratio, except in so far as large quantities of power can be generated more cheaply than small, compared unit with unit.

A valid objection to this reasoning may be advanced—that a point would be found in practice at which it would be cheaper to renew the plates than to pass current through so great a space; but in that case the remaining plates should have been weighed in as a loss of iron, and cost calculated upon the average current through the whole life of the plate, not upon a run of 132½ hours.

I have mentioned this objection to the test before, and was met with the reply that in practice the electro-motive force required is constant. Well, if it is so, surely a fact of such importance, a fact which goes in the face of well-established theory throughout, and all other known practice, should be accounted for and regarded as an important discovery, and not allowed to rest upon *the ipse dixit* of any one.

**Precipitation.**—Concerning the comparative merits of continuous-flow or quiescent tanks, nothing here need be said more than that the choice depends upon local conditions. Where land is scarce, small quiescent tanks give as good results as large continuous-flow tanks, but require more labour, the extra cost of which has to be compared with the interest on capital sunk for extra land.

Continuous tanks could be used at a lower level, except that the sludge must be relieved of the supernatant liquor before emptying, which necessitates pumping. To obviate this difficulty two continuous-flow tanks have been lately brought out, in one of which, the "Ives," the sludge is pumped from a conical-shaped bottom while the tank is in operation.

The principle is a good one, which sooner or later will generally be adopted. The objections to it are:

1. That sludge immediately above the outlet *only* is pumped out, and is followed by the sewage, which soon makes the sludge thin.

2. The sludge remaining on the sides decomposes, so affecting the effluent. The results of the working of this tank at Tamworth are shown in analyses 9 and 10.

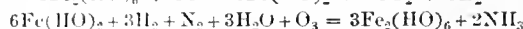
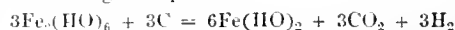
A great improvement on this is the Candy Tank, which has a flat bottom, and from which the sludge is siphoned through a revolving arm. The tank can be worked with a foot head to 10 times its capacity per 24 hours, and emptied while in use. A result of the working is shown in analyses Nos. 11 and 12.

Both tanks are fed at the bottom under a bonnet with a dip pipe.

#### Oxidation Processes.

1. **Precipitants.**—Much has been written and said concerning oxidation by precipitants—but very little done, and I fancy the advantages of ferric salts over ferrous have been very much over-rated. It is no doubt truly contended that ferrous salts are reducing, and ferric salts oxidising agents, but this much is certain, that no ferric salts have yet been put into use which will produce a satisfactory effluent alone, and that much ought to be generally known.

In considering the equations—



we must bear in mind that the ferric hydrate is only sparingly soluble in water. That which is suspended undergoes no such chemical change, as evidenced by the permanency of the bluish-green and red colours. Further, the samples numbered 14 to 23 show the oxidising effects of iron salts to be slight in any case, and that the value of these salts is in their power of precipitating suspended matter more than anything else. It occurs to me that oxidation is more easily brought through the agency of filters than precipitants, and more attention might be paid to these with good result. By this I do not mean oxidation in filters by means of the oxidising action of ferric hydrate in the filter, which hydrate is alternately in the ferric and ferrous state, as it is claimed to be in the case of the Kremmolite precipitant. The effect of such action is shown not to be very startling by the result No. 22. The filter I refer to more particularly is a filter where oxidation is brought about both by direct oxidation and nitrification.

The great amount of attention paid just now to oxidising precipitants suggests the idea of a dynamo tender running short of current devoting all his attention to the exact setting of brushes, oiling, &c., instead of running the engine faster, or decreasing the resistance to the current. *His attention is directed to the wrong point.*

2. **Direct Oxidation by Filters.**—The three specially prepared filtering materials which I had hoped to bring before you, and in fact the only three of whose existence I am aware, were polarite, porous carbon, and Bell's magnetone. I regret that I am not able to do this in the way I desired.

I wrote asking the porous carbon patentees the nearest point to Preston at which it could be seen in use, and was advised that such a point was Richmond. Upon writing to the engineer in charge of Richmond works, however, he informed me that only a portion of the filtering area contained porous carbon, that that was not in constant use, that certain days were set apart for visitors, and that I should not be allowed to take samples without previously making an appointment. It is needless for me to state therefore that samples were not drawn, although I applied for and was supplied with a sample of the porous carbon.

Bishop Auckland was the point to which I was directed for a sample of sewage treated by "Bell's Magnetone," and here also I was unable to get a sample of effluent owing to personal reasons. A sample of the Magnetone, although applied for on the 20th ult., is only just to hand. Concerning this process therefore I will say nothing further.

\* Reports of Salford trials.

† Geor., "Electro-chemistry," p. 9.

‡ Journal of the Chem. Society, Vol. X., p. 203.

The action of polarite and porous carbon I take to be one of direct oxidation, due to the exceedingly fine cellular construction of the materials. The microscopic pores contain oxygen, which acts in the same manner as occluded air in spongy platinum. The pores in polarite, however, as seen under the microscope, are much finer than in porous carbon. Moreover, a jet of hydrogen passed through the two substances raised the temperature of polarite much more readily and to a higher point than it did in the case of porous carbon.

Further, if equal quantities of solution of  $\text{SH}_2$  be passed through equal filtering areas of polarite and porous carbon, that passed through the former will give a much denser precipitate with  $\text{BaCl}_2$ .

Again, Padibam clear sewage containing 0.27 albuminoid ammonia, passed through polarite and porous carbon, gave 0.11 in the one case and 0.19 in the other respectively.

Other results of polarite treatment are shown in the analyses numbered 24 and 25.

### Natural Media.

By the term "natural media" I wish to be understood those media in which oxidation is not brought about by direct oxygen contact, but by the nitrifying organisms; and I must confess at once that this, to my mind, is the best method of sewage treatment where such media can be obtained. Of course the limitation is very necessary and very wide; but at the same time I hesitate not to say that where failures have resulted they have accrued in all cases either from mismanagement or unsuitable physical conditions.

At Lincoln, for instance, where it has failed, a commencement was made with 55 acres of land. This was not under-drained, and crude sewage put on without any attempt at precipitation. Soon after, it was found that the land had become choked, and a resort was made to subsidence tanks and draining. Then precipitation by lime, followed by lime and iron, the whole finally to be abandoned for polarite filters.

In the case of Preston, a tract of sandy land has been embanked from the Ribble estuary (600 acres in extent), portions of which are from time to time literally submerged with sewage. The land is not under-drained, but the sewage left to drain away wherever it may find outlets. Such outlets, however, are very scarce, for during a considerable portion of the day the farm embankment is flanked by sea-water.

The average level of the surface is 13 feet above ordnance datum, and ordinary spring tides rise to this height, the ground water rising under the farm of course with the tide. On the ebb tide the absorbed sea-water makes its way out first, then a mixture of sewage and sea-water, and finally the sewage. But at this point of time the flood tide commences and drives back the sewage to the top, and so on *ad infinitum*. On one occasion I observed about 2 or 3 feet depth of sewage on the farm, and on this, on a floating raft, workmen were engaged repairing the embankment.

The effluent, taken at low water, gave—

Chlorine .....	69.7 parts per 100,000
Dissolved solids .....	359.3 " "

How anyone could select such a site for a sewage farm is surely a mystery.

Other flagrant cases of inappropriate sites are Chorley, Blackrod, and Shepton Mallet (Somerset). Each of these is a farm forming a river bank. In the first case the farm has an average fall of 74 feet in its width of 1,056 feet, or about 1 in 14. Blackrod has an average fall of 130 feet in its width of 400 feet, or about 1 in 3, while Shepton Mallet, in spite of a favourably altered configuration, still has an average gradient of 1 in 6. It is needless for me to point out that the sewage in these three cases just takes a straight line for the river, which explains the figures shown in analyses 27, 28, and 29. In contradistinction to these I will instance the farms at Ormskirk and Wigan. The former is a good loamy soil, with a flat surface, is well cultivated, and not "doused" with sewage. There is sufficient acreage to the population, and the sewage is

treated in tanks as a preliminary step. At Wigan there is a farm of 270 acres now giving a good effluent. The 270 acres of flat loamy land was divided last year as follows:—

	Acre.
Mown hay.....	70
Corn.....	70
Potatoes.....	16
Turnips.....	10

About 6 acres filter beds and under 100 acres grazing.

### AMOUNTS RECEIVED FROM OTHER PLACES FOR TREATMENT OF THEIR SEWAGE.

	£
From Aspull.....	260
" Ince.....	271
" Orrell.....	195
Sewerage rate equal to 7d. in the £.	

### DETAILS FOR YEAR ended MARCH 1892.

	£	s.	d.
Sinking fund, sewers.....	101	8	9
" " farm.....	61	9	4
Interest, sewers.....	2,914	3	2
Assessment of sewer pipe.....	12	0	0
Rates.....	19	15	10
Wages. Screen chamber.....	24	4	6
" Sewerage account.....	165	10	4
Compensation.....	18	15	0
Loss on farm, year.....	1,235	15	8

These figures, together with an effluent giving off only 0.1 part per 100,000 albuminoid ammonia, speak exceedingly well for a sewage farm properly situated and well managed. But there is only one Wigan, unfortunately.

It is such figures as these that visiting deputations ought to ask for, and not figures concerning the maximum quantity of storm overflow allowed to be turned into rivers, injunctions, price obtained for sludge, number of w.c.'s, prime cost of works, &c., &c., as is frequently done.

*Artificially prepared Fermentative Oxidation Media.*—An impression has long been abroad among the local board "practical men" that coke is the proper thing through which to filter sewage.

Reason and figures are of no avail with a sewerage chairman who has set his mind on coke. It is an old-established institution, handed down from father to son, and will die hard, but die it must, for (as shown by the Nelson figures) it has no effect on dissolved organic matter at ordinary filter depths and rates of filtration. This coke, however, was pulled out and burnt during the recent coal famine, doing more good on that particular occasion than it ever did before. Its ghost, however, is now on exhibition at Baildon in the form of a "carbon" filter, *so called*.

Ashpit and privy refuse is here burnt (carbonised, it is claimed) and a filter made of the resulting ash, which is nothing but ash. This forms a sand filter, but that is all; and if of sufficient area would perhaps in time form a nitrification bed.

This is a process which at once appeals to the minds and pockets of local boards. No precipitants! no expensive filters! refuse disposed of! no pollution. The impossibility, however, of its application on a large scale must be apparent at once to any but an outsider. The sewage treated at Baildon by this process is initially very weak. Its effect upon that is a mere screening effect, as shown by the results numbered 34 to 37.

The result of a very recent system is shown in Nos. 38 and 39.

This system was the subject of two very plausible leaders in the "Engineer," which were copied pretty freely in other journals.

In fact the birth and fame of this process is the best example of that utter recklessness with which people launch into the question of sewage treatment, which I ventured to call your attention to in the hope that some definite data for the choice of a system may be suggested here. A person notices certain effects resulting from a filter through

which the sewage of *one house is passed*. These effects (not at all unusual in themselves) form the subject of leaders in a standard engineering journal, patents are taken out, and a 16-page pamphlet issued, offering a process of treatment without money and without price; which process, as seen at Toweester, is neither more nor less than a sand

and gravel filter, and not the best at that, as evidenced by the samples taken without notice.

It is just as easy for good samples to be taken by judicious preparation as it is for bad. For instance, here is a series of analyses to me as showing the effect of an "Ives" precipitation tank.

## WATER.

## COUNTY ANALYST'S LABORATORY, UNITY BUILDINGS, BIRMINGHAM.

## RESULTS OF ANALYSIS EXPRESSED IN PARTS PER 100,000.

Date of Receipt of Sample.	Description.	Total Solid Impurity.	Free Ammonia.	Organic Ammonia.	Nitrogen as Nitrates and Nitrites.	Total Combined Nitrogen.	Oxygen Absorbed in 4 Hours.
1893.							
September 5th	Samples sent by Mr. H. J. Claison, C.E., Tamworth.						
September 7th	Sewage .....	846.0	0.000	2.998	0	..	..
September 11th	Effluent from Glascoate .....	68.0	0.000	0.080	0.22	..	..

Date of Receipt of Sample.	Description.	Chlorine.	Suspended Matter.			Remarks.
			Organic.	Mineral.	Total.	
1893.						
September 5th	Samples sent by Mr. H. J. Claison, C.E., Tamworth.					
September 7th	Sewage .....	10.0	3.3.0	370.0	763.0	Very turbid. Foul odour.
September 11th	Effluent from Glascoate .....	6.1	..	..	0	Fairly clear. No suspended matter. No odour. Neutral in reaction.

These two analyses were shown to me, as they had doubtlessly been to many others, as illustrating the effect of an "Ives" precipitation tank. When, however, I pointed out that the nitrates had gone up and the chlorine gone down, which was a very unusual effect of a precipitation tank, it was courteously explained that one sample was drawn four days after the other.

The method and point of time of sampling sewages appears to me to be one of the many other things omitted from an analysis which should there find a place.

For the purpose of forming the framework of a discussion I suggest here, in conclusion, that the following rules be laid down for the efficient treatment of sewage:—

1. Suspended solids to be removed by precipitation in some form.
2. That the organic matter in solution be oxidised (directly or by means of natural nitrification).
3. That no effluent entering a natural stream or canal should give off more than 0.1 part per 100,000 albuminoid ammonia.
4. That the precipitated sludge be pressed and burnt.

## TABLE OF COMMONER SYSTEMS.

			Point of Sampling.	
I.—Sterilisation systems....	{ Amines.....		Wolverhampton.	
	{ Electrical (duplex).....		Salford.	
	{ Bacillite.....		Kettering.	
	{ Heranite.....		Worthing.	
	II.—Oxidation systems....	{ Direct oxidation.....	{ Precipitants.....	{ Ferric chloride.....
{ Ozoline.....				Horwich and Laboratory.
{ Clarine.....				Darwen and Laboratory.
{ Kremnolite.....				Salford and Laboratory.
{ Levinstein's patent persalt.....				Laboratory.
{ Filters.....		{ Polarite.....	Huddersfield.	
		{ Porous carbon.....	Laboratory.	
		{ Magnetron (Bell's).....	.. ..	
{ Fermentative oxidation....		{ Natural media.....	{ Ridged and furrowed land.....	Lincoln.
			{ Grass land (ploughed occasionally) .	Preston, Blackrod, Chorley, Wigan, Shepton Mallet, Ormskirk.
		{ Artificially prepared media.....	{ Coke.....	Nelson.
			{ Carbon.....	Baldon.
			{ Cultivation bed, sand and gravel (Scott Moncrieff).	Towcester.

TABLE OF RESULTS.

Description of Sample.	Taken by	Date.	Examined by	
1. Wolverhampton raw sewage (clear) before treatment by Amies process.	Author	24th April 1890	Author	0.327 part per 100,000 albuminoid ammonia.
2. Same after treatment by Amies process.	"	"	"	0.320 " "
3. Same after passing over irrigation area.	"	"	"	0.109 " "
4. Effluent from electrolytic treatment at Salford.	"	"	G. E. Davis	0.18 " "
5. Effluent from Bacilline process at Kettering.	Author	27th January 1894	Author	0.569 " "
6. Effluent from Hermite process at Worthing.	Dr. Angell	"	Dr. Angell	1.3895 " "
7. Raw sewage before passing through "Ives" precipitation tank, Glasgote.	Author	26th January 1894	Author	173.0 parts per 100,000 suspended matter.
8. Ditto after passing through tank (precipitant being used in blocks).	"	"	"	3.0 " "
9. Raw sewage before passing through "Ives" tank at Tamworth.	"	"	"	25.0 " "
10. Raw sewage after passing through same tank (no precipitant).	"	"	"	16.0 " "
11. Raw sewage before passing through Candy's tank (Salford).	"	23rd January 1894	"	39.3 " "
12. Raw sewage after passing through Candy's tank (precipitant used).	"	7th March 1894	"	0.9 " "
13. Padstham clear sewage.	"	12th March 1894	"	0.27 part per 100,000 albuminoid ammonia.
14. Ditto after treatment with ferrous sulphate.	"	"	"	0.20 " "
15. " " ozonine	"	"	"	0.19 " "
16. " " clarine	"	"	"	0.20 " "
17. " " kremnolite	"	"	"	0.16 " "
18. " " Levinstein's patent	"	"	"	0.18 " "
19. Horwich effluent (ozonine).	"	18th May 1893	Dr. A. B. Hall	0.472 " "
20. Darwen effluent (clarine)	"	16th October 1892	Author	0.053 (from 0.066 raw sewage) part per 100,000 albuminoid ammonia.
21. Salford effluent (kremnolite)	"	21st March 1894	"	0.567 part per 100,000 albuminoid ammonia.
22. Salford effluent (kremnolite), filtered through sand in laboratory.	"	"	"	0.482 " "
23. Salford effluent (Hamor Lockwood)	"	7th March 1894	"	0.31 " "
24. Huddersfield tank effluent (taken from filter top).	"	24th February 1894	"	0.54 " "
25. Huddersfield filter effluent.	"	"	"	0.08 " "
26. Lincoln effluent.	"	17th February 1894	"	0.8 " "
27. Chorley effluent.	"	17th October 1893	"	0.48 " "
28. Blackro l	"	13th September 1893	"	1.2 " "
29. Shepton Mallet	"	5th January 1894	"	0.31 " "
30. Ormskirk	"	"	"	" " "
31. Wigan	"	2nd January 1894	"	0.10 " "
32. Nelson effluent before coke beds.	"	8th August 1893	"	2.0 " "
33. " after "	"	"	"	2.0 " "
34. Baildon raw sewage	"	17th January 1894	"	0.345 " "
35. " tank effluent	"	"	"	0.239 " "
36. " filter "	"	"	"	0.150 " "
37. " clear sewage	"	"	"	0.139 " "
38. Towcester raw sewage	"	19th February 1894	"	0.42 " "
39. " effluent	"	"	"	0.31 " "

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SESSION 1893-94.

May 7th:—

Mr. H. R. Procter:

"The Qualitative Recognition of Tanning Materials."

"Note on Recent Modifications in the Estimation of Tannins."

Mr. A. G. Perkin: "On a Rapid Method of Testing the Shade of Alizarin Paste."

Messrs. J. J. Hummel and H. R. Procter: "Note on an Improvement in Dyeing Leather with Basic Colours."

Meeting held at the Queen's Hotel, Leeds, on Monday, April 2nd, 1894.

PROFESSOR SMITHELLS IN THE CHAIR.

## THE TINCTORIAL PROPERTIES OF SOME INDIAN DYESTUFFS.

BY J. J. HUMMEL AND A. G. PERKIN.

### PART I.

IN these days, when the artificial colours derived from coal-tar are becoming each year more and more numerous and important, and are gradually pressing into the background the natural dyestuffs, we almost feel it necessary to give some reason why we should have selected not merely natural dyestuffs for detailed scientific and practical examination, but such as are not usually, if at all, met with in the European market. Our explanation is simply this:—It may be known to some of you that during the past few years, owing to the munificence of the Clothworkers' Company of London, a Research Laboratory has been established in connection with the Dyeing Department of the Yorkshire College, and as part of our work, we have set ourselves the task of aiding in carrying out one portion of the programme of the Imperial Institute of London, by taking up the examination of Indian dyestuffs. Many of these dyestuffs are highly spoken of, as yielding colours which possess in a marked degree the qualities of fastness to light, excellence in tone, &c. This being so it is surely desirable to test such statements by actual experiment, and if there are natural colouring matters with distinct and useful properties, it were a pity if we deliberately neglected them.

India, so rich in dyestuffs, and the birthplace of the dyeing industry, has in the past furnished us with our fastest dyestuffs, indigo, and alizarin, also our most important and interesting processes of vat and Turkey-red dyeing, and may we not still have something useful to learn from her vast resources in this domain?

Further, the starting point of the manufacture of artificial alizarin, one of the most notable discoveries of the century connected with colouring matters, was surely the Indian madder. In like manner it seems to us that by patiently investigating the colouring principles present in various natural products, especially those of India, one may meet with important new compounds, and by determining their constitution, perchance, lay the foundations of some industry like the one referred to, to be built upon by others who take up the subject where we relinquish it.

Already, among the few dyes examined by us, colouring matters, apparently new to science, have been isolated, one of which seems to possess such tinctorial properties as may possibly render the dyestuff, even in its natural condition, of commercial value.

### Chayroot or Indian Madder.

This dyestuff is the root of *Oldenlandia umbellata*, Linn., Rubiaceae. It bears a number of vernacular names, among which may be mentioned: *surbuli* (Bengali); *cherivelu*, *chiri-veru* (Telugu); *saya*, *saya-wer*, *imburai* (Tamil); &c. The name *chay-root* or *chayaver* is said to be derived from *chaya* (which fixes colours), and *ver* (root).

It is a low, spreading, bushy, biennial plant commonly met with in sandy soils in North Burma, Bengal, the Madras Presidency from Orissa southwards to Ceylon, on the Malabar coast, and indeed very generally in Southern India.

Although occurring abundantly in some parts of Bengal it is not there used as a dye, but on the Coromandel coast it is extensively cultivated in many parts, especially at Nellore and Masulipatam. The chief market is Madras, where it is sold in small bundles, the retail price being about  $\frac{1}{4}$ d. per lb. The cultivated roots are very thin, and often a yard or more in length, while those of the wild plant are shorter and thicker, being usually 10 to 12 ins. long and  $\frac{1}{2}$  in. thick, somewhat straight and stiff, tough and wiry, and with few or no lateral fibres. Contrary to our general experience in the case of madder, the wild variety is more highly esteemed, and is said to yield a fourth more colouring matter. Roots of two years' growth are preferred, but the farmer finds it more profitable to gather them at the end of the first year, when they are merely dried in the sun for a few days and tied into large bundles. When freshly gathered the roots have an orange-yellow colour, but on drying they assume a yellowish-grey hue, becoming even brownish during long storage.

The colouring principles seem to be situated almost entirely in the bark of the root. The ground root has quite an ashen-grey appearance, but on treatment with hot or even cold water a yellow extract is obtained, and with the addition of alkali the solution is of a deep red colour.

**History.**—In India chay-root has occupied somewhat the same position as a dyestuff, as the madder did many years ago in Europe. It seems to have been very commonly employed by the Hindus on the Coromandel and Malabar coasts from time immemorial, for the production of very permanent reds, purples, and blacks, on calico prepared with oil and aluminium, or iron mordants, a process analogous to, and no doubt the forerunner of Turkey-red dyeing as pursued in Europe. From the fact that the alum mordant, applied by the native calico-printers, was frequently coloured red with sapan wood, the Hindus themselves, and also some early French writers who described their processes, concluded that the chay root was merely used by the natives as a fixing or brightening agent for other colours, rather than as a dye itself. Others again have referred to it as a yellow-colouring matter, or as an astringent substance devoid of any red colour. In this manner one sought to explain why it was found impossible to make any use of a large quantity of chay-root, imported by the French East India Company in 1774. (M. Le Goux de Flaix, "Annales des Arts et Manufactures," No. 51).

Bancroft in his "Philosophy of Permanent Colours," vol. II., p. 298, states that a large quantity of chay-root was brought to England about 1803 by the East India Company. His experiments with this consignment were not very successful, and several dyers and calico-printers, to whom samples were sent, did not succeed with it, and were not disposed to adopt it. This want of success was ascribed by Bancroft to the roots having been of inferior quality or having suffered injury by age or damp. Ultimately, however, he satisfied himself by further experiments on selected samples that chay-root, in conjunction with alumina mordant, was capable of yielding on wool, linen, and cotton a red colour very similar to, both in appearance and permanency, but never exceeding, that given by madder. Calicoes printed with mordants and dyed with it had the whites less stained than with madder. Wool mordanted with tin yielded a very bright and lasting red inclined to orange. The most remarkable difference between madder and chay-root in their dyeing properties, according to Bancroft, was that with the latter and iron mordant nothing darker than drab colours could be produced either upon wool or cotton. Wool boiled with a solution of zinc chloride and dyed with chay-root acquired a bright apple-green colour.

On calico prepared with oil and tannin matter and subsequently mordanted with alumina according to the ordinary Turkey-red process, Bancroft obtained colours very much like those given by madder. He discusses at length the

statements of various writers who, in describing the native processes make no mention of an alum mordant as being used or even thought necessary, but he conclusively demonstrates that the Hindus must have used it in some form or other, and suggests that its employment had escaped the attention of those who observed the native processes, either through ignorance of its importance or because the native dyers guarded it as a secret, or perchance it was present in the water employed.

Comparing European Turkey-red with that of Malabar, he considered that the former was quite equal to the latter in beauty and permanency, indeed the Indian red was usually not very bright, because soiled by certain brownish impurities, not removed by any clearing process as is usual in Europe, and hence it constantly improved in this respect by wearing and washing.

Gouffroy in his work entitled "L'art de la teinture des laines," speaks favourably of chay-root as a dyestuff and states that in 1834 a single firm in Elbeuf used 3,000 kilos. for wool dyeing, which shows that some firms at least appreciated its good qualities.

In 1832 M. Ed. Schwartz read a paper before the Industrial Society of Mulhouse giving the results of his experiments in conjunction with M. D. Keschlin on the dyeing properties of chay-root and a few other dyestuffs brought from India by Gouffroy. They refer to the fact that during a period of 15 years fruitless attempts had been made to introduce this dyestuff to Europe, both by the British East India Company and by various English and French merchants, and although Bancroft and other chemists who had examined the dyestuff had failed to obtain any very good results with it, they deemed it worthy of further experiment, especially in view of the extended knowledge then recently gained concerning madder and its colouring principles.

They found chay-root to have an acid reaction, and although this was less than in the case of "nona," *i.e.*, morinda root, they still found it desirable to wash the root with water before dyeing. They found it beneficial also to add ground chalk to the dye-bath to the extent of one-twelfth of the weight of the root. Turkey-red dyed with it stood the clearing process perfectly, and the colours produced on unmoiled calico were quite as brilliant and as fast to soap as those given by Avignon madder, to which dyestuff indeed it seemed to be very similar in its general dyeing properties. The only drawback to its adoption in Europe, according to these authors, was that it only contained one-fourth of the colouring matter present in a medium quality of madder. Nevertheless they considered chay-root to be more worthy of attention than other allied Indian dyestuffs because of its comparative freedom from a yellow acid principle, which being present in considerable quantity in the others would materially hinder their employment in Europe. They expressed the belief that by a more intelligent culture of these plants, and by leaving the chay-root for a longer period in the ground before gathering, the latter would no doubt become richer in colouring matter, and the others would probably have their properties beneficially modified. (Bulletin de la Société Industrielle de Mulhouse, V., pp. 302-306.)

Such is a brief résumé of all the published information concerning chay-root which we have been able to find. No examination of the chemical principles contained in the root seems to have been made by any chemist except M. P. Schützenberger, who in his *Traité des Matières colorantes* 2, 291 (1867), states briefly that he found it to contain alizarin and chlorogenin, and that it could readily be exhausted by a treatment with alcohol.

**Experimental Part.**—The results of our chemical examination of chay-root have already been published in the Journal of the Chemical Society (1893, p. 1160); we need therefore here merely remind you that the root contains chiefly ruberythric acid and but a very small amount of ready formed alizarin. The most interesting feature is that purpurin is entirely absent, and that there are present in small quantity several yellow crystalline substances not possessing the property of dyeing mordants, including two mono-methyl-ethers of alizarin, two dimethyl-ethers of anthragallol and

metahydroxy anthraquinone; other substances isolated were wax and rubicloric acid.

Although chay-root certainly contains acid principles which tend to dissolve off the mordants, we found no difficulty in dyeing with chay-root without submitting it to a preliminary washing process as recommended by Schwartz and Kœchlin. The only precautions necessary to be observed was to add 2 per cent. chalk to the dye bath, and to raise the temperature to the boiling point gradually, say in the course of  $1\frac{1}{2}$  hours, and to boil for  $\frac{1}{2}$ —1 hour. It presented therefore no features different to those met with in madder.

Comparative dyeing experiments on ordinary stripe-printed calico containing alumina and iron mordants, showed the dyeing power of chay-root to be equivalent to the presence of a percentage of 0.33—0.35 alizarin. Compared with ground madder-root of good quality it seemed to have about half its dyeing power when the comparison was made before soaping, but examined after soaping it was quite equal to madder. The reds, pinks, and chocolates had a distinctly bluer shade than those given by madder, and the lilacs were much fuller and brighter and very similar to those obtained from alizarin. This last feature alone ought to have secured a ready market for chay-root among the European dyers, previous to the introduction of artificial alizarin, and it seems to us somewhat strange that its marked suitability for lilacs should have escaped the observation of those who formerly made dyeing experiments with this root.

On oil-prepared calico mordanted with alumina, chay-root gave an excellent blue shade of Turkey-red, withstanding the operation of clearing with soap and stannous chloride better even than a madder dyed red, and quite equal to one obtained by means of artificial alizarin. Good brown, red, orange, and purple colours were also readily obtainable on wool and also on silk suitably mordanted with chromium, aluminium, tin, and iron, according to the ordinary methods usual with dyers. On wool, the colours not being submitted to any soaping operation, chay root seemed to possess about half the dyeing power of madder.

All the dyeing properties of chay-root are entirely consistent with the results of the chemical examination referred to, namely, the absence of purpurin or any other colouring matter except alizarin capable of dyeing mordanted cotton.

Boiled with dilute sulphuric acid chay-root yielded a garanceine of a very dark green colour and possessing about three times the dyeing power of the original root.

For our several supplies of chay-root we have to thank Dr. George Watt, Simla, Mr. W. Reid, Bombay, the late Mr. R. O. Campbell, London, and Messrs. Binny & Co., Madras.

#### *Rubia Sikkimensis.*

According to Dr. G. Watt, from whom the small sample examined was obtained in 1886, the roots of this plant furnish the brilliant red dye used by the hill tribes of the Naga Hills and Manipur, under the vernacular name of *mujam* (Manipur).

It is a stout handsome creeper found on the Eastern Himalaya, in Sikkim and Bhutan, in the Mishmi Mountains of Upper Assam, and in Manipur, and the Naga Hills.

*History.*—In the Calcutta Exhibition catalogue (1883), II., p. 55, Dr. Watt writes: "Apparently the Lepehas of Sikkim do not know that this plant yields the madder dye; but I suspect that the thick heavy roots which are sold in the bazaars, belong largely to this species, though probably used as an adulterant." Prior to 1874, the plant does not appear to have been named or even known to exist by botanists, it having escaped their attention or been mistaken for *R. cordifolia*.

Whatever similarity, however, there may be in the general similarity of the plants, the dried roots of the two species show a marked difference; those of *R. sikkimensis* are much thicker, rough and furrowed, and covered with a grey powdery pith-like substance, while those of *R. cordifolia* are round, smooth, and straight.

The method of dyeing cotton yarn red, with the roots of *R. Sikkimensis* as witnessed by Dr. Watt in Manipur was probably incomplete, in so far as no use of alum mordant

is mentioned, but enough is described to indicate that the cotton is probably prepared with oil and tannin matter, mordanted with alum and finally boiled with a decoction of the root, i.e., similar to the Turkey-red process.

The chemistry of ordinary *munjeet* or *munjit* (*R. cordifolia*) was studied by Stenhouse (Annalen, 130, 325; also Proc. Roy. Soc. 12, 633 and 13, 145), who showed that it contained essentially purpurin, along with an orange crystalline substance called munjistin, now known to be purpuroxanthin-carboxylic acid, as proved by Schunck and Reemer (Ber. 10, 172, 790).

*Experimental Part.*—A chemical examination of the *R. sikkimensis* root (Journ. Chem. Soc. 1893, 1157), showed that it contained essentially the same principles as munjeet, hence it may be considered that both owe their dyeing properties to the presence of purpurin, since munjistin does not dye mordants properly, and may therefore be regarded as the accompanying yellow substance peculiar to these roots, and which corresponds to those found in madder root.

The application of *R. sikkimensis* root in dyeing presents no difficulty whatever.

Calico printed with iron and alumina mordants may be dyed without any addition of calcium carbonate or acetate to the bath, since there is evidently a sufficiency naturally present in the root. Owing to the solubility of the colouring matter, the mordants quickly attract it even at a comparatively low temperature. Generally speaking the colours with the different mordants are similar to those obtained from madder, but the reds and chocolates are much bluer, being devoid of yellow, and the lilacs are decidedly greyer. The colours are, however, precisely similar to those obtained from *R. cordifolia*, the latter possessing merely a half more dyeing power. They do not bear the action of boiling soap solutions so well as the madder colours.

On oil-prepared calico mordanted with alumina a very bluish shade of red is given, much brightened by soaping but rendered very poor and dull if cleared with soap and stannous chloride. Chromium mordant on oil-prepared calico, gives also a much bluer toned chocolate colour than that given by madder, and with iron mordant a similar difference in the lilacs is apparent. On wool and silk mordanted with chromium, aluminium, tin, copper, and iron in the ordinary manner, colours similar to those obtained from madder are produced, the most marked difference being exhibited by the tin mordant with which *R. sikkimensis* gives a much brighter and yellower orange; in this respect however it does not differ from *R. cordifolia*.

Comparing the colours on ordinary stripe mordanted calico given by pure purpurin and by *R. sikkimensis*, a very marked difference is noticeable; the former gives very yellowish red and chocolate, full pinks and purplish lilacs, while the latter yields very bluish red and chocolates, bare pinks, and greyish lilacs. The poorness of the pinks points to the removal of mordant by some acid principle in the *R. sikkimensis*, but the striking difference in the tone of the reds we are at present unable to account for. Approximately we should say that the dyeing power of *R. sikkimensis* is equivalent to its containing 0.37—0.5 per cent. purpurin.

It may be added that a small sample of *R. khosia* also obtained from Dr. G. Watt during the Indian and Colonial Exhibition of 1886, yielded colours precisely similar to those given by *R. cordifolia* and *R. sikkimensis*, but it possessed a somewhat greater colouring power than either. No doubt, when we have an opportunity of examining it chemically, it will be found to contain the same colouring principles.

#### *Al, Ach, Saranji, or Morinda-root.*

This dyestuff is the root of *Morinda Citrifolia* (*Rubiacæ*), the so-called Indian mulberry, and the Togari wood of Madras. The genus was considered to be so nearly related to that of *Morus*, one species of which furnishes the yellow dyestuff Old Fustic, that its name was thereby suggested to Vaillant and composed from the words *Morus indica*. Numerous species are recognised, of which seven are indigenous to India, and some uncertainty prevails as to which of the species are employed for dyeing by the natives.



Some consider *Morinda tinctoria*, which is the chief dye-yielding species, to be but a wild form of *M. citrifolia*. The latter is a small tree or bush cultivated extensively in many parts of India, also in Burma and Ceylon.

The following are a few of the vernacular names by which it is known:—

Āl, aeh, &c. (Hindi); aeh, aieh, aebu (Bengali); surangi, hārtundi, &c. (Mar.); munja-pavattay, &c. (Tamil); nōna, maddi, &c. (Telugu). A common trade name is surangi or seringi.

No doubt the dye-stuffs Nona, Noona, Hachroul, and Atchroot mentioned by Gouffreville, Schwartz and Koechlin, and referred to in the works of Crookes, Calvert, and others, consist of the roots of this or some closely allied species. The plants are allowed to mature usually for about three years, although in some districts the time may be extended to four, five, or even ten years. If allowed to grow, the tree ultimately attains the height of 20 feet or even more, but after the first three or four years, when it is 2-8 feet high, the roots no longer yield the dye in any quantity. The mature roots which vary from three to four feet in length, and from  $\frac{1}{2}$  to 1 inch in thickness, are carefully dug up, dried in the sun, cut into short lengths and stored for sale. They have a yellowish-grey earthy appearance. A transverse section shows a yellow woody central portion of considerable toughness. The bark has a darker colour. Cold or hot water extracts merely yellow principles, but alkalis at once yield deep red coloured solutions.

Three qualities of the dyestuff, bearing different names, are distinguished, according as they are derived from the thick end, the middle part, or the thin end of the root. The thin roots are considered to be the best quality, the thicker ones being much inferior, and sometimes indeed almost useless. This difference is no doubt largely due to the fact that the colouring matter resides chiefly in the root-bark, of which there is necessarily a larger proportion in the thin roots.

The average commercial value varies from three seers of the thin, to ten seers of the thick quality per rupee (say  $1\frac{1}{2}$  d. to 4d. per pound).

In some parts of Bengal the bark is stripped from the stem of the plants, and the twigs also are used for dyeing, although they are much inferior to the roots; in Maimansingh even the juice of the leaves is used, but it only imparts to cotton a reddish-brown colour.

Frequently, as in Bengal, the cultivation is limited to meet local demands only, or it is grown, as in Orissa and in Burma, in the native gardens for home use. In the Nimar district in the central provinces, the cultivation is wholly in the hands of a caste called Aloes.

Sometimes it is allowed to grow as a weed in the weavers' homesteads to be used by them subsequently in their capacity also as dyers, or they depend entirely upon the wild plant found in sufficient quantity in the jungle.

Here and there however the roots collected in one part are largely exported to another part of India, as for example from the Central Provinces to Madras, from Malabar to Guzerat and Northern India, from Bakarganj in Bengal to the North-West Provinces and the Punjab, &c.

There is no doubt *Morinda*-root takes a foremost place among the fast red dyestuffs of India, and the Āl dye is said to be gradually supplanting the more expensive red obtained from chay-root, although it too is suffering from the introduction of European dyes in some districts.

Occasionally Āl root has been sent to Europe, but it seems never to have met with success in competition with madder. The Hindocs have probably used *Morinda*-root as a dyestuff from a remote period, and though there are no records of the early methods of dyeing employed, they were probably much the same as those now in use.

In Dr. G. Watt's Dictionary of the Economic Products of India, Dr. J. Murray has written, under the head of *Morinda*, a most complete account of the more important species, and full details of the native methods of dyeing are there given. Dr. McCann there reports that it is principally used for dyeing the thread or yarn out of which the coloured borders of the cotton garments worn by the lower classes are woven, and also for dyeing silk thread to

form the borders of the coarse silk fabrics known as *crench* or *enb* cloth. In many places it is used to dye pieces of a coarse cotton cloth known as *kunda* or *kharna*, or for thread to be afterwards woven into such cloth.

The colours given by Āl vary from a reddish yellow, through pink and various shades of red, up to dark brownish-red. The dye contained in the root bark seems to be the best red, whereas that contained in the woody part of the roots is more yellow than red.

The methods of dyeing vary very considerably in detail in different parts of India, but they are all similar in general principle, which may be briefly expressed by saying that they are all more or less crude processes of Turkey red dyeing as known in Europe.

The following process of preparing the cloth is said to be followed in Dinajpur:—"Cloth first cleaned and dried; then mix the ashes of plantain bark and leaves with a quantity of water sufficient to wet the cloth to be dyed; to this add castor-oil seed fried and pounded, mix well, and afterwards mix with this decoction mustard oil."

"Steep the cloth in this for one night; wash in the morning in this same decoction and dry. Repeat this process of steeping and washing the cloth in the same water, and drying, every day for fifteen days. Then wash the cloth in fresh water. On drying it is ready to receive the dye."

"The dyeing solution is prepared by cutting the roots or sometimes only the bark of the roots into small pieces, pounding them well or grinding them, and steeping or boiling in water. Generally the material to be dyed, prepared as above, is boiled with the roots in water, but sometimes after the roots have been treated as above, they are taken out and thrown away, and the cloth or thread is simply boiled in the water containing the dye extracted by the above process from the roots."

It is added that a great variety of substances are used as mordants or auxiliaries, among which one finds mentioned myrobalsam and alum. They are applied to the cloth after the cleaning (oiling) process, before the application of the dye, or in some cases either along with the oil, &c., or with the dyestuff; sometimes indeed the whole of the necessary ingredients are mixed together, and simultaneously applied to the cloth by a steeping or boiling process.

*History.*—About the year 1790 some of the powdered root under the name of *urtech* came into the hands of Dr. Bancroft, and he apparently found little or no difficulty in applying it both to wool and to cotton. On the former with alumina mordant he obtained a bright orange-red colour fast to light, and the action of the weather, and with a tin mordant, it yielded a bright and durable orange. On calico printed with alumina and iron mordants, separately or mixed, he obtained reds, purples, and chocolates, very similar to the analogous madder colours, and equally durable.

At that time he considered that it might be profitably imported into Europe, and that it possessed the advantage over chay-root, that it was less liable to deterioration during storage.

In 1832 Schwartz and Koechlin also examined this root, under the names Nona and Hachroul, and reported on its dyeing properties to the Industrial Society of Mulhouse, in the paper already alluded to under chay-root. They pointed out its extreme toughness as compared with madder and hence the greater difficulty experienced in grinding it. They referred also to the fact that of all the Indian rubiaceae examined by them, it contained the largest quantity of certain yellow principles of an acid character which not only necessitated the washing of the root with cold water before dyeing, but also made it requisite to add a certain proportion of carbonate of soda to the dye bath in order to have a perfectly neutral bath, and to the neglect of this precaution they ascribed the indifferent results obtained by others. They recommended that the root be dried at a low temperature, then ground as finely as possible, and washed with fifty times its weight of pure cold water, filtering the mixture, and pouring fresh water over the root collected upon the filter.

To the dye-bath they added one-fifth of the weight of the root of carbonate of soda, and having introduced the mordanted material, the temperature was raised gradually

to the boiling point. In this manner they were able to obtain on oil and alumina mordanted calico a very full red, which, on clearing in the usual manner with soap and stannous chloride, changed to a scarlet, resembling the Turkey-red obtained from madder. On ordinary calico printed with iron and alumina mordants, they obtained black, lilac, red, and chocolate, differing only from the analogous madder colours by having a much yellower tinge. All the colours stood soaping very well, still preserving their yellow tinge, the lilac alone being deficient in brilliancy.

Their conclusion was that since morinda-root only possessed one-third the dyeing power of a medium quality of madder, it could never compete with the latter in the European market.

At a later date, about the year 1848, some morinda-root was imported into Glasgow under the name of Soorancee with the intention of introducing it as a substitute for madder. It was submitted for trial to some of the most experienced and skilful calico-printers of the district, all of whom concurred in declaring it not to be a dye at all, and to be totally destitute of useful applications.

Professor Anderson of the Glasgow University hearing of this circumstance obtained a supply of the root, submitted it to a chemical examination, and succeeded in isolating from it a yellow crystalline product. Anderson found that this substance did not dye iron and alumina mordants printed and fixed on calico as usual with calico-printers, but cotton prepared with oil and mordanted with alumina as for Turkey-red assumed a dark brownish-red colour, devoid of beauty. The absence of dyeing properties in this *morindine* is readily explained, for we now know it to be a glucoside.

When submitted to dry distillation Anderson found morindine to yield an orange crystalline sublimate which he called *morindone*, and this same substance was also produced by boiling morindine with dilute mineral acids.

*Morindone* represents the true colouring matter of morinda-root, and accordingly Anderson found it to dye ordinary mordanted calico in the normal manner.

In 1852 Roehleider gave it as his opinion that morindine and morindone were identical with the ruberythric acid and alizarin derived from madder. That this was not the case was first adduced by Stein (J. Pr. Chem. 97, 234), and in 1887-88 Thorpe, in conjunction with Greenall and Smith (Jour. Chem. Soc. 52, 52 and 53, 171), showed conclusively the correctness of Stein's view, and further that the constitution of morindine corresponded to a *tri-hydroxy-methyl-anthraquinone*, alizarine being, as is well known, a *di-hydroxy-anthraquinone*.

The most recent experiments in dyeing with morinda-root made in this country are those of T. Wardle, a record of whose results are contained in his Report on the Dyes and Tans of India 1878-1887. No notes are given of the mordants and processes employed, and one cannot, therefore, follow his experiments, but he appears to have been unable to produce the Hindoo reds, and at most succeeded in producing from it yellow, orange, or brownish-red colours. In his concluding remarks he writes, "I was not successful in obtaining good red dyes from the samples labelled *morinda citrifolia* and *morinda tinctoria*. In each case I found a yellow colouring matter prevailed, and I was led to infer that good reds were not obtainable from these plants or their roots without admixture of the other red dyes such as mangit, &c.; but on visiting India I found undeniable evidence in Calcutta, the North-West Provinces, and Rajputana, and other parts of India of the constant employment of both these species yielding excellent reds on cotton, both in prints and dyes. The samples which had been sent to me I found were of very poor quality, and were too small for successful trials."

**Experimental Results.**—Having regard therefore to the extensive employment of morinda root as a red dyestuff by the natives of India, and the undoubted difficulties experienced by many European experts who have in the past attempted to apply it for the production of reds, our attention and interest was attracted to this dyestuff with the view of determining the best method of applying it successfully.

Further, although morinda-root has already been examined by several chemists and the nature of its chief colouring principle has been established, it seemed desirable to determine the character of those accompanying principles analogous to the yellow substances found in madder, chay-root, and munjeet, which from its botanical relationship it might naturally be expected to contain.

With respect to this part of our examination the results will be communicated at an early date to the Chemical Society, but it may already here be stated that several of the above mentioned yellow substances have been isolated in the crystalline condition. As to the essential colouring principle it is found to be present almost entirely in the form of the glucoside morindine which in the pure condition proves to be of a somewhat stable character.

Now it is well known that the colouring matter of the madder-root is there present also largely as the glucoside ruberythric acid, a substance devoid of dyeing properties with respect to mordants, although it will dye wool and silk a bright yellow colour. Moreover, it is a matter of common knowledge, thanks to the researches of Schunck, that the madder glucoside is split up under the influence of acids, alkalis, and ferments into glucose and the useful colouring matter alizarin.

In attempting, therefore, to solve the mystery connected with the successful application of morinda-root in dyeing, our first endeavours were directed to the splitting up of the glucoside of the root by one or other of the methods indicated as effectual in the case of madder.

Fermentation experiments were made with several weighed quantities of ground morinda-root. In one case, presuming some ferment might be naturally present in the root, 60 grms. of root were merely mixed with 700 cc. distilled water and left to stand for five days at a temperature of 35° C. A similar experiment was made with the addition of chalk sufficient to neutralise the natural acidity of the root. Other experiments similar to these were carried on simultaneously with the addition of a small percentage of ground madder-root, on account of its ferment; yeast was also tried. In all cases fermentation took place, and the mixtures were thrown on calico filters, drained and washed rapidly with four litres distilled water. The fermented roots were dried at a low temperature, and the loss of weight (14-17 per cent.) determined.

Eventually pieces of calico printed in stripes with alumina and iron mordants were dyed with equivalent amounts of the various fermented root and the root in its original condition. The latter scarcely dyed at all, whereas the fermented roots gave fairly good reds, chocolates, and lilacs similar to the analogous madder colours. The root fermented with yeast gave the best result. In all cases the addition of chalk to the fermenting mixture was injurious.

It was evident, therefore, that a considerable improvement in the dyeing power of the root had been effected, either by the fermentation or by the washing, but since better results were obtained by other methods carried on at the same time, the fermentation experiments were not continued.

To effect the decomposition of the glucoside by means of alkalis, barium hydrate was selected, for in the course of the purely chemical examination of the root it was found that not only could the glucoside morindine be split up by this agent, but the morindine produced, formed an insoluble lime compound, whereas the accompanying yellow substances formed soluble compounds. By boiling morinda-root, therefore, with lime-water and then washing, the whole of the yellow substances could be removed, and by a subsequent treatment of the residual root with acid and washing the calcium compound of the morindine could be decomposed and the lime removed, leaving behind a purified root containing only morindine in the free state.

For some reason or other the morinda root treated in the above-mentioned manner yielded only indifferent results in dyeing, and having regard to the number of operations required and the better results otherwise obtained in a simpler manner, the matter was not further inquired into.

Decomposition of the glucoside by acids resolved itself into the preparation of a garancine from morinda-root, and this was done not only with the root in its natural condition, but after it had been thrice washed for two hours with



madder, had the same dyeing power as  $1\frac{1}{2}$  lbs. of Nos. 5, 2, 4, and as 3 lbs. of Nos. 6 and 9. No. 8 was very poor indeed, and seemed to have been spoiled. All the above roots in their washed and dried condition had a more or less bright olive-yellow colour, except No. 8, which was decidedly brown.

The results of the foregoing experiments entirely corroborate those obtained by Schwartz and Koechlin, as to the necessity of washing the finely ground roots with water before dyeing, and the addition of an alkaline salt, chalk, or carbonate of soda, to the dye-bath in definite proportion, if good full rich reds, &c., are to be obtained. We never found it necessary, however, indeed it would have been injurious, to add to the dye-bath such a large proportion of sodium carbonate as 20 per cent. of the weight of the root. This difference is probably owing to the different method of washing adopted. Schwartz and Koechlin seem to have washed the root with a much larger quantity of water than we did, but for a short period only, indeed without any steeping at all apparently, whereas we steeped the ground root in three successive quantities of water for a total period of 25 or even 50 hours, and always with beneficial results. The comparatively slight solubility of the glucoside permits indeed of considerable washing of the root without entailing any material loss of colouring power, and it seems to us best to prolong the washing a little in order to escape the necessity of adding large per centages of chalk or sodium carbonate to the dye-bath; excessive washing however must of course be avoided, otherwise loss of dyeing power would ensue through loss of morindine.

One interesting feature in connection with morinda dyeing is, that although the washed root undoubtedly contains the colouring principle still in the form of the glucoside, and which is of marked stability, it nevertheless dyes extremely well. We are unable at present to explain this, and are inclined to think that the presence of the mordant on the fabric has some influence in the matter, although we have had no time as yet to study this point.

For our supply of morinda-root we have to express our thanks to Mr. W. Reid of Bombay, and to Sir F. A. Abel, Bart., of the Imperial Institute. Small samples were also obtained from Dr. G. Watt during his visit to England in 1885 in connection with the Indian and Colonial Exhibition.

#### *Mang-kudu or Wong-koudou.*

This dyestuff is the root-bark of *morinda umbellata*, and may be regarded therefore merely as a variety of the *Al* root of India.

Some of its Indian vernacular names are as follows:—*Al* (Bomb.); *nūna, kai, nūna marum* (Tamil); *mūlūghūdū* (Telugu); *mang-kudu* (Malay).

It is a shrub met with in the hilly regions of Eastern Bengal, also in South Western India, the Southern Konkan, the Nilgiri Hills, the mountains of Travancore, also in Ceylon, the Malay Peninsula and Java.

Although this dye-stuff, said to cost about 6d. per lb., is largely employed by the Javanese for producing the fast reds in their celebrated "*Baticks*," it does not seem to be specially used by the Hindoos, unless indeed the whole root is sold as *Al* root.

Some years ago we received a small specimen of mang-kudu, also "*Baticks*" from our friend Mr. F. Driessen of Leiden, on his return from Java, and to his firm *De Leidische Katoenmaatschappij* we are indebted for the 10 lbs. of root-bark with which our experiments have been made, and for the gift of which we here express our best thanks. Till recently, however, we were ignorant of its botanical origin and for definite information on this point we have to thank Mr. T. W. Thielton-Dyer, the Director of Kew Gardens, although we had already come to the conclusion that it must be some species of morinda root-bark owing to our recognition of its colouring matter as morindone.

The commercial dyestuff has the appearance of a somewhat reddish-brown and wrinkled bark, curled up into

irregular rolls. Water extracts from it only a yellow colour, but alkaline solutions are immediately coloured red.

*History.*—It does not appear to have been subjected to any extended examination either by dyers or chemists. Gonfreville refers to it in his "*L'art de la teinture des laines*," p. 489, as *jong-koutong* or *woon-koutou* coming from Java and associates it with *atch-root* which he had received from Bengal. He recognised its similarity to the root-bark of *nonna* (*Morinda citrifolia*), and although he gives it as the bark of a Chinese tree, he is inclined to think it is the root-bark of the same plant which yields *atch root*. On wool, silk, and cotton he was able to obtain with it by the aid of different mordants, orange, red, and especially fast dark colours. He considered it a very valuable dyestuff, and points out how highly esteemed it is by the Javanese. A large number of samples of cotton, linen, silk, and wool dyed with it were sent to the French Exhibition held in 1839.

Baneroff very briefly refers to morinda *umbellata* as being employed in Cochin China and other parts of Asia as a yellow dye, but does not seem to have had an opportunity of experimenting with it.

It was examined by Schwartz and Koechlin in 1832, under the name of *Ooungkoudou*, and its dyeing properties were reported upon in their communication to the Industrial Society of Mulhouse already referred to. They found it to possess only  $\frac{1}{2}$ — $\frac{1}{3}$  of the dyeing power of good Avignon madder, and although it contained a less proportion of acid yellow principles than munnjet, it nevertheless required an addition of 30 per cent. carbonate of soda to the dye-bath. The colours obtained from it were said to be neither so bright nor so fast as those derived from morinda-root (*nona*).

T. Wardle, in his *Monograph on the Dye-stuffs and Tanning Matters of India and their Native Uses*, descriptive of the Collection in the Indian Section of the Paris Exhibition, 1878, states that the roots of this plant yield a yellow dye, and with the addition of Sapan wood a red dye for cotton.

In Dr. G. Watt's *Dictionary of the Economic Products of India*, 1891, Dr. J. Murray reports that like all other members of this genus, the root yields a dye, but of a brilliant yellow, not a red, colour. From the small space devoted to it we imagine it must be a dye-stuff of little or no importance in India.

*Experimental Part.*—The chemical examination of this dye-stuff, now made for the first time, shows that it contains, like the root of morinda *citrifolia*, the glucoside morindone, also several other crystalline principles in small quantity, a full account of which will shortly be communicated to the Chemical Society of London.

Knowing the botanical origin of the dye-stuff and the colouring principle it contained, its application in dyeing presented no difficulty whatever. Certainly mang-kudu in its ordinary condition was not found useful in dyeing, but as in the case of *al* root, so here, a preliminary washing or steeping in water sufficed to remove the deleterious acid principles present, and thus to transform it into a valuable red dye-stuff. Here, too, an addition of 1.5 per cent. of sodium carbonate to the dye-bath was also found to be necessary.

Ordinary stripe-mordanted calico of the printer, as well as oil-prepared cotton mordanted with aluminium, chromium, and iron, all furnished excellent reds, purples, browns, and blacks, fast to soap, practically the same as the analogous colours produced from morinda-root, but much fuller since mang-kudu possesses a much greater colouring power than *al* root. This fact is, of course, not to be wondered at, for it is well known that in ordinary morinda-root the colouring principle is situated chiefly in the bark of the root.

Our results differ from those of Schwartz and Koechlin in so far, that they lead us to consider mang-kudu as a valuable dye-stuff for India, comparing very favourably indeed with madder. With respect to the yellow colour obtainable from it on silk and wool, referred to by T. Wardle and others, it is, of course, due to the glucoside morindone, and hence of questionable value as a permanent dye.

*Ventilago Madraspatana.*

The root-bark of this plant belonging to the Rhamnear and known in England as the "purple chuckway," furnishes a dyestuff much valued in Southern India.

Some of its vernacular names are as follows: pitti (Hindi); raktapita (Bengali); pappili chukka, saradattai, pupli (Tamil); popli-chukai (Kan).

It is an extensive climbing shrub, found in the Western Peninsula from the Konkan southwards, also in Ceylon and Burma.

At a certain period of the year immense numbers of coolies in Mysore proceed to the jungle to collect the root-bark, which is then conveyed to the nearest towns and sold to dealers, who export it to other districts of India.

The root-bark has the appearance of thin scales, ribbons, or filaments of a deep purplish or chocolate colour. When ground to powder, the dust which is given off irritates the throat in a marked manner. Boiling water or alcohol readily extract red caustic alkalis a deep crimson colour.

*History.*—Gouffreville refers to it as being employed in India and Java for dyeing silk, wool, and cotton fabrics either violet, brownish-red, or black, all of which are accounted permanent.

He gives a long list of the reactions of different chemical reagents upon a strong aqueous decoction, and seems to have made a few dyeing experiments with it himself, and by the use of tin, alum, and iron mordants obtained red, violet, and chocolate colours. In his day the price in different parts of India varied from  $\frac{1}{2}$ l. to 1l. per lb.

We find no reference to any other examination of this dyestuff having been made till 1878—1887, during which period samples of the root-bark were obtained by the India Office and submitted to Mr. T. Wardle. He found it to be exceedingly rich in a red colouring matter, capable of producing many colours, for which cochineal and madder are generally used, and well adapted for *tusar* silk. The colours obtained by him, but with what mordants he does not state, inclined towards purple and chocolate, but generally reds more or less pure. He had not the opportunity of testing them for fastness, but judged them to be fairly permanent. He believed it would be a valuable acquisition to the dye-house, and might be largely used if it could be obtained in quantity at the price of 3—5 annas ( $4\frac{1}{2}$ d.—7 $\frac{1}{2}$ d.) per lb., mentioned in a report by Surgeon-Major Bidie. Some writers state that in 1888—89 it was sold at as low a price at 1—1 $\frac{1}{2}$  annas ( $1\frac{1}{2}$ d.—2 $\frac{1}{2}$ d.) per lb. It seems to be collected annually to the extent of 1—3 tons.

No special chemical examination of the colouring matter seems to have been made hitherto; the only references on this point relate to the variously coloured precipitates given by aqueous decoctions with a few metallic salts, and are to be found in the *Pharmacographia Indiae* l. 355, of Dymock, Warden, and Hooper, where it is suggested that the colouring matter is probably one of the derivatives of anthracene, though on what grounds this statement is made is not given.

*Experimental Part.*—The results of our chemical examination of this dyestuff, which are not yet completed, will be submitted ere long to the Chemical Society of London; suffice it to say here that it contains a considerable quantity of a resinous colouring matter soluble in alcohol, and but slightly soluble in water, which is readily attracted by mordants.

On stripe printed calico the root-bark gives with alumina mordant a claret-red, much bluer than a limawood-red, and not unlike that of alizarin-bordeaux; with iron mordant a greyish lilac which in strong colours approaches a black; and with a mixture of the two a very purplish dark chocolate. The lilac differs entirely from that given by alizarin-bordeaux by being weaker and greyer, and is more similar to the one given by limawood. The colours are moderately fast to soap, very much better than limawood colours, but considerably behind the alizarin colours in this respect. In point of fastness of soap we should class this dyestuff with camwood, although the colours given by

ventilago with the different mordants differ materially in shade from those given by camwood, the reds being bluer, clearer or brighter, and less bronzy, and the lilacs weaker.

Comparative dyeing experiments made with the purified colouring matter showed that the root bark of ventilago contains 8—10 per cent colouring matter.

On oil prepared calico ventilago gives with alumina mordant a rich claret-brown, with chromium mordant a deeper purplish-brown or very black-purple, and with iron mordant quite a good purplish black.

On wool chromium mordant gives a good purplish-brown, alumina a bordeaux-red, tin a brighter red similar to an alizarin red with alumina mordant, and iron mordant gives dark dull purple and black. On silk similar colours are given.

No difficulty at all is experienced in dyeing with this dyestuff, the ordinary modes of mordanting suffice, and no additions to the dye-bath are necessary except in the case of wool, with which it is desirable to add calcium acetate in order to correct the strong acidity of the mordanted fibre. The chief noticeable feature perhaps is that, owing to the slight solubility of the colouring matter in water and its resinous character, the dyeing does not commence until the temperature reaches 70—80 °C.

The fastness of the colours to light have not yet been tested, but owing to the general similarity as we think of the dyeing properties of this dyestuff to those of limwood and camwood, we are not sanguine upon this point. Nevertheless, the strong colouring power of ventilago and the ease with which it dyes mordanted fabrics, coupled with its comparatively low price, warrants us in considering it a dyestuff worthy even of the attention of European dyers, and it certainly must be ranked among the ready useful dyestuffs of India.

For our supply of 10 lbs. of this dyestuff we express our thanks to Sir P. A. Abel, Bart., Director of the Imperial Institute, from whom and his coadjutors we have received every assistance in the supply of material.

Finally we may add that we have already been and still are engaged in examining the chemical and tinctorial properties of other Indian dyestuffs, *e.g.*, Kamela, *Tesla* flowers, &c., and hope in due time to submit to the Society a record of our experimental results.

## DISCUSSION.

Mr. G. W. SLATER regretted that the results of the earlier experimenters had been found to be incorrect; they might, perhaps, have worked with material differing in age and other particulars from that examined by the authors. As so many of the plants examined belonged to the order *Rubiaceae*, it would be worth while to examine whether all the members of this order do not yield dyestuffs.

Mr. RAWSON was glad that natural colouring matters were again being investigated. From the commercial point of view the most important question was, at what price could these dyestuffs be obtained. All the shades which they yielded could be produced equally well with alizarin, and, if these dyestuffs were to compete with this latter, the roots containing them would have to be produced at an exceedingly low price. Was the yellow dye on wool obtained with morinda root fast to light? And was the colouring matter obtained from ventilago fast?

Mr. WILKINSON said it was pleasant to him to find Gouffreville's work corroborated; it was work that had been a labour of love, as all who had read his work must recognise.

Mr. PERKIN said, in reply, that they had worked with larger quantities than the older experimenters used, and so were enabled to discover the presence of, and to remove, certain substances which had a deleterious effect upon the action of the dye; this was why they had obtained better results.

Prof. HEMMEL said that they proposed to extend the investigation to other members of the order *Rubiaceae* or allied plants. There was no probability that morinda

or chay-root would compete with artificial alizarin, for that had already displaced madder-root, which was at least as good as the two first named. They had rather, in their investigation, a desire to gain definite information regarding the chemical and technical properties of various Indian dyestuffs, many of which had not hitherto been examined. The yellow dye, obtained on silk or wool with mordant in an acid bath, was probably not fast to light; it had not, however, been tried in this respect. There was no absolute necessity to find a fast natural yellow, as several good artificial ones were known, *e.g.*, among the acid and benzidine dyes. He had heard, indirectly from a former director of a dyeing school in India, that a natural yellow very fast to light was known in that country. The colouring matter from *Ventilaga* was moderately fast to soaping. It was quite true that Gouffreville did work *con amore*.

The CHAIRMAN said that he felt sure the section was grateful to the authors for bringing forward the results of a piece of work fraught with many difficulties. Mr. Perkin, he believed, had one of the most treasured qualities of the organic chemist, the genius for crystallising, and had succeeded admirably in isolating the beautiful specimens which had been exhibited. It was worthy of note that the paper was the outcome of the assistance of two important institutions, the Clothworker's Company and the Imperial Institute. The functions of the latter body might seem to others as to himself somewhat indefinite, but there could be no doubt that a searching examination into the nature and value of Indian products was a matter of imperial importance. Whether or not these dyestuffs turned out to be specially useful, it was highly desirable that they should be investigated. Pictitious importance was apt to attach to materials coming from a distance, and it was possible that the treasured dyestuffs of the East might prove to be devoid of any of those special qualities of permanence and tint which were still erroneously supposed to differentiate natural from artificial colouring matters. The previous investigation of Indian dyestuffs appeared to have been imperfect and of limited practical value, and it was well that the Imperial Institute was taking up the matter again and securing an exhaustive examination both from the chemical and dyeing points of view.

## THE THEORY OF DYEING.

BY C. F. CROSS AND E. J. BEVAN.

In the last number of this Journal there appear two interesting communications upon this subject, both attacking what appears to be known as the "solid solution" theory, and substituting in general terms, presenting features of similarity, a counter-hypothesis basing the cause of dyeing phenomena primarily upon osmotic action, with the play of chemical reaction between the colouring matter and fibre substance as an auxiliary effect more or less operative. Both authors [Mr. Dreaper, p. 95, and Dr. Weber, p. 122] make mention of some observations of our own [*ibid.*, 12, 104], upon the theory in question, and upon a dyeing process of peculiar and significant bearing thereon, which we made in connection with researches on the chemistry of lignocelluloses. Mr. Dreaper has taken our interpretation of the phenomena as we intended it to be taken; Dr. Weber, on the other hand, has given an inverted and "improved" statement of our conclusions, which he will find in some measure corrected on reading Mr. Dreaper's paper, and which he will, no doubt, pardon our correcting further.

That the jute fibre when plunged into a solution of ferric ferrieyanide not only reduces the compound to the lower blue cyanides, but combines with the blue to the extent of 30—50 part of its weight, distributed with perfect uniformity throughout its substance and without prejudice to its lustre, is, it must be and is, in fact, admitted to be, unique in the range of dyeing phenomena. We were careful to insist upon the special chemistry of the reaction, and to point out that

it is by no means due to mere contact reduction by the aldehydic groups of the fibre substance, as suggested by Dr. Weber—the reaction proceeding in presence of powerful oxidants, such as chromic acid—but depends rather upon the special constitutional relationships of the reagents. We implied, in fact, that it must be interpreted as largely conditioned by the hexene configuration of the characteristic groups of the fibre substance in relation to the probable hexacentric formula of the ferric and ferroso-ferric cyanides; the "reduction" of the ferric ferrieyanide taking place within the fibre substance in virtue of a transference of oxygen, easily accounted for in the presence of CO groups of both Ketonic (R. Hexene groups) and Aldehydic (oxycellulose groups) function. [Ber. 26, 2520.]

We pointed out that the reaction might be considered as taking place in two stages: (1) the fixation of the ferric ferrieyanide by the fibre substance, and (2) the reduction or redistribution of oxygen, as above described, giving the well-known colouring matter. In evidence of the probability of (1), we showed that in the case of another colloid substance, *viz.*, gelatin, such a combination does take place, solutions of this colloid giving a pale greenish, opaque coagulum with the ferric ferrieyanide, converted by subsequent treatment with reducing agents into a transparent, deep blue jelly, which can only be regarded as a solution of the ferroso ferric cyanide (hydrate) in the "organic" colloid (or its hydrate).

From this statement it should be perfectly clear that we do not regard the entire reaction in the light of "a striking instance of solid solution." Dr. Weber, in crediting us with this view, does not sufficiently separate the *process* from the *product*. In the *process* it is quite obvious that the special chemical factors are predominant, and we emphasised it as further evidence of the special constitutional features of the lignocellulose. The *product*, on the other hand, is a solid solution of the pigment in the fibre substance; and we fail to see any necessity to resist the conclusion. The "improved" interpretation which Dr. Weber gives of the phenomenon or reaction, viewed as a whole, we will not criticise in detail. We notice, however, that his interpretation involves a contradiction in terms of his own conclusions, as to the mechanism of substantive dyeing in the case of cotton. In criticising us he says: "The location of the dye within the fibre is by no means a case of Witt's 'solution,' but merely proves the perfect intramolecular penetrability of the jute fibre by aqueous solutions. This perfect penetrability of jute fibre and all other fibres calls for no special explanation here, being an absolute condition of the growth and life of the vegetable and animal fibre." But when the author deals with cotton and the benzidine dyes, this particular fibre is found to have a one-sided penetrability; the process of direct dyeing with benzidine dyes is described "as an aqueous solution of a dye of very small co-efficient of diffusion formed inside the cellular cavities of the cotton fibre by means of a dye bath possessing a high osmotic pressure." We should like to know how the author would regard the dyeing of the jute fibre substance or structureless cotton cellulose with the benzidine dyes; or what function he would assign to the cellular cavities of cotton in direct dyeing with "primuline"?

This colouring matter in its relation to cotton is the subject of Mr. Dreaper's communication. This author also lays special stress upon the osmotic factors of the process of dyeing, and in our opinion does not fully recognise what is stated by the upholders of the "solid solution" theory, and also what is not stated, which under the present circumstances of the controversy is perhaps more important. In the primuline dyed *product*—and again we emphasise the distinction from the *process*—we have preëminent evidence of the existence of the colouring matter in solid solution.

The conception of solid solution we take to be an extension of the modern theory of solution to cover the case of homogeneous distribution of one solid throughout the mass of another, considered as the solvent. This modern theory, as we read it, is not so much an effort to account for the actual mechanism of solution, *i.e.*, the *process*, but concerns itself rather with the condition of the dissolved substance *in solution*. The consensus of evidence from the various



independent directions of investigation (Van't Hoff: Ber. 27, 6) leaves no doubt that it is a condition of dissociation or molecular simplification, such as has long been recognised to prevail in vapours and gases: that is that in a highly dilute solution of one substance in another (the solvent), the properties of the dissolved substance are those which it possesses when gaseified. In our opinion this conception perfectly explains the extraordinarily increased sensitiveness (to light) of diazotised primuline, when diazotised upon a fibre substance or other colloid basis (gelatin), as compared with its comparative stability as diazoprimuline *per se*. More than this: in our opinion there is no other explanation, in the present condition of our knowledge. No doubt in the case of primuline, as of the diazotised primuline, and a fully developed "ingrain" dye, there is a chemical bond of union with the fibre substance: but in virtue of that bond we should expect a decrease of molecular mobility, and therefore a decreased photo-sensitiveness of the diazo-product. To complete the evidence, however, we may remember that the synthesis of the primuline and fibre substance is of such an order that the capacities of the former for further synthesis remain perfectly unaffected; and that again is one of the essential features of *solution* as distinguished from combination.

To sum up these remarks we express our opinion that the dyeing of jute with ferroso-ferrie cyanide and cotton with primuline, are of unique and critical value in regard to the theory of solid solution as applied to the consideration of dyeing phenomena generally: that so far as Witt or others have given any exposition of such a "theory of dyeing," it has been in regard to the *dyed product*, as distinct from the mechanism of the actual process of transference of the dissolved dyestuff to the fibre substance. In elucidating the latter Dr. Weber's recent papers on "lake formation," and his later investigations of the function of the osmotic pressure of the dye solution, are of great interest and obvious value. The differentiation of this factor is only possible, with any precision, when applied to a series of bodies presenting definite constitutional variations. In this respect his choice of the benzidine series is well considered and his results will no doubt prove beyond criticism. But we would still ask him to recall the view of solid solution of the dyestuff in the colloid fibre substance, in spite of his attempt to dismiss it; and to separate this view from that of the *process*, in which we have a complicated play of chemical and physical effects.

In regard to the chemical effects involved they appear to be very largely of the order of double-salt formation. Dr. Weber himself has very largely contributed to our knowledge of the artificial colouring matters in regard to this as part of their activity: and investigation of the constitution of the substance of fibres of both animal and vegetable origin, continues to differentiate their reactive OH groups into those of acid and of basic function. In the case of crystalline double salts the bond of association is usually one of ascertained similitude whether in the function of the dominating element or the form of the individual salts. In colloid substances we have every reason to assume that coalescence is determined by likeness of configuration. What the nature of this "isomorphism" may prove to be in the relationship of *colloid* substances (usually described as "amorphous") is a problem which is being very distinctly opened out by the investigation of dyeing phenomena. In conclusion we take it that the theory of dyeing embraces the following factors:

1. The molecular constitution (configuration) of the reagents, colouring matters and fibre or tissue substance.
2. The presence of salt forming groups, chiefly OH groups differentiated in function both by the groups with which they are in immediate or proximate contact, as well as by the configuration of the C nucleus to which they are attached.
3. The condition of the colouring matter in solution measured by the physical constants of the solution and modified by the presence of the fibre substance in the dye bath.

The final result or dyed product may be considered *per se*, or in relation to the dyeing process: in regard to the

former we fail to see any objection to the theory of solid solution if indeed it is not a fact rather than a theory: as regards the process, which would therefore be a transference from one solution to another, the conditions of the transfer are both chemical and physical. Any theory embracing these conditions will no doubt assist in the advance of knowledge, but to exclude the consideration of the one or the other would be evidence that the subject had been approached from too narrow a point of view.

#### DISCUSSION.

Professor HEMMEL wondered if the jute fibre had been dyed by natural or by artificial light, since ferrieyanides, under certain conditions, were capable of reduction by light. Chromic acid, also, was sensitive to light and need not necessarily prevent the reduction of the ferrieyanide. He himself felt diffident in accepting Weber's view, that the colouring matter diffused through the cell wall into the interior of the cotton fibre, and was there deposited or dried on to the inner cell wall. If this was really the idea in Dr. Weber's mind, which he doubted, the authors put a pertinent question in asking Weber how he would explain, on this theory, the dyeing of amorphous cellulose. His own opinion had always been that the colouring-matter was uniformly distributed throughout the substance of the cell wall.

Dr. WEBER sent the following communication:—

What strikes me more particularly in this paper is the suggestion of the authors that in my paper (this Journal, 1894, 122) I gave an "inverted" account of the reaction between lignocellulose and ferrie ferrieyanide observed by them, and further that my statement of the "solution theory" of dyeing is not what Witt expounded. This announcement of Cross and Bevan I can only understand by assuming that they have not yet realised the difference between the "solid solution" theory, as pronounced by Van't Hoff and Arrhenius, and the "solution theory of dyeing" as put forward by Witt. Witt's "theory" is the outcome of the total misunderstanding of Van't Hoff's theory, which was originally meant to apply to metallic alloys and "isomorphous mixtures" only. Nobody in the least acquainted with the dyeing phenomena, on reading Witt's original paper, will doubt for a moment that Witt's theory does not profess to apply purely to the physical constitution of the dyed fibre as a whole, but distinctly assumes, in designed opposition to the chemical theory of dyeing, that the very process of dyeing, instead of being essentially the consequence of chemical differences between dyestuff and fibre, merely consists in the migration of the dye from one solvent into another. This is proved beyond doubt by the facts Witt cites in support of his theory, all of which are calculated to furnish evidence of the existence of the dye in the fibre in the free state. Witt is too experienced a dyer not to see that chemical interaction between dye and fibre once being granted, the "solid solution" theory becomes as superfluous as it would be for the explanation of the formation of barium sulphate from baryta and sulphuric acid. So much about Witt's notion of the function of "solid solution" in dyeing, which evidently has been as much misunderstood by Cross and Bevan as Witt misunderstood Van't Hoff. Cross and Bevan's complaint of my "inversion" of their experiment is evidently due to the same misunderstanding, as they seem to wish it to be taken as evidence of the state of the dyed lignocellulose, whereas my criticism was directed against the chemical mechanism or dyeing process underlying the reaction. But if the latter does not bear out or renders altogether superfluous their scantily supported hypothesis as to the final state of dye and fibre, a criticism of their experiment with regard to this point would have been superfluous.

Transference of a substance from one condition of solution to another is indeed, as Cross and Bevan assert, a solution phenomenon pure and simple. But then, no chemical change of the dissolved substance must be involved in passing from one solvent into the other. As I have, however, shown in my paper, such a chemical change is involved in all dyeing processes, with the single exception



of the dyeing of benzidine dyes on cotton. Consequently what Cross and Bevan maintain is dissolved in the fibre substance, is an altogether different compound from that held in solution in the dyebath. Cross and Bevan's contention that "the association of colloids in a dyed fibre is a case of double salt formation, the bond of union being probably an isomorphism of molecular configuration," I cannot argue; the meaning of this sentence is as obscure to me as the writings of Paracelsus.

With regard to the considerable difference between the sensitiveness to light of diazopyrimuline produced upon the fibre and of the same compound in the free state respectively, this is certainly a point of considerable interest, but Cross and Bevan have adduced no evidence warranting their bold assertion that this is due to the dye on the fibre being in "a condition of molecular simplification or disaggregation." The authors are greatly mistaken in saying that this is all the solution theory asserts, and I repeat that Witt's theory is not a theory of the state of the dyed fibre alone, but also of the process of dyeing. If Cross and Bevan object to this interpretation, they will have to come forward with a solution theory of their own. They may, however, find some difficulty in this task, for a theory regarding the state of the dyed fibre, but unavailable for the elucidation of the dyeing process, contains all the elements of a scientific abortion.

The three points to be borne in mind, according to Cross and Bevan, in the working out of a theory of dyeing, have been very fully considered in my paper, which, indeed, gives a full account of the chemical mechanism of the phenomena in question. Their finding that this is not so, I can only take as proof that they have not sufficiently digested the contents of my paper.

By a curious coincidence a paper on the theory of dyeing was read before the London Section of our Society by Mr. Dreaper on the same evening as that on which I brought my paper before you. The result of Mr. Dreaper's valuable investigations, in which he attacked the question from quite a different point than I did, leads essentially to the same result as my own, *viz.*, to an unconditional rejection of the solution theory as propounded by Witt.

## ON THE MORDANTING OF WOOL WITH CHROMIUM.

### PART IV.

BY L. LICHTI AND J. J. HUMMEL.

(See also this Journal, 1893, 240 and 332; 1894, 222.)

THE following communication is a continuation of our study of the behaviour of chrome alum as a mordant for wool when it is employed along with acids as assistants. In our previous communication (this Journal, 1894, 222) we cited the effects produced by the addition of sulphuric acid and acetic acid, we propose now to refer to the action of oxalic, tartaric, and chromic acids, and then briefly to the behaviour of one or two other chromium salts as mordants for wool.

#### 3. Chrome Alum and Oxalic Acid.

At a very early period dyers must have noticed that when wool is mordanted with alum alone poor colours are obtained in dyeing, and in order to avoid this defect they began to add, along with it, organic acid, in the form of cream of tartar. No doubt its employment in preference to other acids was the result of pure empiricism, and who the discoverer was is altogether unknown. At the present time one endeavours, wherever possible, to economise by replacing it with the much cheaper oxalic acid.

In the course of the following experiments it was noticed that when organic acids were employed along with chrome alum for mordanting wool, the green solutions became more and more violet according to the amount employed, from which it would appear that their addition retards or prevents dissociation of the chrome alum. Comparing the effect of equivalent amounts of oxalic and tartaric acids the latter gives greener solutions, and such as show less

tendency to become turbid. Closely connected with this is the fact that an addition of oxalic acid to a chrome alum solution only partially prevents its precipitation by caustic soda, whereas an addition of tartaric acid entirely prevents it. Altogether the behaviour of these two acids towards chrome alum is somewhat different.

In determining the efficacy of oxalic acid as an assistant along with chrome alum the first question which arises is, ought it to be used in the free state, or half or wholly neutralised with soda. The results vary somewhat according to the amount of oxalic acid employed. With 2 mols. oxalic acid per 1 mol. chrome alum the best results were obtained with the free acid, with 3 mols. it was best to have the acid half neutralised.

*Experiment 24.*—Wool was mordanted with—

- (1.) 10 per cent. chrome alum + 2 mols.  $\text{H}_2\text{C}_2\text{O}_4$  (oxalic acid).
- (2.) 10 per cent. chrome alum + 2 mols.  $\text{NaHC}_2\text{O}_4$ .
- (3.) 10 per cent. chrome alum + 2 mols.  $\text{Na}_2\text{C}_2\text{O}_4$ .

No. 3 bath gave one the impression of being alkaline, its colour being yellowish green, and it showed distinct evidences of dissociation by its turbidity at the end of the operation, whereas No. 1 bath was perfectly clear, No. 2 bath showing an intermediate appearance. No. 1 felt seemed to be the best mordanted, then Nos. 2 and 3. The colour of the dried mordanted wool was a pale greyish green, No. 3 being distinctly yellower and paler than the others. The patterns were dyed with 40 per cent. quercitron bark; No. 3 was much paler than No. 1, indeed scarcely half as deep, and it exhibited differences in colour in the different fibres, to which reference has previously been made, in a more marked manner than in Nos. 1 and 2.

The result of this experiment is that neutral mordants are distinctly unfavourable for wool.

When the experiment was repeated with 3 mols. oxalic acid instead of 2, the 3 mols.  $\text{NaHC}_2\text{O}_4$  seemed to act slightly better than 3 mols. of the free acid, no doubt because this is already an excess of acid.

*Experiment 25.*—Six fents of wool were mordanted with the following:—

- (1.) 10 per cent. chrome alum.
- (2.) 10 per cent. chrome alum + 1 cc. normal oxalic acid ( $= \frac{1}{2}$  mol.).
- (3.) 10 per cent. chrome alum + 2 cc. normal oxalic acid ( $= 1$  mol.).
- (4.) 10 per cent. chrome alum + 3 cc. normal oxalic acid ( $= 1\frac{1}{2}$  mols.).
- (5.) 10 per cent. chrome alum + 4 cc. normal oxalic acid ( $= 2$  mols.).
- (6.) 10 per cent. chrome alum + 5 cc. normal oxalic acid ( $= 2\frac{1}{2}$  mols.).

With increasing amounts of acid the dissociation apparent in No. 1 liquor gradually diminishes, No. 3 is still faintly turbid, Nos. 4, 5, and 6 are quite clear.

The dried mordanted fents appear as follows:—No. 1 yellowish green, No. 2 similar to No. 1 but less yellow, Nos. 3 and 4 are still less yellow, No. 5 has the deepest colour, a greyish green, No. 6 is slightly paler than No. 5. It is interesting to note that in gas light the mordanted fents exhibit a peculiar reddish tint, and one is tempted to entertain the opinion that the chromium is present on the fibre in some peculiar soluble condition. The liquors too, with increase of oxalic acid, acquire a reddish tint, which may possibly be explained by supposing that a part of the chromium has been taken up by the fibre as  $\text{Cr}_2(\text{SO}_4)_3$  in addition to free sulphuric acid, and that in the bath there remains  $\text{Cr}_2(\text{C}_2\text{O}_4)_3$  as well as  $\text{Cr}_2(\text{SO}_4)_3$ . This change of colour occurs only during the mordanting process, notwithstanding the presence of the oxalic acid the hot liquor is first green, but while the wool gradually acquires a greyish-green colour the bath becomes more and more violet, appearing when cold distinctly red, quite like the colour of pure chromium oxalate. On heating chrome alum solution with 3 mols.  $\text{Na}_2\text{C}_2\text{O}_4$  without presence of wool, a violet solution is also obtained, showing therefore that interaction occurs at least partially; here the sulphuric acid would be taken up by the NaOH in place of the wool.

If the above-mentioned opinion is correct, the purpose of the necessary additions of cream of tartar, tartaric acid, oxalic acid, &c., is evidently to prevent the decomposition of the alum through the absorption of the sulphuric acid by the wool.

The six fests of the present experiment were dyed with 12 per cent. alizarin, with the result that No. 5, i.e., the mordant  $\text{Cr}_2(\text{SO}_4)_3 + 2\text{H}_2\text{C}_2\text{O}_4$  gave the best colour. Under the microscope No. 1 showed dull red and almost colourless fibres, differences which became less apparent from Nos. 2 to 4, and almost disappeared in Nos. 5 and 6. It may be added that a much deeper claret colour was obtained if the wool mordanted with chrome alum and 2 mols. oxalic acid and washed was passed, previous to dyeing, through a very dilute sodium carbonate solution; for 600 cc. water 0.53 grms.  $\text{Na}_2\text{CO}_3$ , i.e.  $(\frac{1}{1000000})$  mols.).

A further observation may be here cited. The appearance of the waste dye-liquor of No. 5 indicated that it contained excess of alizarin, and possibly chromium mordant as well, and this proved indeed to be the case, since on boiling in it a small piece of wool (1.66 grm.) it acquired a pink colour. Wool mordanted with chrome alum and 2 mols. tartaric acid exhausted the dye-bath somewhat more, and the waste dye-liquor dyed a piece of fresh wool a paler pink than the foregoing in consequence. Altogether, wool mordanted with the addition of tartaric acid seemed to be better mordanted, the yellowish-green line being replaced by one of a pure greyish green.

**Experiment 26.**—In this experiment the intention was to discover, if possible, the conditions necessary to prevent the different behaviour of individual wool fibres, since this hinders materially the utility of chromic oxide salts as mordants.

- (1.) 10 grms. wool were first boiled for an hour in 600 cc. water, and then mordanted with 10 per cent. chrome alum, and 2 mols. oxalic acid.
- (2.) 10 grms. wool were boiled for an hour in 600 cc. water with addition of 4 cc. normal oxalic acid (2 mols.), then 1 grm. (1 mol.) chrome alum was added, and the boiling continued for  $1\frac{1}{2}$  hours longer.
- (3.) 10 grms. wool were mordanted in the ordinary manner with 1 grm. chrome alum, and 4 cc. normal oxalic acid (2 mols.).

All the liquors acquired a violet colour, the mordanted wool having a greyish-yellow-green colour, No. 2 being deeper than No. 3. When dyed in alizarin, No. 1 gave the deepest and best colour; then came No. 2, and finally No. 3. Since No. 1 is better than No. 2 the action seems to be physical rather than chemical, as though the preliminary boiling of the wool softened the fibre, and caused it to absorb the mordant better.

**Experiment 27.**—The object of this experiment was to determine the behaviour of pure chromium oxalate as a mordant. The preparation of this salt was effected as follows: a cold and very dilute solution of chrome alum was precipitated with the calculated amount of  $\text{NaOH}$ , and the precipitate after frequent washing with water was collected on a filter, and ultimately dissolved in the calculated amount of oxalic acid.

Wool was mordanted with the following:—

- (1.) 10 per cent. chrome alum + 3 mols. oxalic acid.
- (2.) 10 per cent. chrome alum + 6 mols.  $\text{NaOH}$ , the precipitate being at once dissolved by adding 3 mols.  $\text{H}_2\text{C}_2\text{O}_4$ .
- (3.) 10 per cent. chrome alum + 3 mols.  $\text{Na}_2\text{C}_2\text{O}_4$ .
- (4.) An equivalent amount of pure chromium oxalate prepared as stated above.
- (5.) As 4, but with addition of  $\frac{1}{2}$  mol.  $\text{H}_2\text{SO}_4$  (i.e. 1 cc. normal acid) in order to neutralise the water and the wool.

The liquor of No. 4 was perfectly clear, of a violet-green colour, and mordanted the wool very well, giving it a full greenish-violet-grey colour; No. 5 behaved similarly; Nos. 2 and 3 gave results a little below those of No. 4; the wool mordanted with No. 1 had the palest colour, and this liquor seemed to be decidedly acid.

When dyed in alizarin Nos. 4 and 5 appeared the best colours, Nos. 2 and 3 were less bright, and No. 1 was distinctly the palest. Here, as in all cases where alizarin was used as the dye-stuff, calcium acetate was added to the dye-bath in an amount sufficient to form calcium alizarate.

Chromium oxalate gave much better results than chrome alum and 2 mols. oxalic acid, or than chromium fluoride, and since this mordant is comparatively cheap it may be strongly recommended for use on the large scale. To correct the temporary hardness of the water employed a little sulphuric acid would require to be added to the bath.

A microscopic examination of the dyed fibres supported this favourable view, and it may be added that of all the chromium oxide salt mordant baths that of chromium oxalate was the best exhausted, and the dye-bath following it, in the case of alizarin, was almost entirely exhausted. Here, too, a little chromium oxalate bled out into the dye-bath, as was shown by the waste dye-liquor being capable of dyeing a piece of fresh unmordanted wool. This fact was indeed not to be wondered at, since the 10 per cent. chrome alum employed was far too large an amount, and would, if it had been all fixed on the wool, have taken up 36 per cent. of alizarin (20 per cent. paste). For a nice claret-red, however, 12 per cent. alizarin is sufficient, and this requires only  $\frac{1}{3} = 3.33$  per cent. chrome alum.

It is certainly interesting to find that approximately this very amount of mordant is fixed by the wool, both when bichromate of potash and when chrome alum and oxalic acid (2 mol.) are employed.\*

This agreement, with respect to the amount of chromium fixed, to which reference was already made in Part II., notwithstanding the great difference in the chromium salts employed, seems to favour the view that a chemical combination exists between wool, chromium, and colouring matter.

The study of mordants and their action is one of extreme difficulty, and we are still far from understanding all the questions involved, hence the desirability of this branch of study being pursued more than it is at present. It would be important to discover, if possible, mordanting processes in which the baths are exhausted, so that the dyer who wishes to work according to strict chemical principles is able to fix, on successive quantities of goods, exactly the same amount of mordant. Suppose, for example, one has to mordant equally, 100 woollen pieces with bichromate of potash, in 10 lots, 10 pieces in each lot. According to some of the methods at present adopted, especially that one in which the bath is freshened up with additional mordant before entering the next lot of goods, an equal mordanting of the several lots of pieces is quite impossible. In the case of dyeing dark shades, the foreman dyer, by means of trial bits and frequent matching, will succeed with more or less success to obtain the shade required.

If, however, the problem is to dye a pale shade with alizarin, alizarin-blue, or coerulein, dyes which cannot be boiled off or on at will, inequalities in the different lots are scarcely to be avoided. In this case one is compelled to dye them according to a definite receipt. If now the work-chemist could introduce some volumetric analytical methods to determine rapidly the condition, acidity, &c. of the mordant baths, and if he determined previously the amounts of colouring matter required, it would then be possible to work with greater certainty of obtaining a good result. The peace of mind which would thus come to the dyer by knowing that the two essential elements were right, would leave him free to devote all his attention to other necessary but subsidiary points to ensure complete success. In this manner the period of suspense, the uncertain successive additions of dye-stuff, and the necessarily more lengthened boiling, would all disappear, and a larger output of finer goods would be the result. Another advantage in dyeing,

\* See this Journal, 1893, p. 243, concluding paragraph of second column, where, however, the passage should read thus: "Taking the view that only on third of the bichromate is really effective, and that for a full claret colour that is required 12 per cent. alizarin (20 per cent. paste), we find that the proportions are just those which are requisite to form normal chromium alizarate."

according to a definite receipt, would be that the shades would be obtained almost as well by artificial as by day light.

When wool is mordanted with organic chromium salts it never acquires quite the same green colour as that of chromic hydrate; the peculiar violet-greenish-grey colour always gives one the impression that a chromium salt is present in the fibre. Although wool absorbs organic as well as mineral acids, a dissociation of the chromium oxalate bath could never be detected; on the other hand, this occurred when in one experiment wool was mordanted in the basic chromium oxalate  $\text{Cr}_2(\text{OH})_2(\text{C}_2\text{O}_4)_2$ ; in this case, in which there was a deficiency of acid, the wool acquired a fine green colour.

At this point a few preliminary experiments will be referred to, in which wool was mordanted by padding in mordant solutions, then drying and steaming. The solutions were of such concentration that the amount of mordant presented to 10 grms. wool was equivalent to 1 gm. chrome alum.

(1.) Chromium oxalate applied in this manner gave a tint which, after washing, had a fine full greyish-green level colour. On dyeing with logwood a specially fine black was obtained; differences in the colour of individual fibres were small.

(2.) Chromium oxalate, with 1 mol. oxalic acid, gave a greyish-brown-green coloured wool, not quite level; the dyed colour was fairly good.

(3.) Chromium oxalate with 2 mols. oxalic acid gave a fine greyish green colour with violet tint; the dyed colour was not so good as the previous one; evidently there was already too much acid present.

(4.) Chrome alum gave an uneven yellowish-green pale chromic oxide colour; the dyed colour was worse than when oxalic acid was employed.

(5.) Chromium chloride and (6) chromium nitrate gave very poor results, worse even than the following:—

(7.) Chromium acetate gave an uneven yellowish-green colour; the dyed colour was very bad. The microscope revealed the greatest differences in colour in the different fibres, although chromium acetate is not dissociated, a fact which shows clearly that wool can only be properly mordanted with chromic salts in acid solution. In the case of chrome alum alone the differences in colour of individual fibres were very great, indeed even in the different parts of the same fibre, but by the addition of oxalic acid these differences became less marked; no colourless fibres were seen, at most such as were greyish-blue and bluish-black.

(8.) Chromium fluoride gave an uneven yellowish-green colour; the dyed colour was somewhat better than that given by chrome alum; blue as well as black fibres were noticed under the microscope.

One point worth noting in connection with wool mordanted in the ordinary manner is, that the less acid the mordanted wool is, the lower the temperature at which it begins to dye. Wool mordanted with chromium oxalate was strongly acid, and began to dye with alizarin only at a temperature of 70° C. As soon, however, as the calculated amount of calcium acetate was added to the bath, the wool began at once to acquire a deep colour and the bath was soon entirely exhausted, although it still had an acid reaction at the end of the dyeing process, having previously passed through a neutral if not even a slightly alkaline condition. It is here plainly evident that the acid previously absorbed by the wool is slowly bleeding out into the dye-bath.

Already in 1886 Liechi and Schwitser showed that wool mordanted with  $\text{Al}_2(\text{SO}_4)_3$ , when boiled several times with distilled water gave acid liquors, and that in the case of aluminium tartrate and oxalate the dye-baths were all acid; further, it was shown that no better colours were obtained by boiling out the mordanted patterns with water previous to dyeing (see Mittheilungen des techn. Gewerbe Mus. Wien, III., p. 48, &c.)

In order to throw if possible more light upon this question, the following experiments were made.

*Experiment 28.*—Wool was boiled with 10 per cent. chrome alum and 2 mols. oxalic acid. The fent was dried

and divided, one half being washed in a cold alkaline solution containing 10 grms.  $\text{NaHCO}_3$  per litre; both patterns were then dyed separately with alizarin and calcium acetate. The neutralised wool exhausted the dye-bath better, and acquired a deeper colour. Under the microscope, however, it did not seem as if really more colouring matter had been fixed, but rather that the increased depth was due to the alkaline condition of the colour.

*Experiment 29.*—In all the waste alizarin dye-liquors examined showing an acid reaction, although calculated amounts of calcium acetate were employed, one could always detect excess of lime and alizarin. It seemed probable, therefore, that the acidity of the bath prevented to some extent the entrance of the calcium into the colour-lake in the fibre, and that it might also be the cause of the well-known bleeding of alizarin colours during the milling process. This idea suggested the following experiment:—

First of all it was accurately determined how much lime water corresponded to 1.2 grms. alizarin (20 per cent. paste), with the result that to form the compound  $\text{C}_{14}\text{H}_8\text{CaO}_4$ , 42 cc. were found to be necessary. Woollen fents were then mordanted in the following baths:—

- (1.) 10 per cent. chrome alum and 2 mols. oxalic acid.
- (2.) 10 per cent. chrome alum and 2 mols. tartaric acid.
- (3.) Equivalent amounts of chromium oxalate.
- (4, 5, 6.) 10 per cent. chrome alum and 2 mols. oxalic acid.
- (7, 8, 9.) Same as No. 3.

During the mordanting process a slight difference in the colour of the various baths was noticeable, the oxalic acid baths being more violet, the tartaric acid baths more greenish violet, while the mordanted fents of the former were of a greyish-yellow green, and those of the latter greyish violet.

The fents Nos. 1—3 were dyed with the above-mentioned solution of  $\text{C}_{14}\text{H}_8\text{CaO}_4$ . Up to a temperature of 60—70° C. the wool acquired very little colour, so that it seemed necessary to neutralise the excess of lime with acetic acid. Of the theoretical amount of acetic acid required,  $\frac{1}{2}$ — $\frac{1}{4}$  sufficed for neutralisation. This dyeing experiment with  $\text{C}_{14}\text{H}_8\text{CaO}_4$  led to the conclusion that the acid chromium compound absorbed by the wool corresponds to the formula  $\text{Cr}_2\text{O}_3 \cdot 5 \text{ acid}$ . The chromium oxalate mordant bath was most exhausted, and the corresponding dye-bath was almost completely exhausted, a fine, full, deep claret colour being obtained. The claret obtained on the chrome alum and tartaric acid mordant was somewhat bluer in shade than that with chrome alum and oxalic acid, and both were somewhat paler colours than that on chromium oxalate mordant.

The best mordanting action is undoubtedly obtained when the chromic oxide is combined with an organic acid such as oxalic or tartaric acid.

The fents Nos. 4, 5, 6 were well squeezed after mordanting, and then dyed with 30 per cent. logwood, No. 4 being dyed without any further treatment, while No. 5 was first heated for a quarter of an hour with 600 cc. water containing 0.8 per cent. sodium acetate, and No. 6 with water containing 0.3 per cent. chalk; after thus neutralising the acidity of the wool, the logwood was added, and the dyeing proceeded with. The waste dye-liquors of Nos. 4 and 5 were decidedly acid; No. 6 was neutral. The amounts of these additions were proportional to the amounts of acid absorbed by the wool, as found previously; bath No. 5 was, of course, not really neutralised, but contained acetic acid equivalent to the oxalic or sulphuric acid absorbed. The dyed pattern No. 4 was a somewhat deep, fine, blue-black; No. 5 was slightly darker; No. 6 was bronzy, evidently through want of acid.

Incidentally, patterns Nos. 4, 5, and 6 showed plainly, what is invariably the case, that chromic oxide mordant with logwood does not give a real black. If one uses pure crystalline haematein, the colour obtained is a very fine dark blue, hence it is that some chemists consider that true chrome black is not a compound of chromic oxide with haematein, but with some oxidation product of haematein, and that a good black is only to be obtained with the use of chromic acid mordants. This fact is well known to practical wool dyers. Calico-printers, too, have long been in the habit of printing a mixture of logwood liquor (haematoxylin) and

chromic oxide salt, and then submitting the steamed fabric to a supplementary oxidation, or oxidising agents have been already added to the printing mixture.

Patterns Nos. 7, 8, 9, after mordanting, were well squeezed, and then dyed with 10 per cent. alizarin, No. 7 being dyed without any further addition. No. 8, with addition of 35 cc. lime-water, neutralised exactly with acetic acid, and No. 9 with 35 cc. lime-water only.

The waste dye-liquor of No. 7 was acid, and contained free alizarin; in addition of NaOH it gave a fair quantity of chromium-alizarin lake, which showed, therefore, that in the absence of lime a good deal of chromium oxalate had out into the dye-bath. The dye liquor of No. 9 was only neutral at the very end of the operation, and was perfectly exhausted. No. 8 was very similar. Both gave mere traces of alizarin-lake in addition of caustic soda. The intensity of colour increased from Nos. 7 to 9, No. 9 being a deep bluish claret-red, while No. 7 was much paler and redder. All three patterns were satisfactorily level, even when the fibres were examined under the microscope. Very probably the colours of patterns Nos. 8 and 9 would stand milling better than if dyed on chrome alum and oxalic acid or cream of tartar. Under the microscope No. 4 showed the palest colour with logwood, and the fibres showed slight differences in colour, evidently because of the different amounts of acid absorbed; the colour was a fine blue, not quite so dark as No. 5, which still showed irregularity. No. 6 was still better, a fine dark blue of normal appearance, with fibres of fairly equal colour. Through an error of calculation, too much  $\text{CaC}_2\text{O}_4$  had been used; probably with the right amount the result would have been still better. Of course, in practice, the amount to be used would vary according to the quality of the water employed; hence it is unnecessary to give further details on this point.

In the case of alizarin, No. 9 pattern showed the effect of an alkaline bath, but here it is very necessary to avoid all excess of calcium carbonate, otherwise loss of alizarin may readily occur.

#### *Chemistry of the action of Oxalic Acid as an assistant.*

It has long been recognised in practice that ordinary alum when used alone is unsuitable for mordanting wool, and in an empirical manner it was found that the addition of cream of tartar gave better colours, which would not rub off so readily. The idea appears to have been that it acted beneficially by reason of its solvent action, a view which seems to be both natural and rational in the light of our results concerning the decomposition of chrome alum by the wool, from which we have seen that an addition of acids or acid salts (e.g., cream of tartar) really prevents the precipitation of insoluble basic sulphate, and the consequent rubbing off, &c. Hummel and Gardner have already shown that comparatively large amounts indeed of tartar are necessary to obtain good results, and that a much smaller proportion of oxalic acid is not only as good but even better than cream of tartar in the case of chrome alum. Experiments by Liechti and Schwitzer have also tended to show the utility of oxalic acid as an assistant in mordanting wool, and its comparatively low price is also favourable. When, however, the attempt is made to study its action more closely by analytical methods, difficulties are at once encountered.

It has been already shown that with increasing amounts of oxalic acid (and the same holds good with tartaric acid), that with the use of 2 mols. oxalic acid to 1 mol. chrome alum dissociation is prevented, and that larger amounts give less favourable results on dyeing. Further it will have been noticed that with the increase of acid used, the differences in colour in the different fibres becomes less and less, that the greatest evenness of colour can be obtained by using the chromic salts of organic acids, and the general appearance and beauty of the dyed colour is thereby improved. Dyeing experiments with azo colours have shown that individual wool fibres differ greatly in their absorbent power for acid.

To throw some light on the subject the most suitable method seemed to be to analyse the spent mordant liquors, and the following experiments are attempts in this direction. In some cases the amount of acid absorbed by

the wool was determined volumetrically; in others we endeavoured to determine the relative amounts of chromic oxide, oxalic acid, and sulphuric acid which must be present in the wool after the mordanting operation. To determine the quantity of these three substances remaining on the wool after washing seemed practically impossible and was not attempted. Sufficiently exact results have unfortunately not been obtained, but it seems evident that a relatively larger proportion of acid than of chromic oxide disappears from the bath, which leads us to the conclusion that the mordanting substance fixed on the wool must be of an acid character rather than a hydrate or basic salt as usually stated in the text-books and technical journals. The titration of a chrome solution containing oxalic acid and the estimation of the chromium itself are very uncertain; oxalic acid as well as alkali seem to be dragged down with the precipitate, so that frequently one may observe on calcining the presence of chromate of soda as well as chromic oxide, and by re-dissolving, reduction, and re-precipitation the loss of a little substance is scarcely to be avoided. It was frequently observed, too, that the oxalic acid dissolved the ferric oxide present in the wool as an impurity, and this of course would contaminate the chromic oxide. Considering these things, and remembering the action of the ammonia given off by the wool, we are far from relying upon the formula given subsequently as representing the true nature of the compound fixed upon the mordanted wool. It may suffice, however, for the present to make the following statements: that when organic acids are used as assistants, no decomposition of the chrome alum occurs, that much less chromium (e.g. 40 per cent.) is fixed than when chrome alum alone is used, and indeed as an acid compound.

*Experiment 30.*—Three fents of wool were mordanted as follows, with—

(a.) 10 per cent. chrome alum and 2 mols. oxalic acid (equiv. to 0.4 grms.  $\text{SO}_3$ ). The spent liquor was titrated with normal NaOH, and showed an absorption of acid equivalent to 0.2744 grm.  $\text{SO}_3$ , i.e., = 0.33614  $\text{H}_2\text{SO}_4$ . This number agrees well with the 0.3332 grm. absorbed by wool when boiled with water containing 10 cc. normal  $\text{H}_2\text{SO}_4$ .

(b.) 10 per cent. chrome alum and 1.5 mols. oxalic acid. The amounts of acid absorbed by the wool in two experiments were:

(1.) 0.2716 gr.  $\text{SO}_3$  = 0.33371 gr.  $\text{H}_2\text{SO}_4$   
(2.) 0.2688 „ „ = 0.32928 „ „

(c.) 10 per cent. chrome alum and 3 mols. sodium oxalate.

The bath contained:  $\text{Cr}_2\text{O}_3$  : 0.10692 grm.  
 $\text{SO}_3$  : 0.20129 „

The wool therefore contained:  
 $\text{Cr}_2\text{O}_3$  : 0.04608 grm.  
 $\text{SO}_3$  : 0.03871 „

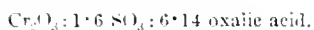
which corresponds to a substance having the following composition:  $\text{Cr}_2\text{O}_3$  : 1.6  $\text{SO}_3$ .

A complete decomposition had not taken place, no doubt because of the need for acid by the wool. In this case, of course, the presence of the  $\text{Na}_2\text{O}$  prevents a large amount of the  $\text{SO}_3$  from being absorbed by the wool, and therefore it remains in the bath.

We have seen above that from chrome alum 0.159 grm.  $\text{SO}_3$  is absorbed by the wool, while here only 0.03871 grm.  $\text{SO}_3$  is taken up, i.e., about one-fourth. In its place, however, oxalic acid has been absorbed, which dissolves the basic sulphate and hence corresponding better results are obtained. We have, namely—

	Chrome Alum alone.	Chrome Alum + 3 mols. $\text{Na}_2\text{C}_2\text{O}_4$ .
In the bath.	14.3 per cent. of the $\text{Cr}_2\text{O}_3$ .	69.8 per cent.
„ wool.	85.7 „ „	30.12 „
„ bath.	33.75 „ of the $\text{SO}_3$ .	87.80 „
„ wool.	66.25 „ „	12.20 „

Calculated for equivalent amounts of  $\text{Cr}_2\text{O}_3$ , the wool absorbs in the case of chrome alum 66.25 per cent. of the  $\text{SO}_3$ , while for chrome alum and sodium oxalate only 34.7 per cent. According to the result of titration, acid equivalent to 0.18972 grm.  $\text{SO}_3$  must have been absorbed; if one subtracts from this 0.3871 grm., there remains the equivalent of 0.14801 grm.  $\text{SO}_3$ , which is equal to 0.233116 grm. oxalic acid. This, combined with the 0.94608 grm.  $\text{Cr}_2\text{O}_3$ , would correspond to the compound:



That the wool has undoubtedly absorbed an acid compound may also be deduced as follows:—The quantity of  $\text{Cr}_2\text{O}_3$  remaining in the bath requires as a salt an amount of acid corresponding to 4.19 cc. normal  $\text{NaOH}$ ; since, however, only 1.332 cc. were used in titration, the bath must, therefore, have contained essentially a basic chromic oxalate, consequently an acid chromium salt must have been fixed upon the wool. Further, the short series of experiments made in connection with the dyeing of series of shades, from dark to pale, show conclusively the truth of the view that acid mordants must be employed. Moreover, the very existence and character of the "single-bath" process for wool has long indicated that the theory of the fixing of the metallic mordants as hydrates or basic salts is untenable. In this connection preliminary experiments have shown that as the amount of mordant is decreased the amount of acid to be used therewith must be proportionately increased, and we might regard a single-bath process as one in which there is a rapid succession of mordanting and dyeing operations recurring in the same bath.

The fact that organic acids are here preferable to mineral acids is probably connected with the chemical constitution of the wool fibre itself. It is interesting that acetic acid, although it prevents the dissociation of the chrome alum, is absolutely worthless as an assistant.

(d.) 10 per cent. chrome alum and 2 mols. oxalic acid.

The bath was found to contain:  $\text{Cr}_2\text{O}_3 : 2.035 \text{ SO}_3 : 0.23 \text{ oxalic acid.}$

The wool was calculated to contain:  $\text{Cr}_2\text{O}_3 : 5.175 \text{ SO}_3 : 6.09 \text{ oxalic oxide.}$

Although in this case the oxalic acid was only calculated and not determined as such, still it is at least certain that in the bath there is relatively too little and in the wool too much  $\text{SO}_3$  in combination with the  $\text{Cr}_2\text{O}_3$ , which points again to an acid compound being on the wool.

(e.) In this experiment an attempt was made to determine the oxalic acid as such, and the following numbers were obtained:—

The total amount of acid absorbed by the wool, in terms of  $\text{SO}_3$ , was according to titration 0.2744 grm. On the wool there was found to be 0.0785 grm.  $\text{Cr}_2\text{O}_3$ , from which it is calculated, expressing the oxalic acid in terms of  $\text{SO}_3$ , that the wool contained the compound  $\text{Cr}_2\text{O}_3 : 6.68 \text{ SO}_3$ .

By gravimetric analysis the bath was found to contain 0.0745 grm.  $\text{Cr}_2\text{O}_3 + 0.08446 \text{ grm. SO}_3 + 0.09513 \text{ grm. oxalic acid}$ ; this amount of oxalic acid is equivalent to 0.6604 grm.  $\text{SO}_3$ , therefore together, the amount is 0.14486 grm.  $\text{SO}_3$ , from which one may calculate that the bath contains the equivalent of  $\text{Cr}_2\text{O}_3 : 3.72 \text{ SO}_3$ .

The wool then contains: 0.0785 grm.  $\text{Cr}_2\text{O}_3 + 6.1556 \text{ grm. SO}_3 + 0.15687 \text{ grm. oxalic acid}$ , the latter being equivalent to 0.6996 grm.  $\text{SO}_3$ , therefore, together the amount is 0.2551 grm.  $\text{SO}_3$ , from which one may calculate that the wool contains the equivalent of  $\text{Cr}_2\text{O}_3 : 6.215 \text{ SO}_3$ . Now by volumetric analysis the acid found was equivalent to 6.68  $\text{SO}_3$ , which is sufficiently concordant, having regard to the difficulties connected with the determination.

Considering the oxalic acid as such, there would, according to the foregoing calculation, be present on the wool the compound:  $\text{Cr}_2\text{O}_3 : 3.78 \text{ SO}_3 : 2.42 \text{ oxalic acid}$ , that is, as previously found, an acid compound; while in the bath the compound would be  $\text{Cr}_2\text{O}_3 : 2.167 \text{ SO}_3 : 1.55 \text{ oxalic acid}$  (= 3.715 acid groups).

In this case 51.3 per cent. of the chromium presented to the fibre was actually fixed on the fibre. The oxalic acid

was determined by permanganate of potash. Not feeling certain about the results given under (d), the analysis given under (e) were repeated.

(f.) 10 per cent. chrome alum and 2 mols. oxalic acid.

In this experiment the spent liquor contained 0.0991 grm.  $\text{Cr}_2\text{O}_3 + 0.1116 \text{ grm. SO}_3 + 0.084 \text{ grm. oxalic acid}$ , i.e., per  $\text{Cr}_2\text{O}_3$  = 3.174 acid groups, or  $\text{Cr}_2\text{O}_3 : 2.154 \text{ SO}_3 : 1.020 \text{ oxalic acid.}$

The wool contained 0.0539 grm.  $\text{Cr}_2\text{O}_3 + 0.1284 \text{ grm. SO}_3 + 0.168 \text{ grm. oxalic acid}$ , i.e., per  $\text{Cr}_2\text{O}_3$  = 8.33 acid groups, or  $\text{Cr}_2\text{O}_3 : 4.55 \text{ SO}_3 : 3.78 \text{ oxalic acid}$ , which is a more acid compound than was found in experiment (e). Here, of the chromium presented to the fibre, only 35.2 per cent. was fixed on the wool. The experiments (e) and (f) differ in so far that in (e) 1 cc. normal  $\text{HCl}$  was added to the bath to neutralise the alkalinity of the wool and the water. In (f) 2.43 cc. normal  $\text{NaOH}$  were required to neutralise the spent liquor, and since the amount of oxalic acid left in it corresponds to 1.33 cc. normal  $\text{NaOH}$ , if this is subtracted there remains 1.1 cc., as having been used in this case for neutralising water and wool.

If instead of free oxalic acid its sodium salt is used as assistant, a compound containing less  $\text{SO}_3$  will be fixed on the wool; 3 mols.  $\text{Na}_2\text{C}_2\text{O}_4 + 1 \text{ mol. oxalic acid}$  along with chrome alum would probably give good results, if one does not wish to employ chromium oxalate, which represents the best mordant.

#### 4. Chrome Alum and Tartaric Acid.

In the first instance a comparison was made of the efficacy of free tartaric acid, cream of tartar, and sodium tartrate, as assistants, with the result, as in the case of oxalic acid, that it is best to use free tartaric acid. This being so it may at once be recommended to practical dyers that, after making an analysis of their cream of tartar, they should add to it the requisite amount of sulphuric acid to liberate the tartaric acid, and for the sake of convenience keep a standard solution of the mixture ready for use.

Experiment 31.—Samples of wool were mordanted as usual with:—

- (1.) 10 per cent. chrome alum + 2 mols. tartaric acid (= 3 per cent.).
- (2.) 10 per cent. chrome alum + 2 mols. cream of tartar (= 3.76 per cent.).
- (3.) 10 per cent. chrome alum + 2 mols. sodium tartrate (= 4.2 per cent.).
- (4.) 10 per cent. chrome alum + 3 mols. tartaric acid.
- (5.) 10 per cent. chrome alum + 3 mols. cream of tartar.
- (6.) 10 per cent. chrome alum + 1 mol. tartaric acid.

Here again it was noticed that the neutral bath was of a yellowish-green colour, the acid baths more and more bluish-green with increase of acidity, indicating that the less acid and neutral baths had a tendency to precipitate a basic chromium salt, while the more acid baths retained the chromium in more stable solution, a fact also indicated by the faint reddish tint of the wool. Even with 3 mols. tartar mordanted wool, when dry, appears grey-yellowish-green, whereas with 3 mols. cream of tartar it has a decided pink-greyish-green colour. The tartar mordanted wool also appears paler than the corresponding tartaric acid mordanted felt, while the one with which sodium tartrate was used is paler still. When the patterns are examined by gas light, as in the case of oxalic acid, those from the more acid solutions have a more pronounced reddish appearance than those from neutral solutions, e.g., neutral chromium oxalate gave a pale heliotrope-grey tint, and only with a certain amount of free acid did the wool appear of a reddish hue; pure chromium tartrate mordanted wool too had only a faint reddish tint. This reddish tint appears more pronounced with oxalic acid than with an equivalent amount of tartaric acid. Already in the experiments previously referred to it was noticed that wool mordanted with  $\text{K}_2\text{Cr}_2\text{O}_7 + 2 \text{ mols. cream of tartar}$  appeared only very slightly of a pink tint, whereas when mordanted with  $\text{K}_2\text{Cr}_2\text{O}_7 + \text{H}_2\text{SO}_4 + 2 \text{ mols. cream of tartar}$  the pink tint became more pronounced. The more the chromium mordanted wool has a yellowish-green hue the worse the results on dyeing and the

more do they approach the "*couleurs alcalins*" of Havrez; with increasing amounts of acid the wool becomes more greyish-green to violet grey-green. It is impossible to fix definitely the best amount of acid to employ, since this depends on the absolute quantity of chrome salt and especially on the amount of water employed. With 10 per cent. chrome alum, for example, 2 mols. tartaric acid were found to be sufficient, with smaller amounts 3 mols. gave better results, but this point will be referred to again later on. Mordanting with 6.75 per cent. chrome alum and tartar, and using 1,000 cc. water, logwood gave a colour of only half the intensity of that given when 600 cc. water were used. Interesting as this fact in itself may be, it renders most difficult, if not impossible, the attainment of positive results in this class of research work. It gives rise also to practical difficulties to overcome which requires the close observation and attention to details. Suppose for example a dyer after much experimenting has devised a very good receipt for a particular mordant, say, for 50 kilos. wool and a corresponding amount of water. If, now, in a subsequent operation only 20-25 kilos. wool are mordanted in the same vessel, *i.e.*, with the same amount of water, and the amounts of mordant and acid are proportionately reduced, he will frequently obtain different results. In this case the remedy would be to use a little more acid, to which no doubt many a practical dyer has been led by careful and intelligent observation even though he has possessed little or no chemical knowledge. Practice is not infrequently in advance of theory, and this has often been the case in connection with dyeing; it is probable that with a little more readiness on the part of dyers in communicating observations regarding defects, or the improvements met with in the course of their daily work, would facilitate the progress of the science connected with dyeing.

The above five woollen fents were dyed in alizarin with the result that the best results were obtained with 2 mols. tartaric acid; 2 mols. cream of tartar gave a slightly paler and less even colour. If, however, 3 mols. cream of tartar (No. 5) are used, the colour is very good, and it is somewhat remarkable that with cream of tartar we may use tolerably large amounts without seriously affecting the colour, *e.g.*, even 6 mols. seem to be still favourable, whereas in the case of tartaric acid, 2 mols. are distinctly better than 3 mols., although if only 5 per cent. of chrome alum is used, 3 are better than 2 mols. Comparing tartaric acid and oxalic acid, 2 mols. of the former give a finer and bluer shade of Bordeaux than 2 mols. of the latter.

An examination of the mordanted material under the microscope gave the following result: slight differences of colour in the different fibres were noticeable in No. 1, but they were less than in No. 6 and 2; little difference existed between Nos. 4 and 5; No. 3 was the worst.

On putting a little unmordanted wool into the waste dye-liquor of No. 1, it acquired a pale pink colour (single bath), which was much paler than was obtained in the analogous experiment with oxalic acid, moreover the mordant bath was most exhausted; neutral and basic baths were always less exhausted.

The amount of acid absorbed by the wool from No. 2 mordant bath expressed in terms of  $\text{SO}_3$  amounted to 0.2744 grms. It may also be added that the less acid the mordant bath is the lower the temperature at which the dyeing of the fibre begins.

**Experiment 32.**—Samples of wool were mordanted as follows, and subsequently dyed with alizarin.

- (1.) Mordant as usual with 10 per cent. of chrome alum + 2 mols. tartaric acid (= 3 per cent.).
- (2.) Boil with 3 per cent. of tartaric acid, then add 10 per cent. chrome and continue boiling.
- (3.) Mordant with chromium tartrate, equivalent to 10 per cent. chrome alum.

The finest and darkest Bordeaux colour was given by No. 3; No. 2 was distinctly better than No. 1, but the difference between the two was less than in the analogous experiment with chrome alum and oxalic acid.

If samples of wool were padded with the following equivalent solutions, then dried and steamed—

- (a.) Chrome alum + 1 mol. tartaric acid.
- (b.) " " + 2 " "
- (c.) " " + 2 mol. cream of tartar.
- (d.) " " + 1 " "

(a.) acquired a greenish violet-grey colour; (b.) a similar tint, although there were evidences of the wool being already too acid; (c.) was also similar to (a.) with a distinct violet tint; (d.) was paler than (b.). When dyed in logwood, unsatisfactory grey shades were obtained, of less value even than the corresponding colours obtained with oxalic acid.

**Experiment 33.**—Wool was mordanted as follows:—

- (1.) 10 per cent. chrome alum + 3 mols. tartaric acid.
- (2.) 10 per cent. chrome alum was precipitated with 6 mols.  $\text{NaOH}$ , and the precipitate redissolved by adding 3 mols. tartaric acid.
- (3.) 10 per cent. chrome alum + 3 mols. sodium tartrate.
- (4.) Chromium tartrate equivalent to 10 per cent. chrome alum.

The appearance of the waste mordant liquors was as follows: No. 1, violet-blue-green; Nos. 2 and 3, slightly turbid and less exhausted; No. 4, clear, violet-green.

The colour of the mordanted wool decreased in intensity in the order 4, 1, 3, 2; No. 4 being a fine dark green, No. 1 a violet-grey. When dyed in alizarin No. 1 gave the finest and darkest colour, bluer in shade than with chromium oxalate, and the dye-bath was well exhausted. No. 1, although apparently well mordanted, gave the palest colour, no doubt because the mordant was already too acid; Nos. 2 and 3 were dark colours, but not very bright, probably the mordants were in these cases not sufficiently acid; No. 2 was somewhat better than No. 3. We have already seen that when using a mixture of chrome alum and sodium oxalate the inter-action under the conditions imposed is not complete. No. 2 is of course really a chromium tartrate prepared in the mordant bath, and ought certainly to give good results if a little more tartaric acid is employed.

**Experiment 34.**—Wool was mordanted with the following:—

- (1.) Chromium tartrate, equivalent to 10 per cent. chrome alum.
- (2.) As No. 1, with addition of 0.272 grm. sodium acetate.
- (3.) As No. 1, with addition of 0.098 grm. sulphuric acid.
- (4.) As No. 1, with addition of 0.150 grm. tartaric acid.
- (5.) Chromium oxalate, equivalent to 10 per cent. chrome alum.

The appearance of the spent mordant liquors was as follows: No. 1, clear, violet-green; No. 2, dark green with violet tint, less exhausted than 1; No. 3, clear, violet-green, not darker than 1; No. 4, similar to 3, or somewhat paler; No. 5, greenish-violet.

The mordanted wool appeared as follows: No. 1, a fine dark green; No. 2, paler green than 1; No. 3, a green paler and greyer than 1; No. 4, the darkest green; No. 5, a greenish-violet-grey.

When dyed with 12 per cent. alizarin (20 per cent. paste) the colours were as follows: No. 1, a fine full Bordeaux; No. 2, about half as deep a colour as 1; No. 3, the depth of colour was between that of 1 and 2, and the hue somewhat redder; No. 4, a better colour than 3, but somewhat paler than 1, and about equal to 5; No. 5, a fine colour, but still not quite equal to 1.

The result of this experiment was to show that the best of all chromic oxide salts as a mordant hitherto examined is chromium tartrate; chromium oxalate is almost as good, and to be preferred in practice because cheaper. The object of using sodium acetate in No. 2 trial was that it might act as a weak alkali perchance the compound  $\text{Cr}_2\text{O}_3 \cdot 3 \text{C}_2\text{H}_3\text{O}_2$  might be too acid, but as noted, the result was very poor.



When dyed with quercitron bark the chromium oxalate mordant gave a full greenish-yellow colour; if the amount of acid was reduced the greenish hue gave place to one more yellowish. A peculiar dark greenish colour was given with chromium tartrate + 3 mols. hydrochloric acid.

The difference in action between chromium sulphate and chromium tartrate is truly remarkable, for whereas the former is almost entirely decomposed, and scarcely mordants the wool at all, the latter mordants it in the best possible manner.

So far as our present knowledge of the wool fibre goes, it seems to us that the cause of this difference lies, first, in the greater affinity of the wool for sulphuric acid, and secondly, that the organic acids are capable of forming much more basic and at the same time soluble compounds than is the case with sulphuric acid.

*Experiment 35.*—This and the following two experiments were made with the view of determining the conditions necessary to obtain a graded series of level shades in a rational manner. Woollen patterns were mordanted in the ordinary manner as follows:—

- (1.) With 20 cc. chromium tartrate, equivalent to 1 gram. chrome alum.
- (2.) With 10 cc. chromium tartrate, equivalent to  $\frac{1}{2}$  gram. chrome alum.
- (3.) With 5 cc. chromium tartrate, equivalent to  $\frac{1}{4}$  gram. chrome alum.
- (4.) With 2.5 cc. chromium tartrate, equivalent to  $\frac{1}{8}$  gram. chrome alum.

The liquors of Nos. 3 and 4, even during the mordanting process, exhibited an alkaline character; No. 4 was pale green and slightly turbid; No. 3 was similar but darker in colour; No. 2 and 1 were clear and violet-green. A drop of methyl-orange solution coloured No. 1 crange-red; No. 2, reddish orange; No. 3, orange; and No. 4, yellowish-orange.

The mordanted fents appeared as follows:—

No. 1, a fine dark green; No. 2, pale green; No. 3 and 4 were yellowish cream colour, and appeared to be deficient in acid.

The fents were dyed with an excess of alizarin in order to saturate the mordants, the amounts chosen, viz., 18, 9, 4.5, and 2.25 per cent., corresponding to half the chromium being fixed on the fibre. Nos. 1 and 2 were deep claret reds, but Nos. 3 and 4 were dull bluish-toned colours which appeared as if the mordant had not been sufficiently acid, and reminded one of the "couleurs alcalins" of Havrez.

Before proceeding farther it seemed desirable to repeat this experiment, but with the modification of adding to each mordant bath 1 cc. normal tartaric acid in order to neutralise the alkalinity of the wool and the water.

*Experiment 36.*—Woollen fents were mordanted as follows:—

- (1.) With 20 cc. chromium tartrate, equivalent to 1 gram. chrome alum.
- (2.) With 10 cc. chromium tartrate, equivalent to  $\frac{1}{2}$  gram. chrome alum.
- (3.) With 5 cc. chromium tartrate, equivalent to  $\frac{1}{4}$  gram. chrome alum.
- (4.) With 2.5 cc. chromium tartrate, equivalent to  $\frac{1}{8}$  gram. chrome alum.
- (5.) With 1.25 cc. chromium tartrate, equivalent to  $\frac{1}{16}$  gram. chrome alum.
- (6.) With 0.625 cc. chromium tartrate, equivalent to  $\frac{1}{32}$  gram. chrome alum.

each with the addition of 1 cc. normal tartaric acid.

The spent mordant liquors of Nos. 1, 2, 3 had an acid reaction, No. 4 was almost neutral, and Nos. 5 and 6 quite so. After dyeing with proportional amounts of alizarin, the waste dye-liquors tested as follows:—No. 1 acid, No. 2 slightly acid, No. 3 very slightly acid, Nos. 4—6 neutral.

The dyed colours were somewhat better than the series in the last experiment, but it was evidently necessary to determine by experiment the amount of acid to be added for each amount of mordant, in order to obtain the best colour. Although the experiments are incomplete and the amounts of acid found necessary only hold good for the

conditions here imposed, and must vary somewhat in practice according to the character of the wool and the water employed, the following details are given to show the method adopted.

*Experiment 37.*—Several fents of wool were mordanted as follows:—

- (1.) With 10 cc. chromium tartrate, equivalent to 0.5 gram. chrome alum.
- (2.) With 10 cc. chromium tartrate + 1 cc. normal tartaric acid.
- (3.) With 10 cc. chromium tartrate. + 2 cc. normal tartaric acid.
- (4.) With 10 cc. chromium tartrate + 3 cc. normal tartaric acid.
- (5.) With 5 cc. chromium tartrate, equivalent to 0.25 gram. chrome alum.
- (6.) With 5 cc. chromium tartrate + 1.5 cc. normal tartaric acid.
- (7.) With 5 cc. chromium tartrate + 3 cc. normal tartaric acid.
- (8.) With 5 cc. chromium tartrate + 4.5 cc. normal tartaric acid.
- (9.) With 2.5 cc. chromium tartrate, equivalent to 0.125 gram. chrome alum.
- (10.) With 2.5 cc. chromium tartrate + 1.05 cc. normal tartaric acid.
- (11.) With 2.5 cc. chromium tartrate + 2.10 cc. normal tartaric acid.
- (12.) With 2.5 cc. chromium tartrate + 3.15 cc. normal tartaric acid.
- (13.) With 2.5 cc. chromium tartrate + 4.20 cc. normal tartaric acid.
- (14.) With 2.5 cc. chromium tartrate + 5.25 cc. normal tartaric acid.

Baths Nos. 4, 8, and 14 each contain as much tartaric acid as is contained in 20 cc. chromium tartrate alone, in the others the amount is conveniently reduced.

The waste mordant liquors tested as follows:—No. 1 slightly alkaline, 2—4 gradually more acid, No. 5 decidedly alkaline, 6—8 clear and apparently exhausted, Nos. 9 and 10 alkaline, 11 and 12 less so, 13 and 14 acid.

The appearance of the mordanted wool was as follows:—Nos. 1—4 green, gradually increasing in depth, No. 5 yellowish, No. 6 similar but more greenish, Nos. 7—8 all a fine green, No. 9 decided yellowish tint, Nos. 9—14 more and more greenish in tint.

When dyed in alizarin Nos. 1 and 2 had an appearance as if alkaline, No. 3 seemed to have had the right amount of acid, although No. 4 was still a good colour, No. 5 was pale and evidently an alkaline colour (Havrez). No. 6 was much better, No. 8 was already worse than No. 7, so that the best amount of acid seemed to be between Nos. 6 and 7, No. 9 was a very poor alkaline colour, No. 10 was little better, Nos. 11 and 12 were slightly better, No. 13 seemed to be of the normal colour, while No. 14 appeared to have been too acid. It may be added that an addition of acetic acid to the dye-bath appeared to act somewhat injuriously if the right amount of acid was present in the mordant bath, the colours being rendered thereby paler.

From this experiment it seems certain that if the dyer desires to work in a strictly rational manner, he must use for decreasing amounts of colouring matter proportionately smaller amounts of mordant, and at the same time add to the mordant bath, with respect to the  $R_2O_3$ , gradually increasing amounts of the assistant acid in order to obtain full bright level colours. The details no doubt require to be worked out in each individual case. In the experiment cited and the conditions observed, the best proportions of acid to use are—

- For 10 per cent. chrome alum about ( $Cr_2O_3$  : 3 mols.  $C_4H_4O_6$ ).
- For 5 per cent. chrome alum about ( $Cr_2O_3$  : 5 mols.  $C_4H_4O_6$ ).
- For 2.5 per cent. chrome alum about ( $Cr_2O_3$  : 8 mols.  $C_4H_4O_6$ ).
- For 1.25 per cent. chrome alum about ( $Cr_2O_3$  : 10 mols.  $C_4H_4O_6$ ).



An exact determination is difficult, because according to the amount of acid present the shade is bluish or reddish, and this variation prevents one from accurately estimating the comparative intensity of colour. To complete the study of this point it would be necessary to determine quantitatively the amount of chromium fixed on the wool in each bath, and from this to calculate the corresponding amount of alizarin to be employed. For a total fixation of 10 per cent. chrome alum one would require to use 36 per cent. alizarin (20 per cent. paste). The 10 per cent. alizarin usually employed in these experiments corresponds fairly well to the amount of chromium fixed in the mordant bath.

From the foregoing result it is evident that the custom in practical receipts of expressing additions of acid to the bath in percentages of the weight of wool used is perfectly justified. In the case before us the condition of the mordant bath to be observed approaches that of constant acidity, and reminds one forcibly of the dyeing of the acid azo colours to which reference has already been made, where, for example, practically the same amount of acid must be used for 3 per cent. and 0.3 per cent. colouring matter.

On examining under the microscope the dyed fents Nos. 1, 5, 9, mordanted with decreasing amounts of chromium tartrate only, an appearance was observed which seems to be very instructive. In addition to colourless fibres there were others presenting all shades, varying from a pale to a dark brownish pink; sometimes even the same fibre showed equally great differences, while others were dyed throughout. The fents mordanted with the addition of acid and then dyed had a much better appearance, although even in these all the fibres were not equally dyed. It was remarked, too, that the pink was of a yellower hue. No formation of precipitated lake could be noticed in either case. With the chromic oxide mordant hitherto examined, we have not noticed the production of an absolutely level colour (*i.e.*, microscopically examined), and although the tartrate and oxalate give quite satisfactory dyes, bichromate of potash gives better results in this respect.

In a subsidiary experiment bearing upon this point, 10 grms. wool were boiled with 600 cc. water and equivalent amounts of chrome alum (0.28 grms.) and alizarin (1 grm., 20 per cent. paste), with the result that a somewhat deep yellow coloured and turbid liquor was obtained, which was almost neutral. The wool had, therefore, practically absorbed the whole of the  $\text{SO}_3$  ( $=0.0672$  grms.), and thus permitted the alizarin to combine with the chromic oxide. The colour thus obtained was a dull uneven Bordeaux, with yellow and pink specks, corresponding to the amount of sulphuric acid which the individual fibres had absorbed. A much better result was obtained by using chromium tartrate corresponding to the 0.28 grms. chrome alum.

In another experiment, in which the alizarin was dissolved in an equivalent amount of ammonia, a poor, bare colour was obtained, of scarcely half the depth of the previous one, a result which shows how greatly the wool needs acid. If sodium alizarate had been used instead of the ammonium salt, probably no reaction at all would have occurred.

**Experiment 38.**—Before recognising chromium tartrate and oxalate as the best chromic oxide mordants, this experiment was made in order to see if an increase of oxalic acid was necessary with a small amount of chrome alum (4 per cent.), and is mentioned here because it also bears upon the point examined in the foregoing experiments.

Wool was mordanted with—

- |      |  |
|------|--|
| (1.) | 4 per cent. chrome alum + 1 per cent. oxalic acid. |
| (2.) | " " " + 1.5 " "                                    |
| (3.) | " " " + 2.0 " "                                    |
| (4.) | " " " + 2.5 " "                                    |

The mordanted fents were afterwards dyed with excess of logwood (40 per cent.). No. 1 was a very poor colour, almost as bad as if 10 per cent. chrome alum had been used; No. 2 was a little better, No. 3 the best, and No. 4 slightly worse than 3. Now, when using 10 per cent. chrome alum, experiment showed that one must use at least 2 mols.  $=2.52$  per cent. oxalic acid, so that in proportion

one might have expected that for 4 per cent. oxalic only 1 per cent. chrome alum would be needed, whereas this experiment shows that double this amount gives the best colour, an amount which corresponds to  $\text{Cr}_2(\text{SO}_4)_3$ ; 4 mols.  $\text{C}_2\text{H}_2\text{O}_4$ , and which for 10 per cent. of chrome alum would be equivalent to 5 per cent. oxalic acid. When microscopically examined, No. 1 was found to exhibit both white and dull blue fibres; No. 2 was much better, No. 3 better still, but showing both pale and dark blue fibres; No. 4 was worse than 3, and about equal to 2. With too much oxalic acid the colour is a reddish grey.

This experiment shows clearly that the wool fibre demands the use of a certain amount of acid, which must be determined for each special case if one wishes to avoid the production of colours which correspond to Hayez's "couleurs alcalines."

**Experiment 39.**—Six fents of wool (Nos. 1 to 6) were mordanted respectively with 10, 5, 2.5, 1.25, 0.625, and 0.3125 per cent. chrome alum, along with correspondingly decreasing amounts of oxalic acid, *viz.*, in the proportion of 1.5 mols. per 1 mol. chrome alum. Six other fents (7 to 12) were mordanted with the same amounts of chrome alum as above, but each with equal quantities of oxalic acid (0.189 grm.) *i.e.*, constant acidity. The latter gave a fine series of bright colours; the former were dull.

A repetition experiment, in which the fents were dyed with logwood, gave similar results; proportional acidity gave unpleasant grey colours, constant acidity fine blue shades. The results of experiment 37 showed, however, that with small amounts of chrome alum one ought not to use as much acid as for 10 per cent. chrome alum. In another experiment it was found that for 2.5 per cent. of chrome alum, 0.126 grms. oxalic acid ( $=4$  mols.) ought to be used.

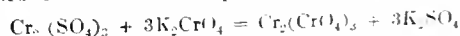
All the experiments on this point showed, therefore, that for decreasing amounts of chrome alum one ought to use relatively increasing amounts of oxalic acid, and this explains most satisfactorily why in practice the same amount of mordant is frequently employed for different amounts of dye-stuff, since with proportional reduction of mordant there would have been too little acid in the bath. This custom is nevertheless not strictly rational, in so far that the necessary acidity for the paler shades should rightly be produced by additions of the *assistant acid* rather than by excess of mordant, although in practice the question of economy may intervene.

The need for acids by the wool is also well illustrated in the case of single-bath colours, where for pale shades it is customary to reduce only the amount of colouring matter and keep the rest of the ingredients constant in amount. In a single bath, yellow, for example, in which  $\text{SnCl}_2$ ,  $\text{SnCl}_4$ , oxalic acid, and flavin are employed, a proportional reduction of all the constituents for a pale shade would give a poor colour, but a bright colour is obtained if only the flavin is reduced. In the latter case an excess of mordant is of course used, but its employment preserves the purity and beauty of the pale colour.

### 5. Chrome Alum and Chromic Acid.

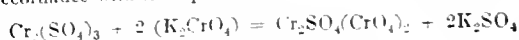
In recent years three chromium-chromate mordants have been recommended for cotton and silk by Von Gallois and introduced to the trade as chrome mordants, GA 1, GA 11, GA 111, by Messrs. Meister, Lucius, and Brüning of Hoechst. It seemed desirable, therefore, to try similar compounds for wool. Experiments were made with mixtures of chrome alum and bichromate of potash.

The simplest method to obtain a chromium chromate seemed to be to follow the equation—



On mordanting wool with the above mixture, equivalent to 3 per cent.  $\text{K}_2\text{Cr}_2\text{O}_7$ , the bath quickly became turbid and the experiment was not continued.

Another experiment in which the proportions were in accordance with the equation—

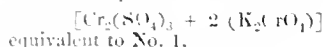


showed this mixture also to be of no use.

*Experiment 40.*—Six woollen fents were mordanted with

(1.) 3 per cent.  $K_2Cr_2O_7$  + 1 mol.  $H_2SO_4$ .

(2.) Chromium sulphate chromate—



(3.) As No. 2 + 1 mol. oxalic acid.

(4.) " + 2 " "

(5.) " + 3 " "

(6.) " + 4 " "

Bath No. 2 decomposed after 10 minutes, No. 3 after 25 minutes, No. 4 after 1 hour, Nos. 5 and 6 remained clear. The appearance of the patterns during the mordanting was as if the oxalic acid had at first simply liberated the chromic acid, which was then taken up by the wool. On dyeing the patterns in quercitron bark No. 1 gave by far the best result, a full brownish orange; the large amount of oxalic acid gave better results than the smaller ones, the shade of orange being greener proportional to the reduction which had occurred. After No. 1 the next best mordant was No. 5.

*Experiment 41.*—This was a repetition of the last experiment, but using tartaric instead of oxalic acid. Here the reduction was still greater, and the greenish orange shades were somewhat fuller, but No. 1 was still the best colour.

*Experiment 42.*—Here woollen fents were mordanted with a mixture of equal mols. chrome alum and chromic acid anhydride both alone and with the addition of increasing amounts of oxalic acid, thus:—

(1.) 3 per cent.  $K_2Cr_2O_7$  + 1 mol.  $H_2SO_4$ .

(2.) One mol. chrome alum + 1 mol.  $CrO_3$  (the Cr equivalent to that in No. 1).

(3.) As No. 2 + 1 mol. oxalic acid.

(4.) " + 2 " "

(5.) " + 3 " "

(6.) " + 4 " "

The result was that although here there was an acid compound present, No. 2 bath decomposed after 20 minutes, No. 3 after 25 minutes, No. 4—6 remained stable. When dyed in quercitron No. 1 appeared the best, No. 5 the next, and No. 6 the palest.

*Experiment 43.*—This was a repetition of the last experiment, using tartaric instead of oxalic acid. Smaller amounts of tartaric acid sufficed to prevent dissociation than in the case of oxalic acid, and the shades were paler and greener. No. 1 was again the best colour.

*Experiment 44.*—In this experiment similar proportions were employed as in No. 42, except that 1 mol. chrome alum + 2 mols.  $CrO_3$  were employed, the amount of chromium being equivalent to that contained in 3 per cent.  $K_2Cr_2O_7$ , with which 1 mol.  $H_2SO_4$  was used. In this case bath No. 2 decomposed after 20 minutes, No. 3 showed a tendency to decompose after 30 minutes, the rest remained clear. With the increase of oxalic acid the yellowish-green colour of the bath gradually gave place to a greenish-violet. In accordance with the fact that here the mordant was richer in  $CrO_3$ , the dyed patterns appeared better than the corresponding ones of experiment 42. No. 1 pattern was still the best, next came No. 4 with 2 mols. oxalic acid.

*Experiment 45.*—This was a repetition of No. 44, using tartaric acid, with the result that the shades were green and fuller. No. 1 was the best.

*Experiment 46.*—This was a similar experiment, but made with the use of 1 mol. chrome alum + 3 mols.  $CrO_3$ , and various amounts of oxalic acid as before. No new feature was developed, and the fact was merely emphasised that the colours improved in proportion as the  $CrO_3$  in the mordant decreased and the  $CrO_3$  increased, but in no case was the result as good as with chromic acid alone. The effect generally gave one the impression that the mordants were simply mixtures.

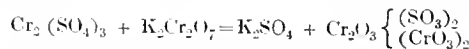
*Experiment 47.*—This was a repetition of No. 46 with tartaric acid. Here, too, baths Nos. 2 and 3 decomposed, the rest remaining clear. The waste liquor of No. 1 was pale yellow, No. 2 dull yellow, Nos. 3—6 the colour gradually

became greener and the baths appeared to be better exhausted. The mordanted and also the dyed patterns were greener than when oxalic acid was used, which was not able to reduce all the chromic acid until 6 mols. were employed. No. 1 pattern had invariably the best colour. The use of solutions of chromium chromates as mordants for wool seems unpromising. Useful results are only obtained when fairly large amounts of organic acid are used as well, in which case there is no advantage over bichromate of potash. It is well to note that the wool mordanted in the manner described always seemed to have a pleasant soft feel.

In a subsidiary experiment a piece of wool was padded with a solution containing 1 mol. chrome alum + 1 mol.  $K_2Cr_2O_7$  + 2 mols. oxalic acid, dried and steamed; it thus acquired a dull olive grey colour, so that here too the reduction was incomplete. Total reduction occurred, however, if instead of oxalic acid 2 mols. tartaric acid were used, and the wool acquired a violet-greenish-grey colour rather than green, indicating that a chromic oxide salt was present in the fibre rather than chromic hydrate.

In comparing the two methods of fixing the mordant by steaming and by boiling it was generally found that in the former the presence of less acid was needed than in the latter, evidently because of the greater concentration of the mordant solution in the former case. If in the last-mentioned experiment the acid was entirely omitted, the fent acquired a brownish-green colour, pointing to the presence of both  $Cr_2O_3$  and  $CrO_3$ , and there was apparently total fixation of the mordant.

*Experiment 48.*—This experiment was suggested by the foregoing observation. Fents were mordanted in the usual manner with a mixture of chrome alum and bichromate of potash, equivalent to 3 per cent.  $K_2Cr_2O_7$ , and which we might consider as a chromium-chromate-sulphate presented to the wool, thus:—



The different fents were mordanted with—

(1.)  $Cr_2(SO_4)_3 + K_2Cr_2O_7$ , equivalent to 3 per cent.  $K_2Cr_2O_7$

(2.) As No. 1 + 2 mols. oxalic acid.

(3.) " + 3 " "

(4.) " + 4 " "

(5.) " + 5 " "

(6.) " + 6 " "

Bath No. 1 had a yellowish colour reminding one of  $K_2CrO_4$ , No. 2 similar, No. 3 a peculiar dichroic yellow-violet appearance, Nos. 4, 5, 6 all greenish violet. At 70° C. No. 1 became turbid, the others remained clear.

The mordanted fents had the following appearances: No. 1 pale reseda colour, Nos. 2—4 rapidly less yellow and more green, No. 5 a pleasing greyish green, No. 6 the most pronounced green of the series.

The samples were dyed with 30 per cent. logwood, which gave somewhat deep black-blue colours, Nos. 2 and 3 being the best, with more than 4 mols. oxalic acid the colours became much paler. The amount of water requisite for the mordanting process by the boiling method demands, therefore, the use of a little more acid.

In this connection two other experiments may be cited here. Wool was mordanted with chromium equivalent to 3 per cent.  $K_2Cr_2O_7$  by means of the following mixtures:—

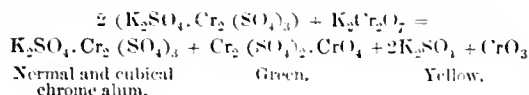
(1.) 2 mols.  $Cr_2(SO_4)_3$  + 1 mol.  $K_2Cr_2O_7$ . ( $=K_2SO_4 + 5SO_3 + 2(Cr_2O_3 \cdot CrO_3)$ ).

(2.) 1 mol.  $Cr_2F_6$  + 3 mols.  $K_2Cr_2O_7$ . ( $=6KF + Cr_2O_3 + 6(CrO_3)$ ).

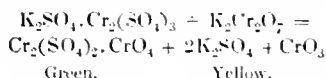
Of these the first seems to have been already tried by Lepetit; both are quite arbitrary equations, and are only provisionally regarded as possibly occurring, e.g., the first would only take place if the wool absorbs the 5 mols.  $H_2SO_4$ ; the second was merely suggested by a desire to render the  $HCl$  harmless, and to obtain a more neutral mordant. The amount of  $SO_3$  to be absorbed in the first case would be 0.16 gms. less, therefore, than is possible. The intention was to find a mordant which would be taken up by the wool as completely as possible.

No. 1 bath was barely acid, and was exhausted apparently very well, the wool seeming to attract merely chromic acid in the first instance, but becoming browner at a later stage, as expected, by the fixing of  $\text{Cr}_2\text{O}_3 \cdot \text{CrO}_3$ . After  $\frac{1}{2}$  hour at 71° C. the bath became slightly turbid through the precipitation of basic chromic sulphate, but on boiling it again became clear. No. 2 bath was slightly acid to litmus, became turbid after 25 minutes at 60° C., but afterwards cleared again; it was of a pale yellow colour, and seemed fairly well exhausted, the wool acquiring the colour of  $\text{Cr}_2\text{O}_3 \cdot \text{CrO}_3$ . When dyed in alizarin pleasing claret-reds were obtained, No. 1 slightly paler but brighter and clearer than No. 2. Slight additions of acid to the mordant bath would probably have given better results even, and further experiments in this direction might be valuable, especially since the mordant baths are so well exhausted.

In order to gain some idea of the nature of the interaction of 2 mols. chrome alum and 1 mol. bichromate of potash a solution of the mixture was allowed to evaporate slowly over sulphuric acid. At first yellow prismatic crystals were formed, in colour more like  $\text{K}_2\text{Cr}_2\text{O}_7$  than  $\text{K}_2\text{Cr}_2\text{O}_7$ ; then appeared violet octahedra of chrome alum, some showing a tendency to become cubical; curiously enough some violet red prismatic crystals were also noticed, as well as colourless dendritic crystals. Finally a deep grass-green indistinctly crystalline mass was produced, evidently a mixture; here and there it was turbid, probably from the presence of  $\text{Cr}_2\text{O}_3 \cdot \text{CrO}_3$ . The amount of each substance was too small to attempt separation and analysis. Possibly the chrome alum by loss of  $\text{H}_2\text{SO}_4$  had become basic and liberated chromic acid, and also furnished  $\text{K}_2\text{SO}_4$ . The grass-green mass might simply be a mixture of  $\text{Cr}_2(\text{SO}_4)_3$  and chromic acid, and the yellow crystals  $\text{K}_2\text{SO}_4$  contaminated with a little chromic acid. Formation of potassium trichromate might also be possible. A simpler and possibly truer explanation may be represented by the following equation:—



If a similar evaporation experiment be made with equal mols. of chrome alum and  $\text{K}_2\text{Cr}_2\text{O}_7$  one obtains at first, in addition to undecomposed chrome alum, orange yellow crystals and a brown resinous mass insoluble in water ( $\text{Cr}_2\text{O}_3 \cdot \text{CrO}_3$  (?)). If the mixture is repeatedly mixed with water and evaporated again and again, there remains finally a brown mass, which, when dissolved in water, separates into an acid yellowish-green part (a solution containing chromic oxide and chromic acid), and an insoluble portion consisting of  $\text{Cr}_2\text{O}_3 \cdot \text{CrO}_3$ , possibly according to the following equation:—



Of these products the first by loss of  $\text{H}_2\text{SO}_4$  on dilution with water would yield an insoluble compound.

### C. Chromium Chloride.

Although the behaviour of this salt towards wool has already been referred to, one or two experiments made with it with the addition of oxalic and tartaric acid remain to be very briefly described.

**Experiment 49.**—Samples of wool were mordanted with the following:—

- (1.)  $\text{Cr}_2\text{Cl}_6$  (equivalent to 10 per cent. chrome alum).
- (2.) " + 1 mol. oxalic acid.
- (3.) " + 2 " "
- (4.) " + 3 " "
- (5.) " + 4 " "
- (6.) " + 5 " "

With the increased additions of acid the baths became more and more exhausted. In the mordanted condition No. 1. fent had a cream colour, Nos. 2 and 3 more greyish, Nos. 4—6 more and more greenish-grey, No. 6 appearing

very well mordanted indeed. When dyed with alizarin, however, No. 1 with 1 mol. oxalic acid gave the best colour, while No. 1 scarcely dyed at all.

**Experiment 50.**—In this trial the following mordants were used:—

- (1.)  $\text{Cr}_2\text{Cl}_6$  (equivalent to 10 per cent. chrome alum).
- (2.) " + 2 mols. tartaric acid.
- (3.) " + 3 " "
- (4.) " + 4 " "
- (5.) " + 5 " "
- (6.) " + 6 " "

All the baths remained clear; No. 1 retained its original colour, Nos. 2—6 ranged gradually from red green to pure green, and became more and more exhausted, so that No. 6 was finally almost colourless. In the mordanted condition No. 2 appeared greenish-grey, No. 3 similar, but deeper in colour, No. 4—6 pure green. When dyed in alizarin cyanin No. 1 gave a very poor colour, No. 2 the best, while Nos. 3—6 gave paler colours, although from the appearance of the mordanted fents a better result even than that of No. 2 was to be expected. No doubt this defect is due to the increased amount of acid absorbed by the wool, and would be remedied by increasing the amount of calcium acetate added to the dye-bath.

In this experiment the addition of 2 mols. tartaric acid gave the best result, and corresponding to this, in another experiment, 4 mols. cream of tartar gave the best colour.

It is well to emphasise the fact that the behaviour of chromium chloride towards wool when used alone is very different indeed from that of chrome alum; with chrome alum nearly the whole of the chromium disappears from the bath, whereas with chromium chloride the opposite is the case: the major portion remaining in the bath and scarcely mordanting the wool at all.

Chromium nitrate behaves like the chloride, yielding no result except with the use of organic acids, even though there is no dissociation of the salt.

In the case of cotton chrome alum and even chromium acetate give poor results, while the chloride and nitrate, especially in the form of basic salts, are very satisfactory. Witt has also referred to the unsuitability of chromium chloride as a mordant for wool.

### D. Chromium Fluoride.

In recent years this salt has been much recommended as a mordant for wool, and is considered to give up to the wool pure chromic hydrate. Some employ along with it, as an assistant, cream of tartar, others oxalic acid, e.g., 4 per cent. chromium fluoride and 2 per cent. oxalic acid.

**Experiment 52.**—Wool (10 grms.) was mordanted in the ordinary manner with 4 per cent. chromium fluoride and 600 cc. water, previously adding 1 cc. normal HCl to neutralise the alkalinity of the water and the wool.

As in the case of chrome alum the bath decomposed already below 100° C., and the mordanted wool had a yellowish-green colour. The waste liquor required 4.4 cc. normal NaOH, i.e., much more than chrome alum, from which one may calculate that only 33.42 per cent. of the salt had been decomposed. These facts show that like chrome alum, chromium fluoride also dissociates, and this accounts for the necessity of employing organic acids along with the fluoride, a point which is distinctly recognised in practice, and we may reasonably ask then, does the fluoride possess, after all, any real advantage over chrome alum? When a chromic oxide mordant is required, one may obtain similar results by using chrome alum along with 2 mols. tartaric or oxalic acid, or better still to use chromium tartrate or oxalate.

If one considers that it will scarcely be necessary to use more chromium oxalate than is equivalent to 4 per cent. chrome alum, surely with the present low price of oxalic acid the cost of chromium oxalate need not be reckoned as an impediment against its general employment, e.g., with alizarin colours.

### Conclusion.

The nature of the reactions which occur when wool is mordanted with solutions of metallic salts has long been the subject of comment and discussion by various writers on dyeing. Gouffreville in his "*L'art de la teinture des laines*," (1849), says: "The mordants are for the most part basic salts: with the use of cream of tartar there is fixed on the fibre a tartrate of the metal, and thus tartaric acid enters into the composition of the colour lake and the defect of rubbing-off is avoided. Acids increase the power of diffusion and lessen the affinity of the wool for the mordant."

Road and Thénard (*Ann. de Chimie*, 74, 267) concluded that when wool is immersed or even boiled in a solution of alum, the latter is not decomposed but absorbed by the wool unchanged, and can be removed therefrom by boiling several times with water. When wool is boiled with cream of tartar these chemists state that the fibre takes up tartaric acid, leaving in the bath neutral tartrate of potash. When boiled with alum and tartar they suppose that the wool may contain undecomposed alum as well as the tartrates of alumina and potash and free tartaric acid.

Chevrel found that wool absorbed alum from its solution, and retained the unchanged salt or some other aluminium compound not removable by washing.

Bolley states that alum solution gives up a sub-salt to the fibre steeped in its solution, leaving the latter more acid.

Girardin, in his "*Leçons de chimie élémentaire*," IV., p. 410, 6th edit., says: "It is certain that mordants and colouring principles react chemically upon each other to produce insoluble coloured compounds or lakes, which permeate wool and silk throughout their mass, whereas in the case of vegetable fibres they remain on the external surface, and although the part played by the fibre is not definitely understood, one is disposed to admit that its rôle is purely physical."

Schützenberger ("*Traité des matières colorantes*, I., p. 149) says: "Many salts, among others alums, are precipitated from their solutions by wool and retained with energy by virtue of some special attractive force."

Nietzki, in his *Chemie der organischen Farbstoffe*, p. 13, (p. 15 of English translation), says: "This peculiar property of dyeing on metallic mordants has as yet had no satisfactory explanation. If cotton mordanted with alumina or oxide of iron is placed in an alizarin bath, the lake is precipitated and enters into intimate combination with the fibre. It is probable, however, that the author is here referring to the different behaviour of the two classes of oxides  $RO$  and  $RO_2$  than to the real nature of the dye upon the fibre."

The views, relative to the theory of mordanting, to which our experiments lead us may be briefly summarised as follows:—

The necessity of employing acids, and preferably organic acids, points undoubtedly to the prevention of a precipitation in the fibre of oxide or a basic salt. We are driven to the conclusion, therefore, that during the mordanting process with metallic salts, the wool does not attract either oxides or basic salts, but, on the contrary, acid salts, and we refer here to compounds of the sesquioxides only. Primarily the wool seems to exert an attraction for the acid of the mordanting salt, and to prevent the otherwise inevitable precipitation of a basic salt the presence of an organic acid is beneficial. Precipitation by the ammonia given off through decomposition of the wool is prevented in like manner. The appearance of a well mordanted wool, too, does not point to the presence in the fibre of a hydrate but a salt.

Although during the process of dyeing mordanted wool the bleeding out of acid into the dye-bath was invariably noticed, it is not unreasonable to assume, until evidence to the contrary is adduced, that at least a portion of the acid remains in combination with the coloured body on the wool, a view which seems to be supported by the existence of the single-bath method of dyeing where the solutions are invariably acid.

It appears to us very probable that when wool is mordanted with a metallic salt, both the amido and carboxyl groups of the fibre come into play, the former attracting the

acid of the mordanting salt while the latter attracts the base, the end result being that an acid salt is fixed in the fibre. Excess of acid or the presence of organic acids is necessary, as already stated, to prevent the more or less superficial precipitation of a basic salt, and the mordanting salt remains sufficiently long in solution for it to penetrate the fibre till a wool compound is formed which is thus equally distributed throughout the mass of the fibre. We may regard the wool fibre as being more or less analogous to the cotton fibre when prepared with oil by the Turkey-red process; here fibre and fixing agent for the mordant are separate, but in wool these two elements are united in one and the same substance, namely, the fibre itself, so that in alizarin red dyed wool we have, a complex lake in which the fibre itself is a constituent element.

Compared with studies in cotton dyeing, those in wool dyeing are much more difficult, because it is not possible to repeat the reactions apart from the fibre as in the former case. In dyeing cotton the fibre appears simply to be the carrier of the metallic hydrate or insoluble basic salt, and this both in the beaker and on the fibre will combine with the colouring matter. With wool, however, it is quite different, for example, in the single-bath method. Here we employ together, the metallic salt, organic acid, and colouring matter. The same mixture may be boiled for several hours apart from the fibre, but no reaction ensues, and no colour is produced, not even when calculated amounts of sodium hydrate are added. Even when wool is present no lake is at once formed, and the dye-bath does not appear to contain the colour as such, but gradually as the temperature rises the colour is developed and indeed within the fibre.

We can scarcely claim to have fully proved the truth of the views just now expressed, for there is no doubt that the reactions which have occupied our attention are of a complex character, but we trust that the various observations recorded throughout the paper may be useful to others working on the same subject, and that our communication, so far as the theory of mordanting wool is concerned, may be regarded as an attempt to solve experimentally a very difficult problem.

### DISCUSSION.

MR. RAWSON thought it was not possible to determine the amount of acid taken up by the fibre by estimating the acid left in the bath; it could not well be determined directly, however, as the amount of sulphur it contained was but small relatively to that contained in the wool itself. Was the wool washed thoroughly before its acidity was determined? And was it possible to distinguish between the acid present as such in the wool and that present in combination with chromium oxide? It was said that a non-oxidising mordant was desirable; potassium bichromate, however, would not destroy indigo, although chromic acid would. And the desirability had been emphasised of a mordant which could be completely exhausted, and not merely, like bichromate, to the extent of one-third; this was not practically of much importance, as the bichromate bath could be used over and over again, and thus nearly exhausted, particularly when acid was added from time to time. The bad results obtained on dyeing the wool mordanted with chrome alum was, as one would expect, due to an irregular absorption of the dye by the wool. He thought that the ammonia, which was found when wool was boiled, might have played a part in the formation of the precipitate in the dye-bath, and so might any soap or alkali still present in the wool. What were the dyes that had been experimented with.

MR. RICHARDSON thought it very desirable to ascertain the exact amido character of wool; and Prof. Hummel had stated that the ammonia liberated from the wool played some part in the mordanting process; the fact that it yielded much ammonia when boiled with caustic potash or baryta showed that it must be rich in groups of an acid-amide character. He thought it would be easy to ascertain if the acid existed in the free form in the fibre; if so, the wool would dye blue with Congo red. If not it might have formed ammonium sulphate with ammonia derived from the

amido groups, and would be closely retained between the septa of the porous fibre; and in this case its amount might be determined by treating with cold potash solution and estimating the ammonia formed by Nessler's test, and any deficit from the amounts equivalent to the sulphuric acid in the fibre would show that some of the acid must be present in the free state. Again, if free sulphuric acid were present, its presence would be indicated by a blackening of the wool when dried. He would like to see a better proof of the presence of free acid in the fibre.

Dr. CONEX suggested that the decomposition of the chrome-alum solution by the wool fibre might be due to two causes, firstly, to dialysis of the salt through the fibre, in which case the sulphuric acid would pass through and a basic sulphate be left behind, and, secondly, to absorption of the sulphuric acid thus set free by certain constituents of the wool. This would account for the gradual precipitation of the basic sulphate as the bath was heated up, and the absence of free sulphuric acid in the bath.

It would be interesting to try whether from a solution of chrome alum in a dialyser, sulphuric acid would diffuse out, leaving a basic sulphate of chromium, which would be precipitated on boiling. If hydrochloric acid had little affinity for the wool fibre this would account on the above hypothesis for the absence of mordanting properties of chromic chloride. In what form was the sulphuric acid in combination with the fibre? He thought that the cause of the colour changes on heating chrome alum solution might be other than that suggested by the authors.

Mr. A. G. PERKIN asked if any experiments had been made with Vignon's amido-cellulose, a substance prepared by heating cotton with ammonia under pressure, and having considerable resemblance to wool. Probably a study of its reactions with mordants would throw further light upon the results under discussion.

Prof. HUMMEL, in reply, said that the experiments recorded in the paper were carried out by Mr. Liecht. The wool used had been thoroughly washed, and the amount of sulphuric acid required to neutralise the alkali in it had been determined. Any such impurity was certainly not the cause of acid absorption by the fibre. The only way to distinguish the acid fixed as such from that combined with chromium as a basic salt was by repeated boiling or prolonged steeping in water, when the acid present as such would slowly "bleed" out. It was true that bichromate did not destroy indigo, but with more sensitive colours, such as alizarin-blue, some other non-oxidising mordant was required. It was also true that it was not absolutely necessary that a bichromate bath should be exhausted, but, after replenishing and using it once or twice, the dyer no longer knew how much chromium it contained, and so could not work in a scientifically correct manner. The dyes used were chiefly logwood, quercitron bark, alizarin-blue, and alizarin. Some of the sulphuric acid absorbed by the fibre would no doubt be neutralised by ammonia given off from the wool. The wool had not been dried to see if blackening ensued. With respect to the cause of the colour changes exhibited by chrome-alum solution on heating, he had merely quoted the opinion of others. Mr. Perkin's suggested experiment had not been tried, the experiments having been concluded before the appearance of Vignon's paper. He had recently compared the behaviour of cotton treated with albumin with that of wool, and found them to be generally similar; for example, the cotton thus treated could be dyed a cochineal scarlet in single bath, after the manner adopted for wool.

Mr. H. R. PROCTOR said that hide fibre would absorb either acid or alkali from dilute solution, together with much water which swelled the fibre, but either could be removed by sufficient washing, so that chemical combination could hardly have occurred. Sodium chloride reduced the swelling produced by acid, and at the same time dehydrated the fibre to a much greater extent than when acid was not present.

As regards the absorption of aluminium salts, the researches of Reimer, conducted much on the lines of those

just described by Prof. Hummel, had shown that from alum, aluminium sulphate was absorbed, either as such or in a slightly basic form.

An interesting process of leather manufacture was now largely employed in the United States in which the active agent was chromic acid, which was afterwards reduced on the fibre by sulphurous acid. The leather produced was pale green, and extraordinarily resistant, even to boiling water. By means of chrome alum a leather could be produced precisely similar in appearance, but no more resistant to boiling water than ordinary alumed leather, while by using a basic chloride it was stated that the result was similar to that produced by reduction on the fibre.

## Scottish Section.

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The names in italics are those of Members of Committee who retire at the end of the current session.

The following have been elected to fill the vacancies, and will take office in July next:—*Chairman*: R. Irvine. *Vice-Chairman*: J. Clark. *Committee*: R. Cox, C. A. Fawcett, D. Harris, E. Ostler, R. Pullar, and H. Rose.

## SESSION 1893-94.

Glasgow:—

May 1st.—

Mr. R. R. Tatlock and Mr. R. T. Thomson. "On the Suitability of Water for Domestic Purposes as shown by Chemical Analysis."

Discussion, "The Production of Gas from Paraffin Oils" (this Journal, 1894, 241-247).

Meeting held in the New University Buildings, Edinburgh, on April 3rd, 1894.

MR. R. IRVINE IN THE CHAIR.

## THE EFFECT OF THE SALINE CONSTITUENTS OF WATER ON THE CHARACTER OF BEER.

BY J. B. READMAN, D.S.C., F.R.S.E.

IN a recent\* treatise on the Science of Brewing, the following passage occurs:—

"Having now considered the classes of water which are in general use, it will be well before describing their treatment in detail, to survey the present state of our knowledge concerning the influence of the saline constituents in relation to the brewing process. It is a somewhat humiliating fact that our knowledge on this important matter is still pretty well limited to the practical teaching of the brewhouse experience." Again, in the Canon lectures on Brewing delivered 20 years ago (1874) by Dr. Charles Graham, it

\* A Text Book of the Science of Brewing, by Morris and Morris (E. and F. N. Spon).

is stated:—"There is a remarkable discrepancy of opinion with reference to the influence of these chlorides of sodium, potassium, magnesium, calcium, and so on, and their sulphates, among the brewers of Europe. If you ask an able brewer in Germany, he will tell you he looks on these mineral salts as bad, but if you ask a Burton brewer, or many English brewers, they will tell you that they look upon them as being almost the sheet anchor of their pale ale system. What then is the right action of these mineral salts in the mashing process?"

It is with the object of endeavouring to throw some light on this important subject that the following experiments, 13 in all, have been undertaken.

The 13 mashings, boilings, and fermentations were all performed with the same quality and quantity of malt, hops, and yeast. There were 12 artificially prepared waters used, beginning with distilled water alone, as the first of the series, while the 13th was carried out with Edinburgh gravitation water. In the 12 cases the various salts were dissolved in distilled water, making up the set of experiments as under:—

- No. 1. Di-stilled water.
- No. 2. Distilled water, containing carbonate of lime in  $\text{CO}_2$  equal to 50 grains  $\text{CaCO}_3$  per Imperial gallon.
- No. 3. Distilled water, containing chloride of calcium equal to 50 grains  $\text{CaCl}_2$  per Imperial gallon.
- No. 4. Distilled water, containing sulphate of lime equal to 50 grains  $\text{CaSO}_4$  per Imperial gallon.
- No. 5. Distilled water, containing carbonate of magnesia in  $\text{CO}_2$  equal to 50 grains  $\text{MgCO}_3$  per Imperial gallon.
- No. 6. Di-stilled water, containing chloride of magnesium equal to 50 grains  $\text{MgCl}_2$  per Imperial gallon.
- No. 7. Distilled water, containing sulphate of magnesia equal to 50 grains  $\text{MgSO}_4$  per Imperial gallon.
- No. 8. Distilled water, containing chloride of sodium equal to 50 grains  $\text{NaCl}$  per Imperial gallon.
- No. 9. Distilled water, containing carbonate of soda equal to 50 grains  $\text{Na}_2\text{CO}_3$  per Imperial gallon.
- No. 10. Distilled water, containing nitrate of potash equal to 50 grains  $\text{KNO}_3$  per Imperial gallon.
- No. 11. Distilled water, containing salts to imitate Burton water, composed of—

	Grains.
Sulphate of lime.....	70.99 $\text{CaSO}_4$ per Imperial gallon.
Carbonate of lime.....	4.04 $\text{CaCO}_3$ per Imperial gallon.
Carbonate of magnesia..	5.88 $\text{MgCO}_3$ per Imperial gallon.
Sulphate of potash.....	0.96 $\text{K}_2\text{SO}_4$ per Imperial gallon.
Sulphate of soda.....	13.30 $\text{Na}_2\text{SO}_4$ per Imperial gallon.
Chloride of Sodium.....	9.12 $\text{NaCl}$ per Imperial gallon.
Ferric oxide.....	1.13 $\text{Fe}_2\text{O}_3$ per Imperial gallon.

- No. 12. Edinburgh gravitation water.
- No. 13. Distilled water, containing sulphate of potash equal 50 grains  $\text{K}_2\text{SO}_4$  per Imperial gallon.

Before going into the details of the several mashings, &c., it will be well, perhaps, in the first place, to say something about the malt and the hops which were used in the experiments.

First. The malt was made from Scotch barley weighing 56 lb. per bushel. It contained the following percentages of nitrogen, ash, and phosphoric acid:—

#### ANALYSIS OF THE MALT.

	Per Cent.
Total nitrogen.....	1.85
Comprising nitrogen soluble in distilled water, at 150°—160° F.....	0.85
Comprising nitrogen insoluble in distilled water, at 150°—160° F.....	1.00
Total phosphoric acid ( $\text{P}_2\text{O}_5$ ).....	0.98
Comprising $\text{P}_2\text{O}_5$ soluble in distilled water, at 150°—160° F. (wort).....	0.73
Comprising $\text{P}_2\text{O}_5$ insoluble in distilled water, at 150°—160° F. (draff).....	25
Total ash in malt.....	2.21
Comprising, soluble ash in extract (wort).....	1.32
Comprising, insoluble ash in residue (draff).....	0.92
In total ash—	Per Cent.
Silica.....	0.56
Lime.....	0.10
Magnesia.....	0.21
Phosphoric acid.....	0.98

	Per Cent.
Extract (wort) soluble in distilled water at 150°—160° F.....	69.36
Insoluble matter (draff) in above.....	27.90
Moisture.....	2.74
	100.00

The hops were "East Kent" and gave on analysis the following results:—

#### ANALYSIS OF THE HOPS (EAST KENT).

	Per Cent.
Total nitrogen in hops.....	2.68.
Comprising nitrogen soluble in boiling water....	0.67
Comprising nitrogen insoluble in boiling water.....	2.01
Total phosphoric acid in hops.....	1.09
Comprising $\text{P}_2\text{O}_5$ soluble in boiling water.....	0.64
Comprising $\text{P}_2\text{O}_5$ insoluble in boiling water.....	0.45
Total ash in hops.....	8.55
Comprising ash soluble in boiling water.....	4.57
Comprising ash insoluble in boiling water.....	3.98
Ash contains—	
Silica.....	1.80 soluble 0.57 insoluble 1.23
Lime.....	1.47 " 0.18 " 1.29
Magnesia.....	0.45 " 0.25 " 1.21
Phosphoric acid... ..	1.00 " 0.61 " 0.45

	Per Cent.
Extract (soluble in boiling distilled water).....	24.15
Insoluble matter.....	63.93
Moisture.....	11.92
	100.00

The procedure was similar in all the experiments, but owing to an accident only 11 of them could be commenced on the same day, the other two (Nos. 8 and 13) had to stand over until the following day, when they also were begun under precisely similar conditions as the rest.

The following is an outline of the general treatment. Six-sevenths of a lb. (= 6,000 grains) of Scotch malt in the form of grist was mashed in 26,000 grains of water.

The water was first of all heated to 160° F. in copper pans. The burner was removed and the grist then rapidly stirred in. The temperature of the mixture fell to 145° F., but was gradually raised until it reached 157° F., at which temperature it was maintained until the completion of the mash.

The mash was now filtered through cloth bags until 16,000 grains of liquor had passed through clear. To each of the worts thus obtained 5,000 measured grains of the original water were added before boiling, thus making up a volume of 21,000 grains.

The draffs left on the cloth filters were washed with hot water until all the soluble matters were removed, the filtrates were thrown away, and the washed and drained draffs were dried and weighed.

The entire time occupied in the mashing process from the first introduction of the grist to the filtration, was an hour and a half, except in two cases—viz., Nos. 5 and 9—carbonate of magnesia and carbonate of soda respectively. When, owing to the liquors, both showing the starch reaction with iodine, the time was extended to two hours.

In the case of the carbonate of magnesia it is well to note that before the expiry of two hours the starch reaction had ceased, but the carbonate of soda experiment gave the reaction to the last. Both experiments filtered badly, and the liquors came through muddy and thick.

The worts were now simultaneously boiled for 1½ hours with 100 grains of hops each, and then rapidly filtered through cloth bags, as before, the liquors being returned to the filters till clear and bright.

The spent hops on the filters were washed with boiling distilled water, and the washings were added to the boiled wort until the latter occupied a volume of 16,000 grains.

The hops were further washed to remove all soluble matters, they were then drained and dried.

The gravities and the temperatures were now taken, and 500 measured grains were removed from each of the boiled worts and placed in sterilised test-tubes for preservation.

The fermentations were all commenced on the same days that the mashings and boilings were undertaken. Flasks holding about 26,000 grains were employed for the purpose; they were provided with india-rubber corks, through which passed a Fahrenheit thermometer and a safety tube for the escape of the carbonic anhydride.

The flasks were all placed in a shallow tin tray, which contained about 2 inches of water—and the temperature was maintained by an Argand lamp kept at a distance of 2½ feet from the tray.

Eleven of the fermentations occupied 9 days, and two of them 8 days.

Full details as to the quantity of malt, hops, and yeast employed, as well as to the original gravities and temperatures, and rates of alternation, are to be found in the description of each experiment which follows.

27 February 1891.

No. I.—DISTILLED WATER.

6,000 grains (½ lb.) Scotch malt { Temperature 145° F.,  
gradually increased to  
157° F.  
26,000 „ distilled water { Duration of mashing  
1½ hours.

Specific gravity of wort from above at 60° F.—1.060.

Volume of wort, 16,000 grains, to which were added 5,000 grains of distilled water, boiled for 1½ hours with 100 grains E. Kent hops, filtered and made up to 16,000 grains with distilled water at 60° F.—500 grains sterilised.

15,500 grains fermented with 150 grains yeast.

Date.	Temperature.	Specific Gravity.	Fall in Gravity.
27 Feb.	48° F.	1057.5	..
28 „	After 18 hours, 48°	1057.0	0.5
1 Mar.	„ 24 „ 55°	1050.0	7.0
2 „	„ 24 „ 56°	1043.0	7.0
3 „	„ 24 „ 58°	1039.0	8.0
4 „	„ 24 „ 61°	1027.0	8.0
5 „	„ 24 „ 67°	1020.0	7.0
6 „	„ 24 „ 72°	1015.0	5.0
7 „	„ 24 „ 72°	1013.0	2.0
8 „	„ 24 „ 73° (decanted from yeast)	1012.0	1.0
			45.5

Weight of spent hops (dry)—70 grains.

Analysis of Beer.	Per Cent.	Grains per Gallon.
Alcohol.....	4.57	..
Total solids (extract) .....	5.16	3.612
Nitrogen .....	0.098	..
Ash.....	0.265	..
Lime .....	0.067	..
Phosphoric acid.....	0.122	..
Acidity calculated to lactic acid (C <sub>3</sub> H <sub>5</sub> O <sub>3</sub> ) .....	0.18	..
Specific gravity to 60° F. ....	1012.8	..

27th February 1894.

No. II.—CARBONATE OF LIME WATER.

50 grains CaCO<sub>3</sub> to gallon dissolved in CO<sub>2</sub>

6,000 grains (½ lb.) Scotch malt { Temperature 145° F.,  
gradually increased to  
157° F. Much frothing.  
26,000 „ of above water { Duration of mash, 1½  
hours. Extreme frothing.

Specific gravity of wort from above at 60° F.—1.059.5

Volume of wort, 16,000 grains, to which were added 5,000 grains of original water, boiled for 1½ hours with 100 grains E. Kent hops, filtered and made up to 16,000 grains with distilled water at 60° F.—500 grains sterilised.

15,500 grains fermented with 150 grains yeast.

Date.	Temperature.	Specific Gravity.	Fall in Gravity.
27 Feb.	48° F.	1041.5	..
28 „	After 18 hours, 48	1050.5	1.0
1 Mar.	„ 24 „ 53	1047.0	3.5
2 „	„ 24 „ 56°	1047.0	8.0
3 „	„ 24 „ 58	1039.0	7.0
4 „	„ 24 „ 61°	1024.0	6.0
5 „	„ 24 „ 67	1018.0	6.0
6 „	„ 24 „ 71°	1012.0	6.0
7 „	„ 24 „ 71	1010.0	2.0
8 „	„ 24 „ 72 (decanted from yeast)	1009.0	1.0
			49.5

Weight of spent hops (dry)—67 grains.

Analysis of Beer.	Per Cent.	Grains per Gallon.
Alcohol.....	3.95	..
Total solids (extract) .....	4.25	2.975
Nitrogen .....	0.084	..
Ash.....	0.19	..
Lime .....	0.013	..
Phosphoric acid .....	0.089	..
Acidity calculated to lactic acid.....	0.144	..
Silica.....	0.008	..
Specific gravity to 60° F. ....	1009.5	..

27th February 1894.

No. III.—CHLORIDE OF CALCIUM WATER.

(50 grains CaCl<sub>2</sub> to gallon.)

6,000 grains (½ lb.) Scotch malt { Temperature 145° F.,  
gradually increased to  
157° F.  
26,000 grains of above water { Duration of mash, 1½  
hours.

Specific gravity of wort from above at 60° F., 1058.

Volume of wort 16,000 grains, to which were added 5,000 grains of original water, boiled for 1½ hours with 100 grains E. Kent hops, filtered and made up to 16,000 grains with distilled water at 60° F.—500 grains sterilised.

15,500 grains fermented with 150 grains yeast.

Date.	Temperature.	Specific Gravity.	Fall in Gravity.
27 Feb.	48° F.	1057	..
28 „	After 18 hours, 48°	1054	1
1 Mar.	„ 24 „ 53°	1055	1
2 „	„ 24 „ 56° (very bright.)	1043	12
3 „	„ 24 „ 58	1036	7
4 „	„ 24 „ 61 (bright.)	1028	8
5 „	„ 24 „ 67°	1022	6
6 „	„ 24 „ 71°	1018	4
7 „	„ 24 „ 71°	1016	2
8 „	„ 24 „ 72° bright, decanted from yeast	1016	0
			41

Weight of spent hops (dry)—74 grains.



Analysis of Beer.	Per Cent.	Grains per Gallon.
Alcohol .....	4.02	..
Total solids (extract) .....	6.10	4270
Nitrogen .....	0.101	..
Ash .....	0.247	..
Lime .....	0.021	..
Phosphoric acid .....	0.104	..
Acidity as lactic acid .....	0.162	..
Silica .....	..	..
Magnesia .....	..	..
Specific gravity at 60° F. ....	1.016.5	..

27th February 1894.

## No. IV. SULPHATE OF LIME (GYPSUM) WATER.

(50 grains  $\text{CaSO}_4$  to gallon.)

5,000 grains ( $\frac{1}{2}$  lb.) Scotch malt { Temperature 145° F.  
gradually increased to  
157° F.  
26,000 „ of above water { Duration of mash,  
1½ hours.  
Specific gravity of wort from above at 60° F., 1.059.5.  
Volume of wort 16,000 grains, to which were added  
5,000 grains of original water, boiled for 1½ hours with  
100 grains E. Kent hops, filtered and made up to 16,000  
grains with distilled water at 60° F.—500 grains sterilised.  
15,500 grains fermented with 150 grains yeast.

Date.	Temperature.	Specific Gravity.	Fall in Gravity.
27 Feb.	48° F.	1059	..
28 „	After 18 hours, 48	1059	0
1 Mar.	„ 24 „ 53°	1052	7
2 „	„ 24 „ 56°	1047	5
3 „	„ 24 „ 56	1039	8
4 „	„ 24 „ 61°	1029	10
5 „	„ 24 „ 67	1022	7
6 „	„ 24 „ (clear.) 71°	1017	5
7 „	„ 24 „ 71	1014	3
8 „	„ 24 „ 72° (decanted from yeast.)	1012	2
			47

Weight of spent hops (dry)—76 grains.

Analysis of Beer.	Per Cent.	Grains per Gallon.
Alcohol .....	4.88	..
Total solids (extract) .....	5.9.5	3593.5
Nitrogen .....	0.112	..
Ash .....	0.283	..
Lime .....	0.018	..
Phosphoric acid .....	0.107	..
Acidity as lactic acid .....	0.162	..
Silica .....	0.007	..
Specific gravity at 60° F. ....	1.011.9	..

27th February 1894.

## No. V.—CARBONATE OF MAGNESIA WATER.

(50 grains  $\text{MgCO}_3$  to gallon dissolved in  $\text{CO}_2$ )

6,000 grains ( $\frac{1}{2}$  lb.) Scotch malt { Temperature from 145°  
F. gradually increased  
to 157° F. Diastatic  
action very slow.  
26,000 „ of above water { Extreme frothing.  
Duration of mash,  
1.45 hours.

Specific gravity of wort from above at 60° F., 1.060.5.

Volume of wort 15,500 grains, to which were added  
5,000 grains of original water, boiled for 1½ hours with  
100 grains E. Kent hops, filtered very badly and very  
muddy.

Total filtrate 5,500 grains.—500 grains sterilised.

5,000 grains fermented with 50 grains yeast.

Date.	Temperature.	Specific Gravity.	Fall in Gravity.
27 Feb.	48° F.	1065	..
28 „	After 18 hours, 48°	1054	1
1 March	„ 24 „ 53°	1050	4
2 „	„ 24 „ 55°	1047	13
3 „	„ 24 „ 58°	1036	11
4 „	„ 24 „ 59°	1028	8
5 „	„ 24 „ 66°	1022	6
6 „	„ 24 „ 70°	1021	1
7 „	„ 24 „ 68°	1021	0
8 „	„ 24 „ 70° (decanted from yeast.)	1021	0
			44

Weight of spent hops (dry)—68 grains.

Analysis of Beer.	Per Cent.	Grains per Gallon.
Alcohol .....	4.14	..
Total solids (extract) .....	7.44	5208
Nitrogen .....	Not estimated	..
Ash .....	0.273	..
Lime .....	None	..
Phosphoric acid .....	0.101	..
Acidity as lactic acid .....	0.054	..
Silica .....	0.012	..
Magnesia .....	0.025	..
Specific gravity at 60° F. ....	Not determined.	..

27th February 1894.

## No. VI.—CHLORIDE OF MAGNESIUM WATER.

(50 grains  $\text{MgCl}_2$  to gallon.)

6,000 grains ( $\frac{1}{2}$  lb.) Scotch malt { Temperature from 145°  
F. gradually raised to  
to 157° F.  
26,000 „ of above water { Duration of mash, 1½  
hours.

Specific gravity of wort from above at 60° F., 1.060.

Volume of wort, 16,000 grains, to which were added  
5,000 grains of original water, boiled for 1½ hours with  
100 grains E. Kent hops, filtered and made up to 16,000  
grains with distilled water at 60° F. 500 grains sterilised.  
15,000 grains fermented with 150 grains yeast.

Date.	Temperature.	Specific Gravity.	Fall in Gravity.
27 Feb.	48° F.	1058	..
28 "	After 18 hours, 48°	1056	2
1 Mar.	" 24 " 53°	1055	1
2 "	" 24 " 56°	1043	12
3 "	" 24 " 58°	1033	10
4 "	" 24 " 60°	1023	10
5 "	" 24 " 67°	1018	5
6 "	" 24 " 70° (clear).	1016	2
7 "	" 24 " 70°	1012	4
8 "	" 24 " 71°	1012	0
			43

Weight of spent hops (dry)—71 grains.

Analysis of Beer.	Per Cent.	Grains per Gallon.
Alcohol.....	4.64	..
Total solids (extract).....	5.12	5.84
Nitrogen.....	0.085	..
Ash.....	0.28	..
Lime.....	None	..
Phosphoric acid.....	0.104	..
Acidity as lactic acid.....	0.144	..
Silica.....	0.005	..
Magnesia.....	0.052	..
Specific gravity at 60° F.....	1012.7	..

27th February 1894.

## NO. VII.—SULPHATE OF MAGNESIA WATER.

(50 grains  $\text{MgSO}_4$  to gallon.)

6,000 grains ( $\frac{2}{3}$  lb.) Scotch malt { Temperature 145° F.  
gradually raised to  
157° F.  
26,000 " of above water { Duration of mash, 1½  
hours.

Specific gravity of wort from above at 60° F., 1060.

Volume of wort 16,000 grains, to which were added  
5,000 grains of original water, boiled for 1½ hours with  
100 grains E. Kent hops, filtered and made up to 16,000  
grains with distilled water at 60° F. 500 grains sterilised.

15,500 grains fermented with 150 grains yeast.

Date.	Temperature.	Specific Gravity.	Fall in Gravity.
27 Feb.	48° F.	1058	..
28 "	After 18 hours, 48°	1058	0
1 Mar.	" 24 " 53°	1055	3
2 "	" 24 " 56°	1044	11
3 "	" 24 " 59° (brisk fermentation).	1035	9
4 "	" 24 " 65°	1028	7
5 "	" 24 " 67°	1021	7
6 "	" 24 " 70°	1019	2
7 "	" 24 " 70°	1017	2
8 "	" 24 " 71°	1016	1
			42

Weight of spent hops (dry)—68 grains.

Analysis of Beer.	Per Cent.	Grains per Gallon.
Alcohol.....	4.34	..
Total solids (extract).....	6.07	4.24
Nitrogen.....	0.104	..
Ash.....	0.206	..
Lime.....	None	..
Magnesia.....	0.052	..
Phosphoric acid.....	0.113	..
Acidity as lactic acid.....	0.123	..
Silica.....	0.004	..
Specific gravity at 60° F.....	1017.5	..

28th February 1894.

NO. VIII.—CHLORIDE OF SODIUM (COMMON SALT)  
WATER.

(50 grains NaCl to gallon.)

6,000 grains ( $\frac{2}{3}$  lb.) Scotch malt { Temperature 145° F.  
gradually raised to  
157° F.  
26,000 " of above water { Duration of mash,  
1½ hours.

Specific gravity of wort from above at 60° F., 1.058.

Volume of wort, 16,000 grains, to which were added  
5,000 grains of original water, boiled for 1½ hours with 100  
grains E. Kent hops, filtered and made up to 16,000 grains  
with distilled water at 60° F. 500 grains sterilised.

15,500 grains fermented with 150 grains yeast.

Date.	Temperature.	Specific Gravity.	Fall in Gravity.
28 Feb.	52° F.	1055	..
1 Mar.	After 18 hours, 53°	1054	0
2 "	" 24 " 58°	1046	10
3 "	" 24 " 60°	1037	9
4 "	" 24 " 60°	1026	11
5 "	" 24 " 67° (clear).	1021	5
6 "	" 24 hours, 71°	1018	3
7 "	" 24 " 72°	1018	0
8 "	" 24 " 74° (clear).	1018	0
			38

Weight of spent hops (dry)—70 grains.

Analysis of Beer.	Per Cent.	Grains per Gallon.
Alcohol.....	3.84	..
Total solids (extract).....	6.15	4.305
Nitrogen.....	0.10	..
Ash.....	0.34	..
Lime.....	None	..
Magnesia.....	..	..
Phosphoric acid.....	0.20	..
Acidity as lactic acid.....	0.144	..
Silica.....	None	..
Specific gravity at 60° F.....	1018.4	..

27th February 1894

No. IX.—CARBONATE OF SODA WATER.  
(50 grains  $\text{Na}_2\text{CO}_3$  to gallon.)

6,000 grains ( $\frac{1}{2}$  lb.) Scotch malt  
26,000 „ of above water

Temperature 145° F.,  
gradually raised to  
157° F.  
Duration of mash,  
2 hours.

Diastatic action *not* complete when filtered.

Specific gravity of wort from above at 60° F.—1,062.

Filtered badly. Volume of wort, 10,000 grains, to which were added 3,100 of original water, boiled for  $1\frac{1}{4}$  hours with 66 grains E. Kent hops, filtered and made up to 16,000 grains with distilled water at 60° F.—500 grains sterilised.

95,000 grains fermented with 100 grains yeast.

Date.	Temperature.	Specific Gravity.	Fall in Gravity.
27 Feb.	48° F.	1054°0	..
28 „	After 18 hours, 48°	1052°5	1°5
1 Mar.	„ 24 „ 53°	1048°0	4°5
2 „	„ 24 „ 56°	1035°0	13°0
3 „	„ 24 „ 59°	1024°0	12°0
4 „	„ 24 „ 61°5	1019°0	4°0
5 „	„ 24 „ 68°	1017°0	2°0
6 „	„ 24 „ 71°	1016°0	1°0
7 „	„ 24 „ 72	1015°0	1°0
8 „	„ 24 „ 73	1015 0	0°0
			39°0

Weight of spent hops (dry)—48 grains = 72·7 grains from 100 grains hops.

Analysis of Beer.	Per Cent.	Grains per Gallon.
Alcohol.....	3·73	..
Total solids (extract).....	5·72	4004
Nitrogen.....	0·047	..
Ash.....	0·285	..
Lime.....	..	..
Magnesia.....	..	..
Phosphoric acid.....	0·093	..
Acidity as lactic acid.....	0·072	..
Silica.....	0·009	..
Specific gravity at 60° F.....	Not determined.	..

27th February 1894.

No. 10.—NITRATE OF POTASH WATER.  
(50 grs.  $\text{KNO}_3$  to gallon.)

6,000 grains Scotch malt  
26,000 „ of above water

Temperature from 145° F.,  
gradually raised to  
157° F.  
Duration of mash,  $1\frac{1}{2}$  hours.

Specific gravity of wort from above at 60° F.—1058.

Volume of wort 16,000 grains, to which were added 5,000 grains of original water, boiled for  $1\frac{1}{4}$  hours with 100 grains E. Kent hops, filtered and made up to 16,000 grains with distilled water at 60° F.—500 grains sterilised.

15,500 grains fermented with 150 grains yeast.

Date.	Temperature.	Specific Gravity.	Fall in Gravity.
27 Feb.	48° F.	1052	..
28 „	After 18 hours, 48°	1051	1
1 Mar.	„ 24 „ 53°	1047	4
2 „	„ 24 „ 56°	1034	11
3 „	„ 24 „ 59°	1027	9
4 „	„ 24 „ 60°	1023	4
5 „	„ 24 „ 66°	1020	3
6 „	„ 24 „ 69°	1019	1
7 „	„ 24 „ 69°	1019	0
8 „	„ 24 „ 70°	1019	0
			31

Weight of spent hops (dry)—72 grains.

Analysis of Beer.	Per Cent.	Grains per Gallon.
Alcohol.....	3·26	..
Total solids (extract).....	6·29	4103
Nitrogen.....	0·09	..
Ash.....	0·282	..
Lime.....	..	..
Magnesia.....	..	..
Phosphoric acid.....	0·118	..
Acidity as lactic acid.....	0·114	..
Silica.....	0·015	..
Specific gravity at 60° F.....	1019·7	..

27th February 1894.

No. XI.—IMITATION BURTON WATER.  
(Composition see page 368.)

6,000 grains ( $\frac{1}{2}$  lb.) Scotch malt  
26,000 „ of above water

Temperature 145° F.,  
gradually raised to  
157° F.  
Duration of mash,  
 $1\frac{1}{2}$  hours.

Specific gravity of wort from above at 60° F.—1,061.

Volume of wort 16,000 grains, to which were added 5,000 grains of original water, boiled for  $1\frac{1}{4}$  hours with 100 grains E. Kent hops, filtered and made up to 16,000 grains with distilled water at 60° F.—500 grains sterilised

15,500 grains fermented with 150 grains yeast.

Date.	Temperature.	Specific Gravity.	Fall in Gravity.
27 Feb.	48° F.	1060	..
28 „	After 18 hours, 48°	1059	..
1 March	„ 24 „ 53°	1056	4
2 „	„ 24 „ 55°	1048	8
3 „	„ 24 „ 59°	1043	5
4 „	„ 24 „ 60°	1034	9
5 „	„ 24 „ 67°	1028	6
6 „	„ 24 „ 70°	1022	6
7 „	„ 24 „ 70°	1018	4
8 „	„ 24 „ 70°	1016	2
			44

Weight of spent hops (dry) 77 grains.

Analysis of Beer.	Per Cent.	Grains per Gallon.
Alcohol.....	4.70	..
Total solids (extract).....	5.49	39.8
Nitrogen.....	0.112	..
Ash.....	0.359	..
Lime.....	..	..
Magnesia.....	0.043	..
Phosphoric acid.....	0.092	..
Acidity as lactic acid.....	0.123	..
Silica.....	0.007	..
Specific gravity at 60° F.....	1.015.7	..

27th February 1894.

## No. XII.—EDINBURGH GRAVITATION WATER.

6,000 grains ( $\frac{1}{2}$  lb.) Scotch malt { Temperature 145° F.,  
gradually raised to  
157° F.  
26,000 „ of above water { Duration of mash, 1 $\frac{1}{2}$   
hours.

Specific gravity of wort from above at 60° F.—1.060.  
Volume of wort, 16,000 grains, to which were added  
5,000 grains of original water, boiled for 1 $\frac{1}{4}$  hours with  
100 grains E. Kent hops, filtered and made up to 16,000  
grains with distilled water at 60° F.—500 grains sterilised.  
15,500 grains fermented with 150 grains yeast.

Date.	Temperature.	Specific Gravity.	Fall in Gravity.
27 Feb.	48° F.	1054	..
28 „	After 18 hours 48°	1052	2
1 Mar.	„ 24 „ 53°	1046	6
2 „	„ 24 „ 56°	1037	9
3 „	„ 24 „ 58°	1030	7
4 „	„ 24 „ 61°	1022	8
5 „	„ 24 „ 67°	1018	4
6 „	„ 24 „ (clear), 71°	1016	2
7 „	„ 24 „ 72°	1015	1
8 „	„ 24 „ 72°	1015	0
			39

Weight of spent hops (dry)—72 grains.

Analysis of Beer.	Per Cent.	Grains per Gallon.
Alcohol.....	3.75	..
Total solids (extract).....	5.67	39.69
Nitrogen.....	0.084	..
Ash.....	0.228	..
Lime.....	..	..
Magnesia.....	..	..
Phosphoric acid.....	0.094	..
Acidity as lactic acid.....	0.144	..
Silica.....	0.003	..
Specific gravity at 60° F.....	1.015.9	..

28th February 1894.

## No. XIII.—SULPHATE OF POTASH WATER.

(50 grains  $K_2SO_4$  to gallon.)

6,000 grains ( $\frac{1}{2}$  lb.) { Temperature 145° F., gradu-  
ally raised to 157° F.  
26,000 „ of above water { Duration of mash, 1 $\frac{1}{2}$  hours.  
Specific gravity of wort from above at 60° F.—1.058.  
Volume of wort, 16,000 grains, to which were added  
5,000 grains of original water, boiled for 1 $\frac{1}{4}$  hours with  
100 grains E. Kent hops, filtered and made up to 16,000  
grains with distilled water at 60° F.—500 grains sterilised.  
15,500 grains fermented with 150 grains yeast.

Date.	Temperature.	Specific Gravity.	Fall in Gravity.
28 Feb.	50° F.	1055	..
1 Mar.	After 18 hours, 53°	1055	..
2 „	„ 24 „ 56°	1045	10
3 „	„ 24 „ 58°	1034	11
4 „	„ 24 „ 61°	1026	8
5 „	„ 24 „ 67°	1023	3
6 „	„ 24 „ rather muddy, 71°	1020	3
7 „	„ 24 „ 71°	1020	0
8 „	„ 24 „ 73°	1020	0
			35

Weight of spent hops (dry)—72 grains.

Analysis of Beer.	Per Cent.	Grains per Gallon.
Alcohol.....	3.73	..
Total solids (extract).....	7.055	49.875
Nitrogen.....	0.087	..
Ash.....	0.310	..
Lime.....	..	..
Magnesia.....	..	..
Phosphoric acid.....	0.067	..
Acidity as lactic acid.....	0.144	..
Silica.....	0.004	..
Specific gravity at 60° F.....	1.021.1	..

Let us now consider some of the points brought out  
by the experiments, taking them up in the following  
order:—

1. Diastatic action.
2. The colour and clearness of the wort filtered from the draff and then boiled with hops.
3. The attenuation.
4. The settling and clarification of the beers.
5. Colour of the beers.
6. Analyses of the beers.
7. Condition, flavour, odour, and stability of the beers.
8. Weights of the residues.

## 1. The Diastatic Action.

Except in the two cases previously referred to (carbonate of magnesia and carbonate of soda) the action was completed in less than an hour: the carbonate of magnesia water, however, took nearly two hours, while the mash prepared with carbonate of soda water had to be filtered incomplete. It is thus evident that the diastatic action is impeded by both these salts, but especially so by carbonate of soda.

This is also true, but to a much less extent, with carbonate of lime water.

## 2. The Colour and Clearness of the Wort filtered from the Draff and then boiled with Hops.

All the worts, while hot, filtered clear and bright, except those prepared with carbonate of magnesia and carbonate of soda waters, respectively, which were both slow in filtering and gave muddy filtrates.

After boiling with the hops and filtering, specimens from each of the worts were sterilised and are in test tubes and in the small flasks on the table for your inspection. Regarding their colour, after a good deal of comparison, both by reflected and transmitted light, I consider that the following is the order of paleness.

- No. 1.—Distilled water. The palest wort.  
 „ 13.—Sulphate of potash water.  
 „ 7.—Sulphate of magnesia water.  
 „ 4.—Sulphate of lime water.  
 „ 6.—Chloride of magnesium water.  
 „ 12.—Edinburgh gravitation water.  
 „ 11.—Imitation Burton water.  
 „ 10.—Nitrate of potash water.  
 „ 3.—Chloride of calcium water.  
 „ 8.—Chloride of sodium water.  
 „ 2.—Carbonate of lime water.  
 „ 5.—Carbonate of magnesia water.  
 „ 9.—Carbonate of soda water, the darkest.

## 3. The Attenuation.

During the fermentations, which occupied nine days, except in the beers prepared with chloride of sodium and sulphate of potash, which were completed in eight days the worts attenuated in the following order :—

No.	—	—	Loss in Gravity.
4	Sulphate of lime water .....	48° F. 72° F. 1.059 to 1.012	47.0
6	Chloride of magnesium water ....	48° F. 71° F. 1.058 to 1.012	46.0
1	Distilled water .....	48° F. 73° F. 1.057.5 to 1.012	45.5
5	Carbonate of magnesia water.....	48° F. 70° F. 1.065 to 1.021	44.0
11	Barton water .....	48° F. 70° F. 1.060 to 1.016	44.0
2	Carbonate of lime water .....	48° F. 72° F. 1.051.5 to 1.009	42.5
7	Sulphate of magnesia water.....	48° F. 71° F. 1.058 to 1.016	42.0
3	Chloride of calcium water.....	48° F. 72° F. 1.057 to 1.015	41.0
9	Carbonate of soda water .....	48° F. 73° F. 1.054 to 1.015	39.0
12	Edinburgh water .....	48° F. 72° F. 1.054 to 1.015	39.0
8	Chloride of sodium water .....	52° F. 71° F. 1.056 to 1.018	38.0
13	Sulphate of potash water .....	50° F. 73° F. 1.055 to 1.020	35.0
10	Nitrate of potash water .....	48° F. 70° F. 1.052 to 1.019	33.0

The beers, after fermentation, were decanted from the yeast into bottles and firmly corked, and were left at rest for two weeks to deposit the floating yeast and clarify.

## 4. The Settling and Clarification of the Beers.

About a fortnight after the beers were made they were examined to see which had cleared the most completely; the following was observed :—

Beer made with—

- No. 1.—*Distilled water*.—Almost clear.  
 „ 2.—*Carbonate of lime water*.—Muddy.  
 „ 3.—*Chloride of calcium water*.—Fairly clear.  
 „ 4.—*Sulphate of lime water*.—Very clear.  
 „ 5.—*Carbonate of magnesia water*.—Clear, but very dark in colour.  
 „ 6.—*Chloride of magnesium water*.—Cloudy.  
 „ 7.—*Sulphate of magnesia water*.—Cloudy.  
 „ 8.—*Chloride of sodium water*.—Cloudy.  
 „ \*9.—*Carbonate of soda water*.—Clear, but very dark.  
 „ 10.—*Nitrate of potash water*.—Slightly cloudy.  
 „ 11.—*Imitation Burton water*.—Slightly cloudy.  
 „ 12.—*Edinburgh gravitation water*.—Almost clear.  
 „ 13.—*Sulphate of potash water*.—Cloudy.

The beers were allowed to settle a few days longer, and exactly one month from the date of the mashing process they were syphoned off from the settling-bottles to the set of 8-oz. bottles you see on the table, and which were then corked and sealed.

## 5. Colour of the Beers.

The most coloured of all are—

No. 5, carbonate of magnesia, and the next No. 9, carbonate of soda (which became cloudy when syphoned into the smaller bottles). No. 2, carbonate of lime is also rather highly coloured.

Probably the palest is that made with distilled water, but the following resemble the latter very closely: chloride of calcium, sulphate of magnesia, and chloride of magnesium.

## 6. Analyses of the Beers.

(a.) *The Alcohol*.—The percentage of alcohol is highest in No. 4 (sulphate of lime water).

The following shows the specific gravities of the beers with the per cent. of alcohol in each, beginning at the highest—

No.	—	Specific Gravity at 60° F.	Alcohol.
			Per Cent.
4	Sulphate of lime water.....	1011.9	4.88
11	Imitation Burton water .....	1012.7	4.70
6	Chloride of magnesium water ....	1012.7	4.64
1	Distilled water .....	1012.8	4.57
7	Sulphate of magnesia water .....	1017.5	4.33
5	Carbonate of magnesia water ....	Not determined.	4.14
3	Chloride of calcium water.....	1016.5	4.02
2	Carbonate of lime water .....	1009.5	3.96
8	Chloride of sodium water .....	1018.4	3.84
12	Edinburgh gravitation water.....	1015.9	3.78
9	Carbonate of soda water.....	Not determined.	3.73
13	Sulphate of potash water.....	1021.1	3.73
10	Nitrate of potash water.....	1019.7	3.26

(b.) *The Extract or Total Solids*, calculated to grains per gallon, varies from 5.208 grs. per gallon in No. 5 (carbonate of magnesia) water to 2.975 grs. in No. 13, sulphate of potash water.

(c.) *The Nitrogens* were all determined by the Kjeldahl method. The highest results are Nos. 4 and 11, viz., gypsum and imitation Burton waters, each of the beers from these waters contains 0.112 per cent. of nitrogen, thus establishing what has been the general opinion, though denied by certain authorities, e.g., Southby, County Brewers Gazette, 1879, that gypseous waters have an appreciable effect on the solubility of the nitrogenous matter of malt.

\* Became cloudy on standing longer.

The lowest percentage of nitrogen in any of the beers is that made from water containing carbonate of soda (No. 9).

I regret that, owing to clogging of the filters, there was not sufficient beer produced from the carbonate of magnesia water to permit of the nitrogen being estimated, or of the gravity being taken.

No attempt whatever was made to determine in what condition the nitrogen exists in the beers, that is, what proportion of the nitrogen is combined, as albumenoids, peptones, or amides, &c., the subject is an exceedingly complicated one, and it appears there is no reliable method for the separation of these nitrogenous bodies.

(d.) *The Phosphoric Acid* ( $P_2O_5$ ).—The highest percentage of phosphoric acid was found in the beer from distilled water, where it reached 0.122 per cent. by weight, the next was from nitrate of potash water, 0.118 per cent.; then sulphate of magnesia, 0.113 per cent., and sulphate of lime, 0.107 per cent.

(e.) *The Acidity calculated to Lactic Acid*.—The least acid of the beers is No. 5 (carbonate of magnesia) water containing 0.051 per cent. of acidity, the next is No. 9 (carbonate of soda) water 0.072 per cent., Nos. 7 (sulphate of magnesia) and 11 (imitation Burton) are both 0.123 per cent.; five others contain acid equivalent to 0.144 per cent. lactic, while the most acid of all is No. 1 distilled water, which contains 0.18 per cent.

#### 7. The Condition, Flavour, Odour, and Stability of the Beers, and Effect of Waters generally.

In regard to the flavour and odour I have been much assisted by practical brewers. The following is their opinion:—

1. *Distilled Water*.—Gave a thin, soft, clean-flavour beer of no character, odour pleasant.

2. *Carbonate of Lime Water*.—Very bitter and yeasty flavour, odour heavier than 1, harsh after-flavour.

3. *Chloride of Calcium Water*.—Full flavour, sweet pleasant odour.

4. *Sulphate of Lime Water*.—Clean tasted beer.

5. *Carbonate of Magnesia*.—(Not tasted).

6. *Chloride of Magnesium*.—Peculiar, heavy, rather unpleasant flavour.

7. *Sulphate of Magnesia*.—Medicinal flavour, but pleasant smell.

8. *Common Salt*.—Smell bad, taste unpleasant, beer very turbid.

9. *Carbonate of Soda*.—Very worty, soft unpleasant taste.

10. *Nitrate of Potash*.—Heavy smell, unpleasant taste, resembles that from common salt.

11. *Imitation Burton*.—Good, full flavoured, clean-tasted beer.

12. *Edinburgh Gravitation Water*.—Thin unpleasant flavour.

*Sulphate of Potash Water*.—Only fairly good, has a clean thin flavour.

Of the stability of the beers we are of course not yet in a position to offer any opinion.

The limits of this paper made it impossible to carry out further and fuller analytical details than have been attempted. The first intention was to estimate the maltose and dextrin, and the other constituents both of the unboiled and boiled worts, but to have done so would have taken us far into the night and trespassed on your patience already sufficiently drawn upon. Perhaps some other member may feel disposed to continue the investigations, and if such can be found I would suggest besides the greater analytical details, further trials should be made with peaty waters, waters containing iron, and, lastly, with various combinations of salts.

#### 8. The Weights of the Residues.

I regret to say that it cannot be affirmed that the weights of the dried draffs contained in the bottles on the table are strictly accurate. Unfortunately we were only provided with one set of filtering bags, which were used first for filtering the mash, and, after emptying out and washing, were

used to filter from the hops. It was not possible in the limited time between two filtrations and with so many processes all going on simultaneously to remove the last portions of the washed draff adhering to the bags, but which could have been readily detached when dry.

The weights of the dried hops, however, are reliable. It will be observed that both the sulphate of lime water and the imitation Burton water dissolve out less of the extractive matter of the hops than any of the other waters, confirming what is, I believe, well understood by brewers generally.

It is perhaps right to mention here, that when these experiments were first contemplated, application was made to the Inland Revenue Authorities at Somerset House for permission to proceed, and that whenever the object was explained, the permit, which I have here, was courteously sent me.

In conclusion, I have to thank Messrs. Jeffrey and Co., Brewers, Edinburgh, for kindly supplying the raw materials, and for their assistance generally, in the conduct of the experiments; also to our assistant Mr. G. H. Gemmell, F.C.S., for his useful and ready help; and finally to the Council of the University of Edinburgh, for kindly lending the chemical class room for this meeting of the Society.

#### A USEFUL ELECTRIC FURNACE FOR LABORATORY WORK.

EXHIBITED BY J. B. READMAN, D.S.C., F.R.S.E.

The small model furnace placed before you is likely to prove of much use to chemists who have the electric current at hand, and who are making investigations which require very high temperatures. I may say that my experience of the little furnace is somewhat limited, still I have worked it satisfactorily several times. I have observed that there is a tendency in the outer casing (brass) to become overheated, to the injury of the adjusting screws. These difficulties might be due to higher currents than the carbon electrodes were suited to, or to my want of skill in handling the apparatus.

#### DISCUSSION.

The CHAIRMAN in inviting discussion, said Mr. Readman had given assurance that his paper would be commented upon.

Mr. FORD said that Dr. Readman confirmed the general opinion regarding the effect of sulphate of lime.

Mr. G. BEILBY asked if Mr. Readman could tell them whether the small scale laboratory experiments were corroborated by large scale workings.

Mr. FORD referring to Mr. Beilby's remarks, said he had never found it possible to get a good tasting beer in the laboratory. The beer as a rule retained a yeasty flavour, and the clarification was always rather imperfect. The stability of beer depended greatly upon the purity of the yeast.

Dr. MARSHALL said it was difficult to judge from Dr. Readman's experiments what the actual results in brewing would be, because, in nearly every case, Dr. Readman used solutions of single salts. Calcium in combination with different acid radicles gave widely different results, while similar acid radicles in combination with different bases produced very different reactions, e.g., chloride of calcium and chloride of soda had different actions. A mixture of salts would have given a better idea of the actual results in brewing. Two salts of one metal had, as they knew, a different action to that of corresponding salts of different metals. Take the case of sulphate of soda and of chloride of calcium, how would they behave? They did not know that yet. These experiments Dr. Readman had given were merely preliminary. The chief interest would be when the dissolved salts were mixed in definite quantities. In such a case they would deal with results similar to those which were obtained in brewing.

Dr. FAIXONER KING asked whether the various salts acted as solvents upon the matter of the raw material, or if they acted principally by influencing the chemical changes which took place afterwards. They knew, of course, that distillers wanted as soft a water as possible, while on the other hand, brewers wanted a hard water. He wished to know if sulphate of lime acted merely as a limited solvent, or whether its action was due to induced chemical changes. Dr. Readman had told them that soft water, e.g., water containing carbonate of soda, was not a water from which good beer could be made. That raised the question—did the carbonate of soda acted prejudicially by its extractive powers?

Mr. STANFORD (PRESIDENT OF THE SOCIETY) said the experiments had shown that with distilled water the palest colour was obtained. Was it not generally understood that the use of lime salts produced a paler colour? He understood that carbonate of soda produced a dark solution, and he enquired whether the carbonate of soda did not produce any marked effect upon the fermentation on account of its solvent action.

Mr. PETER BOY considered that one special salt was the chief factor in a water used for brewing. Apparently Dr. Readman's experiments showed that sulphate of lime was the salt of most value. As he apprehended the Burton water contained a large quantity of calcium sulphate. He should expect that with carbonate of soda water a larger quantity of extractive matter would be taken out of the malt than with any of the others, and probably a similar result would be obtained, though only to a smaller extent, with carbonate of magnesium. It seemed that the nitrate of potash water had given the least percentage of alcohol. That was what he would naturally expect. The antiseptic action of nitrate of potash would retard the action of the yeast to a greater degree than would any of the other salts. When extracting such a substance as malt, water containing any dissolved salt invariably had a tendency to darken the resulting product.

Dr. JOHN CLARK said that as a preliminary to further investigation, the best method was to experiment as Dr. Readman had done; to examine individually the different effects of various salts. The better to grasp the effect of these individual salts, Dr. Readman had exaggerated the quantities that would naturally occur in water. They could not judge of the taste of beer when it was made on so small a scale for various reasons, more especially on account of the yeast. One could not expect to get such good results in small quantities as in large, e.g., in clarification the results varied greatly. It might be quite true that if one mixed various ingredients in different proportions, one might have totally different results, but the results of Dr. Readman's various experiments were useful in that they showed what effects might be expected, when exaggerated quantities of these different ingredients were present. When Dr. Readman pointed out the influence of exaggerated quantities, he brought out interesting facts; facts which would lead to investigations of the effect of mixed salts such as approached more closely to those found in the actual practice of brewing.

Dr. READMAN, in replying to the discussion, explained that he had found the yeasty flavour very difficult to get rid of, but in some of the beers, especially where sulphate of lime was present, and where imitation Burton water was used, the yeasty flavour practically disappeared. It was very difficult to carry out small experiments in brewing and be, at the same time, consistent with large scale workings, but the results were at all events suggestive, and he thought that to a certain extent large scale experiments would bear out the results of the small set. The experiments showed that in the case of sulphate of lime and the imitation Burton water, there was less extractive matter taken out of the hops than in any of the others. It was curious how much more difficult carbonate of magnesium water was to work than carbonate of soda water. It was really not possible in the limits of one paper to go beyond single salts. Mixed saline constituents he hoped might form the subject of a paper in the future.

## Journal and Patent\* Literature.

Class.	Page
I.—General Plant, Apparatus, and Machinery .....	376
II.—Fuel, Gas, and Light .....	378
III.—Destructive Distillation, Tar Products, &c. ....	380
IV.—Colouring Matters and Dyes .....	381
V.—Textiles: Cotton, Wool, Silk, &c. ....	384
VI.—Dyeing, Calico Printing, Paper Staining, and Bleaching .....	386
VII.—Acids, Alkalis, and Salts .....	391
VIII.—Glass, Pottery, and Enamels .....	395
IX.—Building Materials, Clays, Mortars and Cements..	396
X.—Metallurgy .....	398
XI.—Electro-Chemistry and Electro-Metallurgy .....	402
XII.—Fats, Oils, and Soap Manufacture .....	404
XIII.—Pigments and Paints; Resins, Varnishes, &c.; India-Rubber, &c. ....	406
XIV.—Tanning, Leather, Glue, and Size .....	408
XV.—Manures, &c. ....	409
XVI.—Sugar, Starch, Gum, &c. ....	410
XVII.—Brewing, Wines, Spirits, &c. ....	410
XVIII.—Chemistry of Foods; Sanitary Chemistry and Water Purification; Disinfectants .....	412
XIX.—Paper, Pasteboard, &c. ....	414
XX.—Fine Chemicals, Alkaloids, Essences, and Extracts	415
XXI.—Photographic Materials and Processes .....	417
XXII.—Explosives, Matches, &c. ....	417
XXIII.—Analytical Chemistry .....	418

## I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

*The Dervaux Feed-Water Purifier.* J. C. A. Simon  
Thomas. Zeits. ang. Chem. 1893, 535—536.

THE author refers to Dr. Jones' paper on purifying feed water (Zeits. ang. Chem. 1892, 474), which gives preference to those methods in which the water is treated before it is fed into the boiler. He agrees with this opinion, and states that a further objection to purifying the water in the boiler itself is that the dense purifying solution used does not mix readily with the water in the boiler, and does not therefore attain its object at once. This renders it impossible to make trustworthy tests to determine what quantity of purifying solution is to be added day by day. He then describes at length his experiments with a Dervaux purifying apparatus at the Maiden Powder Factory (Holland) which led him to form the opinion above mentioned. Working for 39 days, according to the instructions supplied by the makers of the apparatus, he arrived at the enormous quantity of 8.5 kilos. of caustic soda and 7.5 kilos. of carbonate of soda as the amount required. On the 40th day the tests showed that too much had been added. The daily quantity was then gradually reduced and finally omitted altogether. He was surprised to find that the makers' tests continued to show, for six days after the additions had entirely ceased, that too much purifying solution, and sufficient for nine days, had been added daily. A second trial was made, which confirmed the first. A table of results is given, which shows that the alkalinity of the water increased constantly, although the amount of cleaning solution added had been daily decreased. The author concludes by saying that in view of this experience he does not hesitate to condemn the purifying of feed water in the boiler itself.—R. B. P.

\* Any of these specifications may be obtained by post by remitting 8d.—the price now fixed for all specifications, postage included—to Sir Henry Reader Lack, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.



*Silicon Carbide ("Carborundum") as an Abrasive for the Polishing Wheel, &c.* O. Mühlhäuser. Zeits. ang. Chem. 1893, 637.

See under XI, page 402.

*Paranitrophenol Sodium as Acid Indicator in Testing Boiler Feed Water for the Presence of Magnesium Chloride.* A. Goldberg. 12 Ber. d. naturw. Gesellsch. 2. Chemnitz, 1893, 68.

See under XXIII., page 419.

# PATENTS.

*An Improved Process for Separating Impurities from Water Introduced into a Boiler and Apparatus therefor.* D. H. Lyon, London. Eng. Pat. 2547, February 4, 1893.

THE invention has reference to an improvement in the means described in Eng. Pat. 5036 of 1882 for purifying the feed water to boilers. In the improved arrangement a thin stream of water is fed into the top of the boiler, through a spring check-valve, which stream is immediately vaporised by the surrounding steam, leaving the contained impurities free to fall into a flipped conical basin, placed vertically below the valve and above the water line. The basin communicates with the external atmosphere by means of a branch pipe closed by a cock, so that the collected impurities can be blown out of the boiler at suitable intervals.—E. G. C.

*Improvements in Filters used in the Manufacture of Alcohols, Oils, Greases, and other Matters.* N. Iseli, Chaux-de-Fonds, Switzerland. Eng. Pat. 2896, February 9, 1893.

THE filter described contains a chamber having within it a thick layer of a special filtering medium, which lies between two perforated plates, with a layer of asbestos on its upper side. The liquid to be filtered is passed upwards under pressure through the filtering medium, suitable cocks being provided. The layer of filtering medium can be compressed by means of a pressure screw. The medium consists of ligneous material, which is impregnated with a concentrated solution of magnesium sulphate or chloride, and then mixed with an amount of alkali equivalent to the magnesium salt used. The intimate mixture is dried and carbonised at a white heat and is introduced into the apparatus in a moist state.—J. G. W.

*Improvements in Apparatus applicable for Evaporating Liquids or Saccharine Solutions.* J. A. Morrell and J. Fromherz, both of New Orleans, U.S.A. Eng. Pat. 3170, February 13, 1893.

THE apparatus consists of a metal shell provided with tubes arranged longitudinally between two transverse diaphragms; these tubes have central cores, so that the liquid passing through the tubes assumes an annular form, thus placing the liquid or saccharine solution near the outer periphery of the pipe to facilitate evaporation. The shell is provided with ports for the admission of steam and the liquid is first led through a worm heated by exhaust steam, to bring it up to a suitable temperature; it is then forced through the evaporating tubes of the shell, where it meets with a stream of nitrogenous vapour, surcharged with electricity. This combined action facilitates evaporation, and the resulting vapour is led off in any suitable direction. The electrified nitrogenous vapour is produced by passing air through a red hot chamber containing scrap copper, and then forcing the vapour through an iron pipe containing a copper coil through which a current is flowing.—E. G. C.

*Improvements in Apparatus for Dyeing and Washing Yarn in the Hank.* J. Rhodes, J. Rhodes, jun., and A. B. Perkins, Bradford, Yorks. Eng. Pat. 5291, March 11, 1893.

See under VI., page 390.

*Improved Apparatus for Filtering and Purifying Water.* E. Winkler, Vienna, Austria. Eng. Pat. 5619, March 15, 1893.

THE patent applies to apparatus containing sand, for the filtration of impure water. Preferably, two vessels are employed, of similar construction, and the improvements are principally involved in the arrangement of pipes and valves for regulating and changing the flow of the water through the vessels, as well as for the injection of chemical reagents.—B.

*Plates for Oil- and other Presses.* W. C. Leechman, Colombo, Ceylon. Eng. Pat. 7039, April 6, 1893.

THE invention has reference to improvements in the plates for oil presses described in Eng. Pat. 9526 of 1890 (this Journal, 1891, 708), in which a compound plate was formed consisting of a thick plate with comparatively large holes, overlaid by a thin plate with very fine holes concentric with those below. The construction is now simplified by making the two plates in one piece, and by drilling or punching holes nearly through the plate, and afterwards piercing fine holes through the diaphragm pieces remaining.—E. G. C.

*Improvement in Apparatus for Filtering Liquids or for Separating Liquids from, or Drying, Crystallised or other Granular Matters.* J. Drummond, Govan, N.B. Eng. Pat. 7525, April 13, 1893.

IN this invention a rotary disc or table is provided, with a number of cavities disposed around the common centre, and sliding over a fixed bottom plate. The cavities pass successively under the mouth of a hopper, where they are filled, thence under the mouth of a pipe in communication with a reservoir of compressed air, at the same time that the bottom plate at that place exposes a perforated surface, allowing the liquid to be driven through. As the motion of the table continues the dry granulated matter is either allowed to escape through an open portion of the bottom plate, or it may be retained for further treatment. The movement of the table is intermittent, and the same first motion shaft also regulates the tap for the admission of compressed air, steam, or liquid.—B.

*Improvements in Dyeing Apparatus.* E. Gessler, Metzingeo, Germany. Eng. Pat. 7841, April 18, 1893.

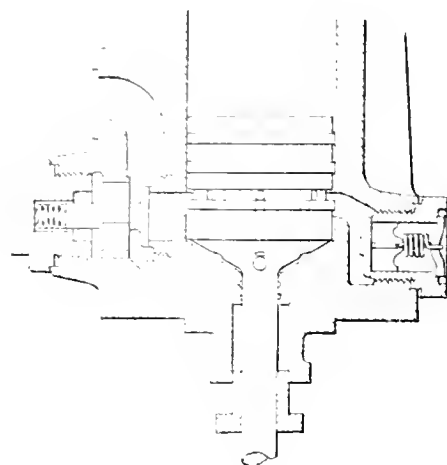
See under VI., page 390.

*Improvements in Pumps for Pumping or Compressing Gases or Vapours.* T. R. Murray, Glasgow. Eng. Pat. 7912, April 19, 1893.

THE object of the invention is to reduce the clearances at the end of cylinders of pumps employed for the compression of air or gases, and is applicable principally to the lower ends of inverted cylinder compressors, as shown in the illustration.

THE end of the cylinder is bored out below the passages or ducts for the air, and is preferably finished conical, the space being filled with oil. The lower end of the piston being provided with a block fitting into the said space and having a number of vertical holes ending in an annular recess, the oil is driven out into the passages behind the

valves, displacing every particle of gas left therein as the piston descends.



—B.

*Improvements in or connected with Apparatus for Cooling Liquids.* W. H. Webb, Bootle, Lancashire. Eng. Pat. 8125, April 22, 1893.

The apparatus comprises a vertical rectangular box with corrugated sides connected by end plates. The milk or other liquid to be cooled is distributed by means of a hopper at the top, and provided with a perforated double conical bottom. The liquid falling down over the upper halves of the corrugated sides is cooled by means of water circulating inside the casing. The lower half of the box is provided with a battery of coils, through which expanded ammonia is forced, the space between the pipes and the corrugated surfaces being filled with brine, kept in circulation by means of a pump or motor. The milk falling over these surfaces is thereby considerably cooled, and is afterwards collected by any suitable arrangement. In cold weather the cooling by ammonia may be dispensed with and the operation conducted with water alone.—E. G. C.

*A New and Improved Method of Connecting Joints in Sanitary Earthenware and other Articles.* A. and F. G. Johnson, Hailey, Staffordshire. Eng. Pat. 9765, May 16, 1893.

The pipes are of the spigot and socket type, lipped at their extremities, and the joint is made by means of an annular elastic packing of round or nearly round section compressed in between the spigot or nozzle and the socket. The spigot may be corrugated to prevent the packing ring from slipping out of place. This form of joint allows considerable play, which is of great advantage in sanitary work.

—E. G. C.

*Improvements in or connected with the Rolls of Machinery for Mixing and Calendering India-rubber or other Materials.* A. G. Brookes, London. From J. E. Davis, Lynn, Mass., U.S.A. Eng. Pat. 21,095, November 7, 1893.

The rollers of the machines used in the mixing of india-rubber compounds and in other operations involving chendering, rapidly become so hot when at work through friction that it is often necessary to stop the machinery for some time to allow them to cool. To obviate this, they have been cast with hollow spindles, so that water could be circulated through them, but owing to their large diameter (14 in.—16 in.) this device is not satisfactory. The inventor now proposes to cast them with a series of longitudinal passages placed close to the circumference of the

roller, fed with water from the ordinary central channel, or alternatively he arranges the circumferential passages in a helical direction.—F. H. L.

*Improvements in the Construction of Filtering Apparatus.* S. Allen, London. Eng. Pat. 23,210, December 2, 1893.

The filter is produced by pouring paper pulp over a perforated disc placed near the bottom of a vessel. After allowing the fibres to settle, the block is covered with another disc of textile material so as to prevent disturbance.—B.

*Process and Apparatus for Cleansing Filtering Material.* H. Reiser, Cologne, Germany. Eng. Pat. 24,526, December 20, 1893.

This invention applies to sand filters, the cleansing, by reversing the current of water, being assisted by air being blown under the perforated plate which supports the sand, in order to cause movement and agitation of the particles, thereby enabling the impurities to be more easily carried away.—B.

*Improvements in the Method of and Apparatus for Determining the Density of Gases.* G. Frères and M. Meslans, both of Paris, France. Eng. Pat. 70, January 1, 1894.

The method and apparatus are sufficiently described in this Journal, 1894, 64. It is claimed for the apparatus that "it indicates the density and the composition of gases in a continuous manner, and at each moment of production." The apparatus is also said to permit, in the special case of combustion and by means of the method, of the regulation of this combustion and of the admission of air to the furnaces of steam boilers, furnaces, &c. It can also, it is stated, be easily arranged so as to act in a mine containing fiery gases.—E. G. C.

## II.—FUEL, GAS, AND LIGHT.

*Processes for Manufacturing Gas from Oil, with Special Reference to the Peebles Process.* W. Ivion Macadam. Abstract of lecture delivered before the North of Ireland Association of Gas Managers, August 1893.

THE Peebles process essentially differs from any of the other oil-gas processes; for, whilst these involve one direct heat action, the Peebles process, by means of a lower heat and constant return circulation of the condensable products, only gives a partial breaking up at each circulation. In the newer forms of apparatus, as at Galashiels, the coal-gas and oil-gas retorts are back to back, and the waste heat of the former serves to heat the latter. The retorts need not, however, be so placed, for at Peebles the coal-gas and oil-gas are made in the same oven, but for economical working the Galashiels setting should be best. The coal-gas retorts are of the usual  $\Omega$ -shape and size, and set in the ordinary way, whilst the best form of retort for the oil-gas is cylindrical. The Galashiels retorts are 8 ft. long and 27 in. internal bore, and they are laid with a backward inclination. From the front of the retort there is an ordinary ascension, bridge, and dip-pipe to the hydraulic main. The condensers are nearly horizontal, with a slight upward tendency, and lead to a scrubber on the principle adopted regularly in shale-oil works. The trays in the scrubber are fed with oil from a cistern attached to the upper part of the apparatus. From the scrubber the gas passes to the ordinary dry purifiers, where it meets with the coal-gas, and the mixed gases then go to the holder in the usual manner. The oil to the retort is obtained from what is called the compensating tank. In this tank the overflow from the hydraulic main, the liquids from the condensers, and the fresh oil are intermingled, with the result that a perfectly liquid substance is maintained throughout the whole working. Moreover, the apparatus, being of considerable

size, acts as a settling-tank, in which any solid carbonaceous matter is removed. The oil passes from the compensating tank by means of a pipe with regulating valve to the ascension pipe, where the introduction of the liquid oil tends to wash the ascending gas and remove any suspended carbonaceous particles. By this circulation method liquid residuals are avoided, and the residue left in the retort, when it is coked down, is a dense and very pure carbon with less than 0.5 per cent. of ash and readily burned. For this solid residue 20s. per ton has been regularly obtained at Peebles. It yields a smokeless flame of great heat intensity. The gas yielded by this method runs with "blue" oils about 85 to 86 cubic feet per gallon, and the illuminating value when the gas is burned by itself is about 59-candle power. When burned in admixture with poorer illuminating gas the value is much greater. A month's actual working yielded—

Coal-gas .....	1,217,000 cubic feet of 18-candle power.
Oil-gas .....	253,400 "
Total gases ....	1,671,000 " 30.71 "
	Cubic Feet. Candles.
Coal-gas...	$1,217,000 \div 5 = 243,500 \times 18 = 4,383,360$
Total gas...	$1,671,000 \div 5 = 334,200 \times 30.71 = 8,976,013$

Therefore candle power from 253,400 cubic feet of oil-gas is 4,592,682 candles, which, divided by the make (253,400 cubic feet) = 18.12 candles per cubic foot, and  $\times 5 = 90.60$  candle power as the true illuminating value of the oil-gas as an enricher. This gain in illuminating value by mixing is equal to 53.74 per cent. To explain this gain we need to consider several points. If in the gas flame the heat, in this case derived from the burning of the hydrogen, carbon monoxide, &c., be sufficient to produce a great enough temperature the maximum of light will result. If these gases are too small in amount then the flame becomes smoky and the light less. In oil-gas the last condition holds good, and the balance is brought about by the admixture with a poorly carbonaceous gas containing much hydrogen, and which on burning gives sufficient heat to render the excess carbon of the oil-gas incandescent. In other words, the excess of carbon in the oil-gas is rendered incandescent by the excess of heat-producing bodies in the poorer gas, and the result is an excess of light over that obtained from either gas alone.

*Conditions for the Production of Generator Gas observed during Combustion of Carbon in Air.* R. Ernst. J. Prakt. Chem. 49, 31—45.

COKE was coarsely powdered, freed from dust by sifting, and heated to a very high temperature in a hessian crucible to remove hydrocarbons. The material thus obtained was burnt in a current of air passing through a porcelain tube heated in an ordinary combustion furnace. The temperature of the tube was ascertained by means of Le Chatelier's thermo-electric pyrometer. The gases formed during the passage of the air over the coke, were absorbed and determined.

The length of the layer of coke, the rate of passage of the air, and the temperature, were independently varied in the experiments. It was found that the composition of the gaseous products of combustion depend almost entirely on the temperature. The formation of carbonic oxide and carbonic anhydride begins at about 100° C., the production of the latter increasing rapidly up to 700°, when it forms about 20 per cent. of the gases collected. The amount of carbonic oxide at this temperature is still small, being only about 3 per cent. As the temperature continues to rise, however, the carbonic oxide increases in amount at the expense of the carbonic anhydride, and at 1,000° it forms one third of the gases, the remainder being nitrogen from the air. It was shown that at this temperature carbonic oxide is the direct product of the combustion, for carbonic anhydride could not be entirely reduced to carbonic oxide under similar conditions of experiment.

Since carbonic oxide is the only oxidation product at 1,000°, generator gas should be made at about that temperature. If high temperatures are desired, the carbon should

not be supplied with all the air necessary for its complete combustion, but only with that proportion sufficient to form carbonic oxide, which should then be burnt with a further supply of air.

The experiments explain why carbon burns at a moderate heat (730° C.) without a flame, but at a brighter heat (1,000°) with a flame.—J. W.

*Valuation of Fuels.* F. Fischer. Zeits. ang. Chem. 1893, 677.

See under XXIII., page 420.

## PATENTS.

*Improvements in Horizontal Coke Ovens.* F. Brunck, Dortmund, Germany. Eng. Pat. 4475, March 1, 1893.

This invention relates to the arrangement of the heating channels or flues in horizontal coke ovens with vertical wall-heating channel systems.

In ordinary coke ovens of this kind, constructed for recovering by products, the upper part of the charge receives less heat than the lower, with the result that the coking period is longer, and the quality of the coke inferior in the upper part.

The improvements consist in the arrangement of the upper horizontal channels connecting the vertical flues in ovens with double vertical-heating flues, and in horizontal ovens, with single or double vertical-heating flues, in making the walls of the oven chambers and vertical flues of varying thickness so as to equalise the heating effect. Details are given of the arrangement of the flues, &c.—B. B. P.

*A Process for the Manufacture of Oil-Gas.* J. Moeller, London. Eng. Pat. 8510, April 27, 1893.

This invention relates to the manufacture of oil-gas from ordinary petroleum, shale-oil, or other oil, or any fatty substance that can be melted. A vessel is charged to a high pressure with some combustible gas, which is used for operating an injector for forcing the oil, or melted fat, into a highly heated retort. The retort is partially filled with some porous refractory material. The spray of oil is directed into one end of the retort, and, passing over and through the porous refractory material, becomes converted into gas.—R. B. P.

*Improvements in Washers for the Extraction of Tar, Ammonia, Carbonic Acid, and Sulphuretted Hydrogen, from Coal-Gas.* F. S. Cripps, Sutton, Surrey. Eng. Pat. 14,783, August 2, 1893.

FIG. 1 shows a vertical section, and FIG. 2 a plan, with the top plate removed, of the washer. A is a horizontal plate with annular rings B east on its lower side; to these are attached rings C of thin sheet iron with their lower edges serrated, the form of which is shown by FIG. 3. A distributing box D projects inwards towards the centre. It has openings E on both sides, which communicate with the annular spaces formed by the rings on A. In A are openings F. When working, the washer is filled with liquor until the edges of the rings C are sealed, the level being regulated by the overflow valve J. Gas entering at H is distributed by the box D into the annular spaces L. It depresses the liquor in them and forces its way out past the serrated edges C, issuing in streams of fine bubbles, and is thus brought into intimate contact with the washing liquor. It then passes up through the openings F into the gas space above A, and out at I. Tar collecting in the lower part of the washer is continuously withdrawn through the overflow K. This consists of two concentric tubes, the outer one of which is fixed, and communicates at its lower

end with the bottom of the washer, where the tar collects, and at its upper end with the gas space above A. The inner tube is adjustable in height. The tar enters and rises

Fig. 1.

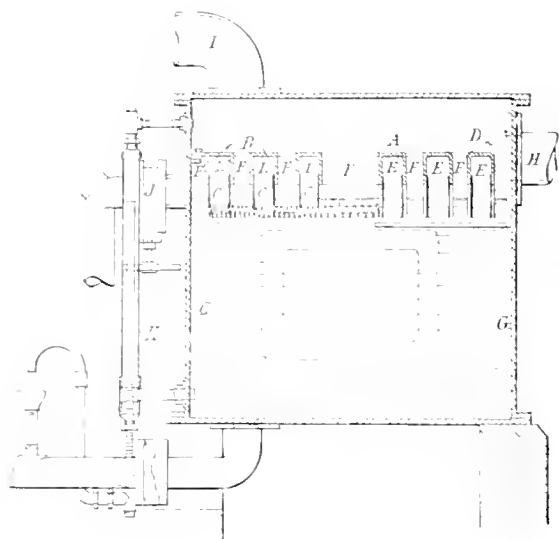


Fig. 2.

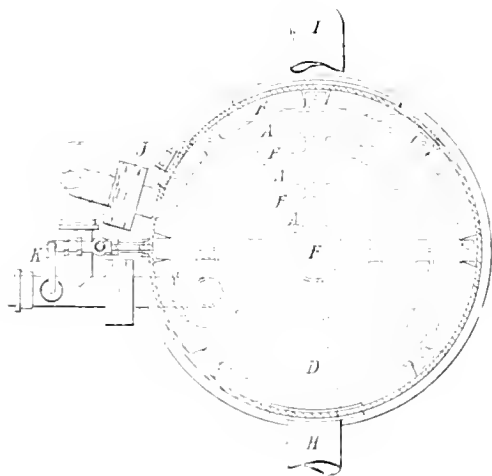


Fig. 3.



up the outer tube until it reaches the top edge of the inner one, into which it flows, and runs out through the siphon attached to its lower end.—R. B. P.

*Improvements in or relating to the Vaporisation of Petroleum and other Liquids.* A. J. Boulton, London. From S. C. Penchen and P. Clarke, both of Toronto, Canada. Eng. Pat. 340, January 6, 1894.

These inventors propose to vaporise liquids by means of heat generated electrically in two carbon plates. The liquid is contained in a closed vessel, and the carbon plates suspended in it from a float. In place of the carbon plates, wire resistance coils may be used.—R. B. P.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Note on Stearin Pitch.* E. Donath and R. Strasser. Chem. Zeit. 17, 1788.

In the distillation of the fatty acids used in the manufacture of candles, from 2 up to 7 per cent. of a tarry residue remains behind in the still, which on redistillation with superheated steam at 300° C. leaves a residue of stearin pitch in the form of a black, asphaltic mass, amounting to from 2 to 3 per cent. of the original fatty acids. At present this stearin pitch is used at Ronbaix for the production of oil gas. About 21·6 per cent. of the pitch is soluble in absolute alcohol, forming a fluorescent and faintly acid solution. Of saponifiable matter about 9 per cent. is present. On distillation in a spacious retort about 80·5 per cent. distils off, about 19·5 per cent. of solid, carbonaceous matter remaining behind. On distilling the distillate the following fractions were obtained:—

	Per Cent.
1. From 50° to 150° C. ....	10·3
2. From 150° to 200° C. ....	8·8
3. From 200° to 250° C. ....	10·0
4. From 250° to 300° C. ....	15·0
5. From 300° to 350° C. ....	25·9
6. Above 350° C. ....	29·2

The first fraction contains acrolein, the other fractions, the specific gravity of which varies from 0·7861 to 0·8877, are of yellowish-brown colour and are all strongly fluorescent. Fractions 5 and 6 have an average specific viscosity (water = 100) of 3·26, and at temperatures below 10° C. considerable quantities of hydrocarbons of the nature of vaseline separate from them. These two fractions, which represent 55 per cent. of the total distillate, are doubtless suited for use as lubricants, and the stearin pitch itself, preferably after saponification of the free fatty acids, may be found useful for thickening of lubricants. The formation and composition of stearin pitch is of interest with regard to the formation of petroleum, inasmuch as it furnishes some additional support for Engler and Höfer's theory of the formation of petroleum from fatty acids.

—C. O. W.

*The Carbonisation of Wood.* J. Sartig. Chem. Zeit. 17, 1269.

RELIABLE data on the yield of products resulting from the destructive distillation of various kinds of wood on a large scale are scarcely to be found in chemical literature. (See E. T. Chapman, Chem. News, 1871, 91—93, and Watson Smith, J. Chem. Soc. 1871, 1101—1108; also Muspratt's and Spon's Dictionaries, and Ramsay and Chorley, this Journal, 1892, 395 and 872). The author communicates the results obtained in working with upright portable wrought-iron retorts of 12 mm. thickness of plate, a height of 3·1 metres, and a diameter of 1·25 metres. These retorts were charged cold and transferred to the furnace by means of a crane, this method being preferable to the employment of horizontal retorts, on account of the smaller production of charcoal waste.

3,647 kilos. of beech wood (from logs not less than 10 centimetres thick), split into pieces of equal thickness and seasoned for two years, formed the total charge of three retorts working simultaneously. 15 hours' heating to a temperature of about 350° C. completed the distillation. The yields of the single retorts differed from each other, probably in consequence of unequal heating. Thus the quantity of tar and acid amounted to 553·09, 582·65, and 532·70 kilos., that of charcoal to 378, 359, and 385 kilos. respectively. In spite of the varying quantity, the pyroligneous acid was in all cases equally strong, possessing a specific gravity of 1·039 at 15° C. and containing 11·78 per cent. total acid (calculated as acetic acid). The yield of the three retorts taken together was:—

	Kilos.
Pyroligneous acid	1,448·44
Tar	220
Charcoal	1,122 (of which 89 kilos. of dust).

The total consumption of coal (medium quality) amounted to 419 kilos.

The pyroligneous acid was found to contain 2·946 per cent. of absolute wood-spirit. The tar had a specific gravity of 1·080, and yielded, on distillation from an ordinary cast-iron still, per 100 kilos.:—

	Kilos.
1. Crude wood acid	20·1 (giving 0·656 kilo. absolute wood-spirit and 2·94 kilos. calcium acetate).
2. Light tar oil	5·1 (sp. gr. 0·970).
3. Heavy oil	12·0 ( " 1·043).
4. Pitch	61·0
5. Gases	1·5 (by difference).

A charge of birch wood (cut only one month previously), freed from bark and weighing 3,209 kilos., produced 1,731 kilos. of acetic acid, sp. gr. 1·010 (11·82 per cent. acid), containing 1·5 per cent. absolute wood-spirit (laboratory determination only), and 177 kilos. tar, sp. gr. 1·078. The yield of tar and charcoal shows no material difference on comparison with that obtained from beech wood, while the yield of acetic acid is somewhat better, and that of wood-spirit smaller. Products from birch wood (the charcoal more especially) fetching a better price in the market, a preference should be given to this wood wherever its cost, inclusive of the expense of the removal of the bark, does not exceed that of beech wood.—F. M.

*The Distinction of Birch- from Pine Tar.* E. Hirschsohn. Pharm. Zeits. Russl. 1893, **32**, 657.

See under XXIII., page 423.

*The Electrical Excitation of Petroleum Spirit (Benzine).* R. Kissling. Chem. Zeit. **17**, 1773.

See under V., page 385.

## PATENT.

*Improvements relating to the Distillation and Breaking-up of Liquid Hydrocarbons and similar Substances, and to Apparatus therefor.* F. W. Clark, South Norwood Hill, Surrey. Eng. Pat. 4510, March 1, 1893.

This invention relates to the distillation or breaking up of liquid hydrocarbons and to the separation and recovery of the resulting products, "the distillation and breaking up" being effected by a method "in which air or other gas is passed over the liquid in an evaporator or retort."

The process consists essentially in introducing the liquid in thin streams into suitable evaporators, in which the more volatile parts are driven off by heat, or by reducing the pressure, or by both together. The vapours are then forced into a cooler by aid of a "carrier," such as air, and are there subjected to cold, or an increase of pressure, or both together, which causes them to condense. By varying the temperatures and pressures in the evaporator and cooler, products of higher or lower specific gravity can be obtained at will.

The evaporator consists of a closed cylinder slightly inclined from the horizontal. Two tube-plates, one near each end of the cylinder, divide it into three compartments. The front compartment is divided into halves by a horizontal plate. Between the two tube plates run a number of tubes parallel to the axis of the cylinder, which connect the two end compartments. In the front compartment the division plate divides the tubes into two sets, an upper and a lower one.

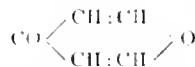
Into the lower set the liquid to be evaporated is admitted and its more volatile components driven off as it passes along them, by heat, which is applied to the exterior of the tubes by admitting hot air, water, or steam to the central compartment, through which the tubes pass. At the same time the air, or other carrying medium, which is admitted to the lower half of the front compartment, passes along the lower set of tubes to the back compartment and returns to the upper half of the front one along the upper set of tubes, carrying with it any vapour driven off from the liquid under treatment. This vapour and the "carrying" medium are then forced into the cooler by any suitable means. The cooler is similar in construction to the evaporator, only in it, instead of being subjected to heat and a diminished pressure, the vapours are subjected to cold and an increase of pressure, which causes them to condense.—R. B. P.

## IV.—COLOURING MATTERS AND DYES.

*Chrysin.* G. Darier. Ber. **27**, 21—22.

RÉCARD discovered a yellow colouring matter in the buds of the poplar, termed chrysin,  $C_{15}H_{10}O_4$ , which Graebe believed to have a constitution similar to that of euxanthone. The author succeeded in getting a yield of 2·5 to 3 grms. of chrysin from 1,000 grms. of the poplar buds.

Evidence was obtained, through the formation of a di-acetyldinitrochrysin, of the presence of two hydroxyl groups in chrysin, such as have been shown to be present in euxanthone; also the general similarity of the two bodies lends further support to the belief of the presence in both of a pyrone ring—



Euxanthone is derived by the action of heat or of sulphuric acid on euxanthic acid, the magnesium salt of which is the chief constituent of the well-known water-colour pigment *Indian yellow*.—W. S.

*Salicylic Acid Azo Colouring Matters. A Study on the Influence of Constitution on Fastness.* M. D. Bona. Farb. Zeit. **5**, 120—121.

THE author arrives at the following conclusions with regard to colouring matters containing salicylic acid:—

(1.) Salicylic acid, by its presence in an azo-dyestuff, influences the shade of that dyestuff in the direction of yellow.

(2.) Salicylic acid tends to increase the fastness to light.

(3.) Owing to the close connection of the carboxy- and hydroxy-group; in salicylic acid, it yields dyestuffs which are generally fast to alkalis. But this is not the case if some component sensitive to alkalis is closely combined with the salicylic acid. The behaviour of resorcinol needs further explanation.

(4.) Salicylic acid yields colouring matters which are fast to acids, unless some basic group is present in the molecule.

(5.) No dyestuff containing salicylic acid appears to be fast to chlorine.—R. B. P.

*The Electrolytic Reduction of Indigo.* J. Mullerus.  
Chem. Zeit. **17**, 1454.

*See under Vl., page 387.*

*The Reduction of Indigotine to Indigo White.*  
F. Goppelsroeder. Chem. Zeit. **17**, 1633.

See under VI., page 388.

*The Estimation of Indigotine in Indigo.* C. Donath and R. Strasser. *Zeits. f. ang. Chem.* 1894, 49.

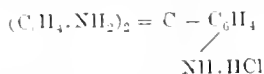
See under XXIII., page 426.

*The Synthesis of Azo Colours on Animal Fibres.* J. Pokorný. With Report on the same by P. Werner. Bull. Soc. Ind. Mulhouse, 1893, 282-287.

*See under VI., page 388.*

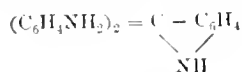
*The Constitution of the Colouring Matters of the Magenta Group.* Prud'homme and C. Rabaut. Bull. Soc. Ind. Mulhouse, 1893, 272—277.

For some years Rosenstiel has advocated the theory that the colouring matters of the magenta group are in reality the ethers of aromatic amido alcohols, and that the formula, for instance, of para-magenta instead of being written—



should be  $(C_6H_4.NH_2)_3.C.Cl$ , i.e., that the chlorine is directly united to the central carbon atom. To throw light on this question the authors have made researches as to the influence of ammonia and other agencies on these colouring matters.

**Action of Gaseous Ammonia on Magenta.**—When ammonia is passed in the cold over dry finely-powdered magenta (rosaniline hydrochloride), the green metallic crystals change to a violet powder, which finally becomes red. The violet powder corresponds to the absorption of one molecule of ammonia, the red to two molecules. On heating the new compound ammonia is liberated and magenta regenerated. The ammonia is also liberated *in vacuo* in the cold. If magenta were the hydrochloride of a secondary amine, as the older formula would indicate, it would form with ammonia, ammonium chloride and—

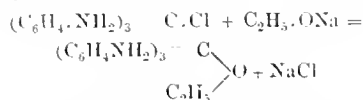


but this reaction does not take place.

**Decomposition of the Chlorides and Iodides of Amines by Ammonia.**—To show more conclusively that the effect of ammonia on the hydrochloride of an amine would be the production of the amine and ammonium chloride, experiments were made with haloid salts of various primary and secondary amines, the result being invariably that indicated. The salts of tertiary amines are without interest, being very unstable and readily decomposed by water.

*Compounds formed by Ammonia and the Colouring Matters of the Magenta Group.*—Rosaniline sulphate absorbs ammonia less readily than the hydrochloride; under its influence, however, it takes up two ammonia molecules, becoming brownish-black and finally dark red. The oxalate exhibits similar changes. Rosaniline violet (hexamethylosaniline) becomes indigo blue, and with two ammonia molecules violet-grey. Malachite green (oxalate) contains an excess of oxalic acid, and the ammonia first combines with this and then with the colouring matter to form a reddish-grey product. The increase of weight when ammonia acts on this dyestuff varies with the amount of free oxalic acid present, and might serve for a method of quantitative estimation of this free acid.

*Action of Sodium Ethylate on Magenta.*—Sodium ethylate completely decolorises magenta in alcoholic solution. If magenta were the hydrochloride of a secondary amine the anhydrous base would be formed, but as this hypothesis has been proved incorrect, the reaction is as follows :—



This ether is so unstable that it is decomposed on adding water, into alcohol and rosaniline, the latter being thrown down as a colourless precipitate, which, on exposure to air, acquires a pink shade.—R. B. B.

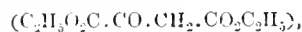
## PATENTS.

*Improvement in the Manufacture of Refined Indigo.*  
S. J. Simpkin, Leeds. Eng. Pat. 1113, January 18, 1893.

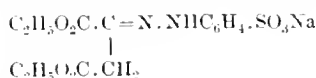
The crude indigo as imported is treated by any of the known methods for reducing and dissolving the indigo, in a closed jacketed vessel provided with an agitator, and so arranged that the operation can be conducted out of contact with the air, and in the presence of a non-oxidising gas, such as coal gas, nitrogen, hydrogen, or a mixture of any of them. As an example of the method employed, the following details are given. After filling the vessel with gas a mixture of 100 lb. of raw indigo finely ground in water, 200 lb. of ferrous sulphate dissolved in water, and 80 lb. of lime made into a thin sludge, is run through a siphon pipe into the apparatus, thoroughly mixed by means of the agitator, and the vessel is filled with hot water to dissolve the indigo white. The solution is then separated, and the indigo blue precipitated by oxidation. Other reducing agents may be used, and the proportions may be varied. A sectional drawing of the apparatus accompanies the specification.—T. A. L.

*Manufacture of New Colouring Matters by the Action of  
Diazo Compounds on the Hydrazone Derivatives of  
Orallygluetic Ether.* J. H. Ziegler, Bâle, Switzerland.  
Eng. Pat. 5693, March 16, 1893.

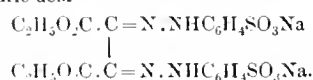
THE colours produced according to this patent are ethers of the tartrazines (Eng. Pat. 9858 of 1885; this Journal, 1886, 427). The method employed for their preparation is based upon the observation that the hydrazones of oxalylacetic ether are capable of combining with diazo compounds, the hydrogen of the methylene group being replaced by the hydrazone group, forming an osazone. If the colouring matter, produced as described below, by the interaction of phenylhydrazine sulphonic acid, oxalylacetic ether, and diazobenzene sulphonic acid, be heated with the calculated quantity of caustic soda, it is converted into tartrazine. About 19 kilos. of phenylhydrazine sulphonic acid, 18·8 kilos. of oxalylacetic ether—



and 15 kilos. of crystallised sodium acetate are mixed with 100 litres of water, agitated, and heated to 50° C., when the hydrazone compound is produced—



After cooling, 5 kilos. of sodium carbonate are added and subsequently the diazobenzene sulphonic acid prepared by diazotising 22 kilos. of sodium sulphanilate with 7 kilos. of sodium nitrite and 22 litres of 33 per cent. hydrochloric acid in 100 litres of water. The solution turns an intense yellow, owing to the formation of the oxazine of the ether of dioxystartic acid—

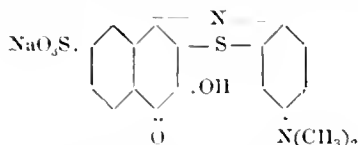


The dyestuff can be separated by adding salt, and, as described above, on heating with caustic soda it can be saponified. The phenylhydrazine sulphonic acid can be replaced by the tolyl-, xyl-, or naphthyl-hydrazines, or their sulphonic or carboxylic acids or their nitro-, hydroxy-, or ethoxy-derivatives, and similarly for the sulphonic acid may be substituted, aniline, the toluidines, the xylinines, the naphthylamines, benzidine, or tolidine, and their sulphonic or carboxylic acids, and nitro- and amido- derivatives, and compounds like amido-azobenzene may also be employed.

—T. A. L.

*The Production or Manufacture of Colouring Matters.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 6035, March 21, 1893.

AN extension of Eng. Pats. 825 and 4757 of 1893 (this Journal, 1894, 31 and 245), which refer to the production of blue mordant colouring matters from  $\beta$ -naphthoquinone sulphonic acid or  $\beta$ -naphthohydroquinone sulphonic acid and the nitroso derivatives of secondary or tertiary amines in presence of thiosulphates, or else from certain sulphonic acids of  $\beta$ -naphthoquinone and alkylated *p*-phenylenediamine thiosulphonic acids. It has now been discovered that it is not necessary to prepare the naphthoquinone or naphthohydroquinone sulphonic acids, but that the nitroso-naphthols and nitrosonaphthol sulphonic acids can be used. For instance, the same dyestuffs are obtained by taking instead of 1-2-4 naphthoquinone sulphonic acid, either 2-1-4-nitrosonaphthol sulphonic acid, or 1-2-nitrosonaphthol, and reacting with them on the condensation products from the nitroso-derivatives of secondary and tertiary amines and thiosulphates. It is supposed that during the reaction the nitroso group is reduced to the amido group, and this is subsequently replaced by oxygen. The following example is an instance of the method employed. A solution of 9.3 kilos. of nitrosodimethylaniline hydrochloride and 18 kilos. of 33 per cent. acetic acid in 300 litres of water, is mixed with 26 kilos. of sodium thiosulphate dissolved in hot water and the mixture is heated until the liquid becomes colourless, when the formation of the dimethyl-*p*-phenylenediamine sulphonic acid is complete; 1.4 kilos. of sodium 1-2-6-nitroso-naphthol sulphonate are then stirred in gradually. The dyestuff begins to form at once and separates in small bronze-coloured crystals. It is filtered off whilst hot and is sufficiently pure for dyeing purposes. The colouring matter is identical with that obtained from nitrosodimethylaniline sodium thiosulphate and  $\beta$ -naphthoquinone 6-sulphonic acid. It probably has the following formula:—



Other nitroso derivatives may be substituted for those mentioned, and in general the dyestuffs claimed are formed by the interaction of the condensation products from the nitroso derivatives of secondary or tertiary aromatic amines or substituted amines and thiosulphates on the nitroso derivatives of the naphthalene series, or on the quinones corresponding to such nitroso derivatives, excepting certain  $\beta$ -naphthoquinone sulphonic acids already described in Eng. Pat. 4757 of 1893 (this Journal, 1894, 245).—T. A. L.

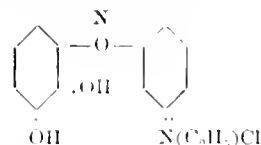
*The Manufacture or Production of Colouring Matters.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 6035A, March 21, 1893.

THIS is an extension of Eng. Pat. 825 of 1893 (this Journal, 1894, 31), according to which blue mordant colouring matters were produced by condensing  $\beta$ -naphtho- or  $\beta$ -naphthohydroquinone sulphonic acid with the nitroso derivatives of secondary or tertiary amines in presence of

thiosulphates. As already described more fully in the preceding specification (see above), these quinones can be replaced by the corresponding nitroso-naphthols, and the colouring matters obtained are identical. A mixture of 9.3 kilos. of nitrosodimethylaniline hydrochloride and 14 kilos. of sodium 1-2-6-nitroso-naphthol sulphonic acid, is dissolved in 300 litres of water mixed with 18 kilos. of 33 per cent. acetic acid, and then with a hot solution of 26 kilos. of sodium thiosulphate in 100 litres of water. The formation of the dyestuff commences immediately and the colouring matter separates in bronzy crystals, which are filtered off whilst hot. The dyestuff is identical with that described in the preceding abstract.—T. A. L.

*The Manufacture or Production of a New Blue Colouring Matter.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 7181, April 7, 1893.

THE blue-violet dye-stuff obtained from nitrosodimethylaniline hydrochloride and gallamine, according to Eng. Pat. 2941 of 1889 (this Journal, 1889, 981), is insoluble in water and has to be treated with sodium bisulphite in order to utilise it for dyeing or printing purposes, when it is known as "*Gallamine Blue*." The present specification describes the preparation of the higher homologue of this substance from nitroso-diethylaniline, and the product obtained has valuable properties. It dissolves easily in water, dyes evenly, and a purer blue than any of the known oxazines. It stands furling equally as well as gallamine blue, is fast to light, and is unaltered by alkalis, dilute acids, or chlorine. It is prepared by heating 1 kilo. of gallamine with 1.5 to 2 kilos. of nitrosodiethylaniline hydrochloride in 1 kilo. of alcohol until a test dissolves in water with a pure blue colour. The melt is then poured into 10 litres of water and a small quantity of hydrochloric acid is added. After a short time the dyestuff separates as a crystalline precipitate. It has the formula—



and is analogous to "*Gallocyanine*."—T. A. L.

*The Manufacture or Production of New Alizarin Dyestuffs.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 7182, April 7, 1893.

THE process consists in methylating or ethylating  $\alpha$ -amido-alizarin and its homologues, such as  $\alpha$ -amido-flavopurpurin,  $\alpha$ -amido-anthrappurpurin, &c., by heating them in presence of alcohol with an alkyl haloid or other alkylating agent to a high temperature under pressure. The alkylated colouring matters produced yield bluer shades with mordants than the non-alkylated products. Thus  $\alpha$ -amido-alizarin gives garnet-red shades on alumina, whereas ethylated  $\alpha$ -amido-alizarin dyes a Bordeaux shade with a strong bluish hue on the same mordant. This product is obtained by heating 10 kilos. of dry  $\alpha$ -amido-alizarin, 10 kilos. of ethyl bromide, and 100 kilos. of ethyl alcohol in an autoclave for five hours to 150° C. The excess of acid is then distilled off and the residue is extracted with dilute soda lye, from which solution, after having been heated to boiling, the dyestuff is precipitated by adding an acid, filtered off and washed to remove acid. It forms a dark violet paste which dissolves in alkalis to a bluish solution and in hot concentrated hydrochloric acid to a yellow solution, from which the hydrochloride partially separates on cooling. Its solution in concentrated sulphuric acid is yellower than that of  $\alpha$ -amido-alizarin.

—T. A. L.



*Manufacture of Colouring Matters.* O. Imray, London. From "The Farbwerke vormals Meister, Lucius, and Brüning," Hoechst-on-the-Maine, Germany. Eng. Pat. 7185, April 7, 1893.

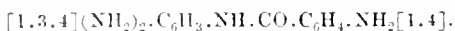
According to Eng. Pat. 19,820 of 1892 (this Journal, 1893, 922) a red colouring matter is produced by heating rhodamine with *m*-dinitro-chlorobenzene in alcohol. Similar, but yellower, colouring matters are obtained by condensing dinitro-chlorobenzene and *o*- and *p*-nitrobenzyl chlorides with the symmetrical diethyl- or dimethyl-*m* amidophenolphthaleines. The compounds thus obtained are less soluble in alcohol, acetic acid, and water than the corresponding tetra-alkyl colouring matters. They dissolve in 50 percent. acetic acid or in alcohol at the ordinary temperature, and in water on heating. Salt precipitates the colour from the aqueous solution in a flocculent condition, and the dry colouring matter forms a light yellowish-red powder having a green metallic lustre. The two following examples explain the methods employed:—(1.) 21 kilos. of diethyl-*m*-amido-phenolphthaleine hydrochloride, 8.6 kilos. of *p*-nitrobenzyl chloride, and 2.65 kilos. of calcined soda dissolved in 10 kilos. of water are boiled in 50 litres of alcohol for several hours. The alcohol is then distilled off and the colouring matter is dissolved in hot water and after filtration precipitated with salt. (2.) 19.3 kilos. of diethyl-*m*-amido-phenolphthaleine and 10.2 kilos. of dinitro-chlorobenzene are heated in 50 litres of alcohol for several hours, and after distilling off the alcohol the residue is extracted with a large quantity of boiling water containing a little hydrochloric acid. After filtering this solution the dyestuff is precipitated with salt.—T. A. L.

*Manufacture of a Novel Diamido-Base and of Colouring Matters from the same.* O. Imray, London. From "The Farbwerke vormals Meister, Lucius, and Brüning," Hoechst-on-the-Maine, Germany. Eng. Pat. 7187, April 7, 1893.

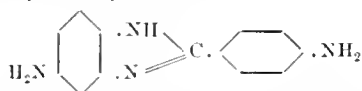
THE base referred to is obtained by nitrating benz-*p*-nitranilide, reducing the trinitro-compound obtained, and eliminating water from the triamido derivative. This produces *p*-diamido-phenyl-benzimidazole, which yields a tetrazo compound capable of combining with phenols and their derivatives, giving cotton colouring matters. The successive operations are as follows:—When benz-*p*-nitranilide is dissolved in five times its weight of sulphuric acid monohydrate and nitrated by a mixture of fuming nitric acid and monohydrate, it is converted into *p*-trinitro-benzanilide—



This compound is insoluble in water, alcohol, or ether, but soluble in glacial acetic acid or acetic ether. On reduction with iron and hydrochloric acid it yields triamido-benzanilide—



The triamine is soluble in hot water and crystallises out on cooling. It also dissolves in hot alcohol and acetone. Its salts are easily soluble in cold water. When heated *in vacuo* to 250° C. one molecule of water is split off and the following compound is produced:—



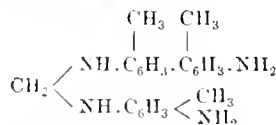
This anhydro base is insoluble in water or ether, soluble in alcohol and acetone, and its melting point is above 250° C. With sodium nitrite and a mineral acid it forms an easily soluble tetrazo compound, which readily combines with phenols, naphthols, their sulphonic and carboxylic acids, and with amido acids. The usual variations may be made in the combinations as with the Congo colouring matters. The colours are fast to soap and acids and the shades produced are brighter and bluer than those from the colouring matters obtained according to Eng. Pat. 10,667 of 1892 (this Journal, 1893, 515).—T. A. L.

*The Manufacture of Blue Colouring Matter containing Sulphur.* W. H. Claus and A. Rée, Droylsden, Manchester. Eng. Pat. 8221, April 24, 1893.

THE following is the preparation of methylene blue according to this specification. About 18 kilos. of dimethyl-aniline are converted into the nitroso derivative, which is filtered off, yielding about 26 kilos. of *p*-nitroso-dimethyl-aniline hydrochloride. This is dissolved in 150 litres of hot water and 120 kilos. of acetic acid (12° Tw.) and heated to 75° C. To this solution is added 75 kilos. of sodium thio-sulphate in 350 litres of water. When the yellow colour of the liquor has disappeared the temperature is raised to 80° C. and 120 kilos. of zinc chloride (102° Tw.), 15 kilos. of dimethyl-aniline, and 17 kilos. of hydrochloric acid (28° Tw.) are added. Immediately afterwards, a solution of 25 kilos. of sodium bichromate and 5 kilos. of sulphuric acid in 250 litres of water is run in, the temperature being raised to 90° C., and the whole being well stirred during the oxidation, which takes about three minutes. The colour, which is formed at once, is filtered off and purified by dissolving it in water and precipitating with salt.—T. A. L.

*Improvements in the Manufacture of a Diamido Base and of Tetrazo Colouring Matters derived therefrom.* W. G. Thompson and J. Moore, Middleton, Lancashire. Eng. Pat. 2232, February 1, 1894.

By heating together tolidine, formaldehyde, and the basic hydrochloride of *m*-tolylene diamine in alcohol on the water-bath for several hours, the next higher homologue of one of the products described in Eng. Pat. 20,301 of 1893 (this Journal, 1894, 32) is obtained. The melt is treated with hot dilute hydrochloric acid, the solution filtered, and the new base precipitated as a brown powder by adding ammonia. The following formula is assigned to it:—



When heated above 100° C. the base becomes viscous. It is not very soluble in alcohol, but the hydrochloride and sulphate are easily soluble in water. It is easily converted into the tetrazo compound, and the colouring matters claimed, which dye unmordanted cotton red, are obtained by combining one molecular proportion of the tetrazo compound with two molecular proportions of naphthionic acid or of 2-3'-naphthylamine sulphonic acid. Both these colouring matters dissolve in concentrated sulphuric acid to a blue solution.—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Wild Silk.* H. Silbermann. Färb. Zeit. 5, 19—21.

WILD silk is of constantly increasing importance, not only as a substitute for cultivated silk, but also as a fibre capable of definite applications, and for certain purposes unsurpassed. Wild silks are distinguished from cultivated by their chemical composition and morphological structure. In the raw condition they are dull, dark coloured, and consisting of a thick, double fibre. Tussah silk fibre shows longitudinal striations, due partly to fine air-canals within the fibre, and partly to the fact that each single fibre is composed of a number of excessively fine fibrils, easily seen under the microscope in a transverse section. This structure and the thickness of the fibre cause great tenacity. The diameter of wild silk fibre is often 60—70 thousandths of a millimetre, that of the cultivated fibre seldom exceeding 32 thousandths.

While ordinary silk is readily dissolved by a solution of zinc chloride at 15° B., tussah requires a solution at 60° B. It dissolves only with difficulty in ammoniacal solution of copper hydrate, and is less readily attacked by boiling hydrochloric acid than the cultivated product.

Wild silk cocoons are large and rich in silk; certain kinds measure over 120 mm. in length. The price is considerably below that of cultivated silk. Wild silk appears in commerce as a brownish fibre with a somewhat penetrating odour, due probably to a rotting process to which the cocoons are subjected before reeling. Certain kinds of wild silk are capable of being reeled, while others are treated like silk waste, being carded, combed, and spun.

The most important variety is Indian tussah (*Antheraea mylitta*). Several crops of cocoons are obtained in the year, the September crop being best suited for reeling. The cocoon is attached to a twig by a handle-like appendage. It is oval, regular in shape, and evenly spun. Its hardness is due to urate of soda, with which the caterpillar impregnates each layer as completed. The cocoon is 45 to 50 mm. in length, and 25–30 in breadth. When dry it weighs 2–3 grms., and contains 30 per cent. silk fibre. 1 kilo. silk is obtained from 10–15 kilos. of fresh or 3–5 kilos. dried cocoons. The length of the fibre is 1,200–1,400 metres, of which 600–700 metres can be reeled. The diameter varies from 50 to 90 thousandths of a millimetre. The increasing demand for tussah has led to the frequent admixture of this fibre with less valuable wild silks, *eg.*, *Antheraea fruhii*, *Ant. puphia*, *Ant. rivalica*, &c.

Chinese tussah (*Ant. pernyi*) in structure and chemical behaviour greatly resembles Indian tussah. The caterpillar lives upon the Chinese oak, and produces irregularly spun, soft cocoons, 10 mm. long by 25 mm. broad, having a small opening at one end. Reeling in the ordinary manner is impossible, since the cocoons sink in boiling water; they are therefore softened by steam in specially constructed baskets, and then reeled. They contain about 20 per cent. silk fibre. The length of the fibre is 650–700 metres, and its diameter 68 thousandths of a millimetre.

Wild silk is more hygroscopic than cultivated silk. The amount of moisture contained exercises great influence on the tenacity and elasticity of the fibre, increase of moisture diminishing the tenacity but increasing the elasticity.

—R. B. B.

*Connection between Tenacity of and Moisture present in Hemp and Flax.* C. A. Lobry de Bruyn. Chem. Zeit. 17, 1432–1433.

In a previous paper on this subject (Chem. Zeit. 17, 172–173; this Journal, 1893, 825) the author showed that the effect of increased moisture present in fabrics of hemp and flax is to increase their tenacity. This he has fully confirmed by subsequent experiments. The trials were made with strips of the fabrics cut according to the warp, and each number given is the mean of three to six estimations.

The results with four samples were:—

A.		B.		C.		D.	
Moisture.	Breaking Strain.	Moisture.	Breaking Strain.	Moisture.	Breaking Strain.	Moisture.	Breaking Strain.
Per Cent.	Kilos.	Per Cent.	Kilos.	Per Cent.	Kilos.	Per Cent.	Kilos.
0	178	0	189	0	143	9	147
4.1	197	2.2	190	3.9	162	2.2	153
7.5	243	5.5	232	6.4	193	5	179
9.8	277	9	288	8.8	235	6.7	205
12	315	12	350	10.7	255	11.2	250
13.5	378	15	402	13.3	290	13.3	265
26.4	405	19.1	417	22	320	31	290
..	..	35	425	38	325	..	..

From these results it is seen that with increase of moisture beyond 15 per cent. there is but very slight increase in tenacity. This result does not agree with that described in the previous paper, the difference being possibly due to a tighter twist of the thread in spinning the yarn in the former instance.

It is difficult to give figures which are absolutely exact, as it is found that so-called air-dried cloth contains, according to season of the year, from 6.5–12 per cent. moisture. The condition of the surrounding air during the experiments also affects the results. Different strips cut from the same fabric may show a difference of 5 per cent. in the breaking strain.

Hemp and flax are more hygroscopic than cotton; this may be due partly to difference of structure, partly to small quantities of pectic matters present in the hemp or flax. These substances may be present in scoured yarn even up to 10 per cent.—R. B. B.

*The Electrical Excitation of Petroleum Spirit (Benzene).* R. Kissling. Chem. Zeit. 17, 1773–1774.

In a letter on this subject the author states that in many chemical washing works it is customary to add a soluble soap to the petroleum spirit (benzene) in order to prevent the electrical excitation of this solvent. In some cases the soap is used all the time, in others it is only added when the crackling noise, which indicates the excitation, is detected. The fires that have not unfrequently broken out

in the chemical wash-houses, are attributed to extreme cases of the excitation referred to.—A. G. B.

*The Prevention of Petroleum Spirit Fires in Chemical Cleaning.* C. F. Göhring. Färb. Zeit. 5, 49.

When dried wool and silk are immersed in pure petroleum spirit (benzene), in the neighbourhood of an electroscope, lifted out and again immersed, a wide divergence of the leaves of the electroscope is observed. The amount of electricity may be even so great that it cannot be measured by the electroscope. If the experiment is conducted in the dark, electric sparks and flashes of light accompanied by crackling sounds are seen and heard, which may readily increase until the benzene is ignited. The method of prevention is to add to the benzene from 0.2 to 0.3 per cent. of a soap soluble in benzene, when the sparks and crackling at once cease and all danger is avoided.

It is well known that pure, freshly distilled benzene, in the cold, dry days of winter, is liable to take fire spontaneously, owing to the production of electricity. As soon as the atmosphere becomes warm or moist the conditions for carrying off the electricity, or for its total prevention, are secured.

In the first case mentioned possibly the soluble soap provides the necessary moisture, which by means of the soap is dissolved in the benzene, water being otherwise non-miscible with benzene.—R. B. B.

## VI.—DYEING, CALICO PRINTING, PAPER-STAINING, AND BLEACHING.

*Improvements in Bleaching and Printing.* Oesterr. Wollen- u. Leinen-ind. 1894, 14, 8.

As an improvement in calico-printing, the patent bowking process, already introduced into several printworks, is laid considerable stress on. The material is boiled in caustic soda-lye out of contact with the air, and an article is thus furnished which resembles half-bleached goods.

Reference is made to variegated goods, produced till recently for the most part in England, and it is stated that a strong effort is now being made to obtain appropriate shades that can be fixed on the material prepared with  $\beta$ -naphthol.

Success has been attained by printing with basic dye-stuffs with the addition of some tartaric acid and "tin salt," in obtaining effects similar to those obtained with paranitraniline red or naphthylamine Bordeaux.

After printing, the pieces are steamed, treated with tartar- emetic, and washed. As yet the whites in the case of paranitraniline red are unsatisfactory.

The discharge of direct-dyed colouring matters is carried out as heretofore; the extension of this branch is favoured by the fastness of the new direct-dyeing colours.

The discharge of basic colours progresses slowly; it is effected by means of caustic soda on the tannin-mordanted pieces, with subsequent treatment in tartar- emetic and dyeing with basic dye-stuffs. Chrome - discharged goods and resists with alizarin are produced in quantity. As suitable new colours, Alizarin green, Alizarin grenat, and Alizarin cardinal may be mentioned.

Repeated trials with the new judigo salt "Kalle" are in progress, though it would seem to be of interest for calico- printers only, on account of expense.—W. B. H.

*The Fixation of Uranium Oxide on Textile Fibres and the Application of Uranium Salts as Mordants for Dye-stuffs.* E. Odenheimer. *Färb. Zeit.* 5, 17—19.

**Uranium Yellow.**—This colour is produced by a method analogous to the author's process for "Golden purple" (*Bull. Soc. Ind. Mulhouse*, 1892, 529—539; this *Journal*, 1893, 432—443), the fabric impregnated with a uranium salt being subjected to pressure by a hot metallic surface. A bath containing 14 grms. uranium nitrate per litre gives an orange-yellow, more dilute solutions straw-yellow colours, while stronger solutions attack the fibre. The production of the colour depends on the formation of uranium hydroxide. It is affected by acids and alkalis.

**Uranium Brown.**—The fabric is first steeped in a hot solution of a uranium salt, then worked in either a solution of potassium ferro- or ferri- cyanide, or of gallic acid, hydro- quinone, tannic acid, or some tannin material. The brown obtained by the use of ferro- or ferri- cyanide is fast to acids, but destroyed by alkalis, while the colour produced by gallic acid, &c. is fast to alkalis, and destroyed by acids, the colour being restored by an alkaline solution.

These colours cannot be produced on cotton, and are darker on wool than on silk. They resemble very closely in appearance and properties the shades resulting from chromates and tannin materials, and in both cases there is little doubt that the colour is a true compound of tannin with uranium or chromium.

**Uranium Salts as Mordants.**—Experiments have been made with about 200 dye-stuffs, both as to the alteration of shade on the fibre, and the formation of a lake with the colour solution. Although a few results appear useful, these mordants do not possess advantages which would make them of general practical interest. The usual effect of uranium mordants is to change yellow to orange, orange to light red, blue or red to black, the colours being fast to soap and alkalis, but in most cases affected by acids.

In boiling wool with a 0.2 per cent. solution of uranium acetate, after one hour the wool has taken up about 30 per

cent. of the salt, and after 2—3 hours' boiling the solution is completely decolorised.

The author has been unable to make use of uranium compounds in the production of aniline black.—R. B. B.

*The Substantive Dye-stuffs in the Dyeing of Half-Wool Goods.* F. Ackerman. *Färb. Zeit.* 5, 33—34 and 52—54.

The author refers to two previous papers which have appeared on this subject by Lindemann (*Färb. Zeit.* 4 [7]) and Mecklenburg (*Färb. Zeit.* 4 [17]).

The numerous difficulties attending the use of the "direct cotton" colours for wool and cotton unions are due to three facts—(1) the entirely different behaviour of different dyes towards the same material; (2) the different behaviour of the same dye towards different materials; and (3) that the methods of dyeing differ greatly from those previously in use. Minor differences are also due to varying relative proportions of cotton and wool in the mixed fabrics.

As to the best method of dyeing, the author considers a boiling dye-bath indispensable, especially for thick and heavy goods. The best addition to the dye-bath is 20 grms. sodium sulphate per litre. Sodium bisulphate is not recommended.

According to results obtained on half-wool material the dye-stuffs may be grouped into six classes, as shown in the table on next page. In every case the dyeing was for two hours, boiling.

Most of these dye-stuffs tend to dye the cotton at first, the colour being only taken up by the wool after long boiling; hence if the time is less than two hours the division into groups will not be as above, e.g., a colour might appear in Groups 1 or 3 which is classed above in Group 2. The dividing lines between the various groups are of course not very clearly defined. The next question is, which groups of dyes are adapted for the dyeing of unions? That the dyes of the first group are so is evident, but the use of these alone does not afford a sufficient range of colours. Several of those in Group 2 are useful, viz., those which border closely on Group 1, such as Diamine Red NO, Benzopurpurin 4 B, Congo Red R, and Deltapurpurin 5 B, and, with a shorter time of dyeing, these may be counted in Group 1. The same applies to Chicago Blue. Or other dyes of this group may be used in mixtures with Groups 3 or 5. Diamine Searlet B and Diamine Fast Red F give good results at 70—80° C., the goods remaining after dyeing in the bath until cold. Diamine Fast Red F has the special advantage of great fastness to light. The dyes of Group 3 are applied together with Group 2 or with acid dyes for the wool, as mentioned later. Group 4 is unsuitable. Dyes of Group 5 are used in connection with acid dyes, and this is the case also with Group 6, the wool being shaded up to the cotton by the acid colour.

Sulphonyanin and Brilliant Sulphonazurin (Farbenfabriken), which dye the wool only, can be used with the blues of Group 5.

A number of acid colours can be applied to wool with the sole addition of sodium sulphate, though the bath is not thereby exhausted. Such colours are suitable for mixing with dye-stuffs dyeing cotton deeper than, or a different shade from, wool. Examples of these acid dyes are:—Azoflavine (B.A.S.F.), Croceine Orange (Kalle), Acid Violet 6 B (Bayer), Formyl Violet (Cassella), &c. Alkali Blue may be employed with those cotton colours unaffected by acids, if the goods are developed in a weak, cold, sulphuric acid bath. For shading are also useful:—Milling Red R (Cassella), Fast Acid Violet (ML and B), Rosinduline (Kalle), &c.

Every shade can be obtained by the various methods indicated except bright greens, blue-violets, and a brilliant black to compete in the matter of cost with logwood.

The following methods may be specially recommended:—

**Yellow.**—Oxyphenine alone, Direct Yellow with Diamine Gold or with Chrysophenine.

**Orange.**—Direct Orange with Croceine Orange.

**Red.**—Diamine Fast Red F, Diamine Searlet B, or Benzopurpurin 4 B.

(1.)	(2.)	(3.)	(4.)	(5.)	(6.)
Dyeing Wool and Cotton alike.	Dyeing Wool more than Cotton.	Dyeing Cotton more than Wool.	Only Dyeing Wool.	Only Dyeing Cotton.	Dyeing Wool and Cotton together in Shades.
Oxyphenine (Cl).	Diamine Red NO (C).	Congo Brown G (Bl).	Hessian Purple NB (L).	Diamine Sky Blue (C).	Diamine Black BH (C).
Chloramine Yellow (Ba).	Congo Red R (G).	Direct Yellow G (K).	Cotton Brown A (C).	Diamine Blue 1 B (C).	Diamine Blue 2 B (C).
Thiodiam S (C).	Chicago Blue G (Bl).	Direct Yellow 2 G (K).	Cotton Brown X (C).	Zinnser Blue R (C).	Diamine Black BO (C).
Alkali Yellow R (D).	Chicago Blue R (Bl).	Direct Yellow 3 G (K).		Zinnser Blue B (C).	Diamine Black BO (C).
Chromine (K).	Brilliant Congo R (Bl).	Direct Orange R (K).		Benzen Yellow G (Ba).	Diamine Blue 3 B (C).
Mimosa (G).	Brilliant Purpurin R (Bl).	Direct Orange 2 R (K).		Congo Fast Blue R (C).	Diamine Blue BX (C).
Turmerine (B).	Erika (Bl).	Clayton Yellow (Cl).			Diamine Blue 3 R (C).
Primuline.	Diamine Scarlet B (C).	Cotton Yellow (K).			Benzen 3 G (Ba).
Congo Corinth B (Bl).	Benzopurpurin 4 B (Ba).				Vio Blue (Ba).
Titan Red (RH).	Benzopurpurin 10 B (Ba).				Brilliant Azurine 5 G (Ba).
	Congo Corinth (Bl).				Diamine Violet N (C).
	Deltapurpurin 5 B (Ba).				Diamine Bronze G (C).
	Diamine Gold (C).				Congo Brown A 2 B (Bl).
	Chrysophenin (L).				Diamine Brown V (C).
	Chrysamine G (Ba).				
	Acridine Orange W (L).				
	Diamine Green B (C).				

B = Badische Anilin- und Sodafabrik. Ba = Farbenfabriken (Elberfeld). Bl = Actiengesellschaft zur Anilinfabrikation (Berlin). C = L. Cassella and Co. Cl = Clayton Aniline Co. D = Dahl and Co. G = Rudolph Goetz. K = Kalle and Co. L = Leonhardt. RH = Read Holliday.

**Red-Violet.**—Diamine Violet N with Sulphoneyanine.

**Navy Blue.**—Diamine Black with Brilliant Sulphonazurine, or Chicago Blue G with Diamine Black BH.

**Light Blue.**—Diamine Blue with Alkali Blue 6 B. This shade is not fast to light.

**Brown.**—Diamine Bronze G with Brilliant Sulphonazurine and Congo Brown G.

In dyeing a second lot of goods in an old bath the amount of sodium sulphate used must be greatly diminished. To arrive at good results the bath should be tested with the hydrometer. The correct strength of the liquor is  $2\frac{1}{2}$ —3° B.

The effect of large quantities of sodium sulphate is to swell out the wool, at the same time giving it a flabby feel. The increase of volume may be preserved and the flabbiness avoided by an addition to the bath of  $1\frac{1}{2}$ —2 grms. of starch or gum.—R. B. B.

*The Production of many-coloured Fabrics by Mordanting the Yarn before Weaving.* H. Lange. *Färb. Zeit.* 5, 49—52 and 67—71.

THE production of parti-coloured fabrics, consisting of two or three different fibres, by choosing suitable colouring matters and methods, presents little difficulty. For example, with a mixed wool and cotton fabric the wool is dyed with an acid colour, the goods treated with tannin and an antimony salt, and then with a basic colour, the cotton becoming dyed and the wool shade being modified. With the benzidine colours good results may be obtained if it be remembered that in an acid bath wool and silk are dyed more than cotton, while in a neutral (salt) bath the cotton acquires the deeper shade, this being the case to a still more marked extent in an alkaline bath.

Fabrics of wool and silk are dyed in two colours by dyeing the wool with certain acid colours (e.g., Orange II) in a boiling bath, removing as far as possible the faint colour from the silk with boiling distilled water, or by other means, and then dyeing the silk in a warm bath with another acid colour (e.g., Water blue).

The special processes indicated in the title of the paper are applicable to fabrics of wool, cotton, or silk, or to mixed fabrics containing two or all of these fibres.

With fabrics of one fibre only, one method is to weave mordanted with unmordanted yarn, and then dye with some colour requiring a mordant. Then pass through a weak soap bath to remove colour from the unmordanted portions, and if necessary top with some basic, acid, or benzidine dyestuff. If three or more shades are desired, then unmordanted yarn is woven with yarn treated with salts of chromium, aluminium, iron, &c., and the fabric dyed with alizarin, logwood, &c.

Similar methods may be used for mixed fabrics, e.g., unmordanted and mordanted cotton, unmordanted and mordanted wool are all woven together, and dyed in one bath, cleared with soap, and perhaps topped with a second dyestuff, which may dye the wool only, the cotton only, or both fibres.

Numerous examples are given, illustrating these processes as applied to all classes of fabrics. This method has for some time been employed in the production of black fabrics with undyed margins, the main part of the fabric being woven from wool and mordanted cotton, while the edges consist of unmordanted cotton.—R. B. B.

*The Electrolytic Reduction of Indigo.* J. Mullerius. *Chem. Zeit.* 17, 1454.

THE possibility of reducing indigo by electrolysis was first demonstrated by Goppelsroeder. An electric indigo vat would possess the very great practical advantage of not containing any sediment which from time to time has to be removed, thus causing an interruption in the working of the vat. Having prepared a vat by electrolytic reduction of indigo, it would be necessary to pass through this vat while it is being used a feeble electric current. But Goppelsroeder, as also Wartha, observed that prolonged electrolytic treatment causes a further change of the indigo white, or even its total destruction, and for this reason the practical use of this vat is impossible. The electrolytic reduction of indigo cannot be satisfactorily effected in the

cold, but is easily accomplished at the boiling temperature. On the other hand, solutions of indigo carmine are easily reduced by the electric current even in the cold.—C. O. W.

*The Reduction of Indigotine to Indigo White.* F. Goppelsröder. Chem. Zeit. 17, 1633.

The reduction, or rather the hydrogenation of indigotine, for the purpose of forming a vat, can be effected in the presence of an alkali by various substances, some of which act in the cold, while others require higher temperatures. In the cold vats, which are especially used for the dyeing of cotton and linen, ferrous sulphate, zinc dust, glucose, hydro-sulphite, stannous chloride, and arsenic, or stannous chloride and orpiment, are used as reducing agents; while of the warm vats, which are used for the dyeing of wool and silk, the wool vat, the soda or potash vat, and the urine vat are those generally employed. Mixtures of indigo and various alkalis were subjected to the action of electric currents of varying power, but the reduction of the indigo was in every case found to be exceedingly slow and imperfect in the cold. At a boiling temperature, however, it proceeds very rapidly, but the prolonged action of the electric current upon the vats obtained, leads to further changes of the indigo white, and finally altogether destroys it. By impregnating cloth with a suspension of finely-ground indigo in a solution of caustic soda or lime and passing the cloth in contact between two copper plates forming the electrodes, and then sending an electric current through these plates, reduction of the indigo on the cloth takes place, and on subsequent exposure of the cloth to the air, it becomes dyed with indigo.—C. O. W.

*The Synthesis of Azo Colours on Animal Fibres.* J. Pokorny. With Report on the same by P. Werner. Bull. Soc. Ind. Mulhouse, 1893, 282—287.

Wool and silk possess the property of attracting from an aqueous bath certain amines, e.g., naphthylamine, provided that the amines are present as bases, and in a finely-divided condition. Naphthylamine is dissolved in alcohol, the solution poured into the bath, in which the wool is steeped over night. The next day the wool is passed through a solution of sodium nitrite acidified with hydrochloric acid; after some minutes the colour is developed, when the wool is taken out, washed, and dried. The colours are fast to soaping. The quantity of amine determines the depth of the shade. In the nitrous acid bath part of the naphthylamine is first diazotised, and the diazo compound combines with the remainder of the naphthylamine to produce the corresponding amido-azo body. The colours are only formed on wool or silk. Stannous chloride decolorises the fibre.

All amines which have the para-position free, and which are capable of yielding amido-azo compounds behave in a similar way.  $\alpha$ -Naphthylamine yields brown,  $\beta$ -naphthylamine, orange-red colours. For printing, the amine, together with thickening, is printed on the fabric, which is then passed through the nitrous acid bath to develop the colour.

The fixation of the amine on the fibre in the first bath is entirely mechanical, and it can be completely removed by washing with hot water.

Wool and silk prepared with naphthylamine and exposed to sunlight become darker. Brown or orange designs on a lighter ground can be obtained by preparing with naphthylamine, exposing portions of the fabric to sunlight, and developing with nitrous acid, when the exposed portions take a much darker colour than the covered portions; this is especially the case when  $\alpha$ -naphthylamine is used.

(It may also be noted that if wool or silk are steeped in a solution of a diazo or tetra-azo body, e.g., compounds derived from naphthylamine or di-anisidine, the fibre absorbs the azo-body, which can be converted into a useful colouring matter by further combining it with a phenol or amine.)

*Report by P. Werner on the above Paper.*—In carrying out the process described in detail in the foregoing, it is necessary to use alcohol and water in proportions depending on the nature of the amine. Thus, with  $\alpha$ -naphthylamine

(5 grms. per litre of liquid), as the amount of alcohol increases from 50 to 300 grms. per litre, the quantity of naphthylamine absorbed by the wool also increases. With larger amounts of alcohol it again decreases, until, with a bath consisting entirely of alcohol, no naphthylamine is absorbed. It appears, from experiments made, that the wool absorbs not only the dissolved naphthylamine, but also a part of the precipitated base, not by direct contact of the fibre with the precipitate, but through the medium of the liquid. The fact is, probably, that the naphthylamine passes from a medium in which it is less soluble, viz., water, to one in which it is more soluble, viz., wool. This absorption must take place in a bath in which the naphthylamine is to some extent soluble. Only the colours resulting from the use of  $\alpha$ -naphthylamine are of practical interest.

—R. B. B.

*The Action of Light on Metatungstate of Soda and its Applications.* C. Schoen. Bull. Soc. Ind. Mulhouse, 1893, 277—279.

If cotton be worked in a solution of metatungstate of soda of 5 per cent. strength, it rapidly becomes blue when exposed to light, owing to the reduction of the compound. In a dark room the fabric again becomes white, the blue colour re-appearing on renewed exposure to light. On treating the blue fabric with water a blue solution is obtained, which, on the addition of lead acetate, gives a blue precipitate. Both the solution and precipitate rapidly became colourless, even in full daylight. A simple solution of the metatungstate does not change colour. The effect of adding a salt of copper to the metatungstate is to retard though not to prevent the action of light; fabrics prepared with metatungstate and a copper salt become at first yellower and then slowly bluer. The reducing action of light is partly overcome by the oxidising properties of the copper salts.

—R. B. B.

*An Aluminium-Bronze "Doctor" for Printing-Machines.* (Reports on a "Doctor" sent in Competition for a Prize of the Society.) Bull. Soc. Ind. Mulhouse, 1893, 270—272.

(1.) *Report of M. Albert Frey.*—The new doctor has been in use for six months with satisfactory results. It presents a great advantage over the steel doctor with colours containing sodium bisulphite, which substance readily attacks steel, e.g., Coerulein S, Alizarin Blue S, &c. For reds and pinks also, the results are better than with doctors of either steel or ordinary bronze.

(2.) *Report of M. Albert Scheurer.*—The new "doctor" is superior to any previously made of the same material and to the "composition" doctor, but lacks the power of giving such clear and finished printing as is obtained by the use of the steel doctor. It does not, therefore, entirely fulfil the conditions of the prize, viz., that the doctor should offer the resistance of steel to mechanical work, and the resistance of "composition" to chemical agencies.—R. B. B.

*The Dyeing of Light Shades on Wool by Means of Alkalin Solutions of Alizarin Cyanine.* E. Schnabel. Farb. Zeit. 5, 145—147.

Like other alizarin dyestuffs, alizarin cyanines are soluble in caustic alkalis, alkaline carbonates and silicates, and borax. If wool is steeped in a hot alkaline solution of alizarin cyanine it is dyed, according to the shade of dyestuff, reddish-grey to light violet-blue. These colours are very fast to washing and fairly fast to milling, and approach in brilliancy the shades obtained with aniline colours, e.g., methyl violet. In choosing the alkali, sodium hydrate and carbonate are excluded owing to their effect on wool, and, in practice, ammonia is found most suitable. The bath must not be brought to the boil and the time of dyeing must not exceed 30 minutes, as hot ammonia solutions tend to mat and felt the wool. The method of dyeing, which is only suitable for loose wool, is as follows:—

The bath is heated to 85° C., when the alizarin cyanine, dissolved in 3–4 times its volume of liquor ammoniac, is added, and in addition 3 litres ammonia for every 50 kilos. wool. Enter the wool, and work 20 minutes at the same temperature. Take out, allow to cool, and rinse. The ammonia left in the wool quickly evaporates. After rinsing and drying, the wool, which becomes slightly matted during dyeing, recovers its original properties unimpaired. The dye-bath may be utilised for a second lot if certain proportions of alum and bran are added and the scum taken from the surface, whereby soap, &c. dissolved off from the first lot of wool are removed.

The disadvantages of the process are that, owing to the short time of dyeing, it is impossible by additions of dye-stuff to match a given shade, and that the colours are, as might be expected, sensitive to acids, the effect of which is to redden the shade. The former disadvantage is obviated by the fact that the dyer is soon in a position so to arrange the concentration of the bath that the desired tone may result, as the same mark of dye-stuff with the same amount of alkali gives always similar results.

The method is specially useful for pearl greys and light indigo blues. The colours withstand the action of sunlight better than the shades obtained with methyl violet or with indigo extract and orchil.

It is as yet difficult to say whether these colours should be classed as substantive or adjective, *i.e.*, whether the ammonia merely acts as a solvent, or whether it plays the part of a true metallic oxide.—R. B. B.

#### *Some Notes on the Dyeing of Logwood on Cotton.*

W. Bungert. *Färb. Zeit.* 5, 129–130.

When cotton is dyed by means of logwood extract and copper sulphate, the addition of tannin to the logwood tends to render the shade lighter. This appears to be due to the acid nature of the tannins, the same effect being noticed when acetic acid is added to the dye-bath. In the case of logwood extracts containing tannin this defect can partly be overcome by the addition of small quantities of soda to the bath, but the shade is not so dark even with this precaution, as when pure logwood extract is employed.

If, however, the logwood is applied by means of iron mordant, the addition of tannin darkens the shade. It is much better that the dyer should add the tannin extract to the logwood, than that he should use impure logwood extracts containing tannin. The best tannin material to use is chestnut extract, which tends to render the shade yellower, thus converting the logwood blue-black into a dead black, and rendering unnecessary the addition of a yellow colouring matter.

Although the deepest shades are obtained without alkali, in practice  $\frac{1}{2}$  per cent. of soda is added to the dye-bath; this causes the dyeing to take place more gradually, and therefore more evenly.

When a yellow dyewood is used to produce a dead black with logwood, either fustic or quercitron bark is chosen. The latter gives a darker shade with iron mordants, owing to the greater amount of tannin it contains, but for the best blacks a fresh logwood decoction is employed, together with pure fustic extract.—R. B. B.

#### *The Dyeing of Paper in Pulp.* H. Falke. *Färb. Zeit.* 5, 97–100 and 113–116.

A COLOURING matter to be suitable for paper-dyeing must possess a certain fastness to light, must not rub off, and should not injuriously affect the strength of the paper. The mineral colours have been largely used for this purpose, and the effect of the deposition of mineral particles in the fibre is that the paper becomes tendered. The coal-tar dyes are largely replacing the mineral colours, but the following of the latter class are still used:—

**Red Dyes.**—Vermilion, cinnabar.

**Yellow and Orange.**—Chrome yellow, ochre.

**Blue.**—Prussian blue, ultramarine, smalt.

**Green.**—Chrome yellow with Prussian blue.

**Black.**—Lampblack, graphite.

Of the vegetable colouring matters only logwood, catechu, and occasionally fustic are employed. The two former are used with potassium bichromate, alum, and nitrate of iron in the production of a black.

**Coal-tar Colours.** If the colour solution is merely mixed with the pulp, the greater part of the dye is carried away in the waste drained-off water. It is necessary to precipitate the dye-stuff in an insoluble form, *i.e.*, a mordant is required. Fortunately the substance used as sizing for the paper may act also as a mordant. The best substance of this class is the resin-soap made from colophony. This soap in solution is added to the pulp, and, after thoroughly grinding the mass, aluminium sulphate solution is poured in. Insoluble aluminium resin-soap is formed, and this serves as a mordant for both basic and acid dyes. With the former the colour precipitate consists of resinate of colour base and resinate of alumina, and with acid dyes of a compound in which aluminium is combined both with the colour acid and the resin acid. It follows that the amount of resins up must bear a definite ratio to the amount of dye-stuff, *e.g.*, water-blue and ponceau require 3–4 times and crystal violet 2½ times their weight of resin, in the form of soap, for complete precipitation. For thorough penetration of the pulp by the colour, it is best to add the soap and dye-stuff first, followed by the aluminium sulphate.

The following dyestuffs are completely precipitated by aluminium resin-soap, and the waste water is almost colourless if correct proportions of mordant and dye are employed:—

Cotton-scarlet.	Mandarin.
Rosecelline.	Orange II.
Croceine orange.	Metanil yellow.
Azotavin.	Victoria blue.
Diphenylamine orange.	Induline.
Indazine.	Phosphine.
Nigrosin.	Bismarck brown.
Brilliant crocein M.	

When the basic colours are applied to a paper which is only slightly sized, tannin and sodium acetate are added to complete the precipitation.

Too quick drying and too hot calendering injuriously affect the shade, rendering it duller.

The direct cotton dyestuffs can be fixed on paper by simply mixing their solutions with the pulp. Owing to their comparatively high price they are as yet only used for specially fine qualities. Many of them are exceedingly fast to light on paper, though this quality is not always of importance.

The dyes chiefly used for paper, besides those in the above list, and not including direct cotton colours, are:—

Water blue.	Safranine.
Hochst scarlets.	Brilliant green.
Eosine.	Malachite green.
Rose bengal.	Erythrosin.
Magenta.	Phloxin.
Acid brown.	Methyl violet.

In dyeing paper to match a given shade a preliminary trial should be first made with small quantities of material and the correct proportions thus ascertained. In matching a sample with the pattern, care must be taken that both are in the wet or both in the dry condition.—R. B. B.

#### PATENTS.

*Improvements in Machines for Dyeing and Securing Yarn.*  
J. M. Collins, Bridgeton, Glasgow. Eng. Pat. 977.  
January 17, 1893.

THIS refers to certain improvements in the machine described in a previous patent granted to the same inventor (Eng. Pat. 14,507 of 1892; this Journal, 1813, 825). The yarn-carrying poles are made in sections, capable of being reversed or interchanged, the end of each section being so formed that it can interlock with the end of the adjoining section. The poles are revolved by means of gearing fitted on the machine; this gearing drives a socket spindle for

each pole, into which the end of the pole firmly locks. Special rods are also provided for keeping the yarn stretched, and these are fixed in such a manner that they do not turn with the yarn.—R. B. B.

*Improvements in Bleaching and Discoloration.* J. Clapham, Bradford, J. Picard, Paris, C. Villedien, Paris, and W. W. L. Lishman, Brautord. Eng. Pat. 1623, January 25, 1893.

This invention relates to bleaching cotton, linen, flax, wool, leather, wool, &c., and in addition to the use of alkaline boils, includes a preparation of the material with olein soap, &c., followed by a treatment with permanganates and bisulphites, or, in lieu of the latter, with peroxides or alkaline chlorites. It is claimed that the preparation with the fatty body protects the fibre from the destructive action of the oxidising agents subsequently applied to it.

—W. E. K.

*Improvements in Printing and Dyeing on Fabrics.* O. Osteretzer, Sagan, Germany. Eng. Pat. 2153, January 31, 1893.

This invention relates to the production of discharge effects by the use of direct-dyeing colours which are capable of diazotisation on the fibre. The ground shade is dyed with a colour capable of destruction by means of reducing agents (diamine black, diazine, &c.), whilst the discharge is printed with a colour incapable of being so destroyed (primuline). After the discharge is effected the colours are then diazotised on the fibre and combined with a phenol or amine. For example, a Turkey-red shade on dark blue is produced as follows:—The cloth is dyed with diamine blue black E, a discharge containing primuline, zinc dust, and bisulphite is printed upon the dyed cloth and then steamed. The goods are then treated with muriatic acid, diazotised with sodium nitrite and hydrochloric acid, and lastly passed through a bath of  $\beta$ -naphthol and caustic soda. A blue print on a black ground is obtained in a similar way by dyeing with diamine black and developing with phenylenediamine.

—W. E. K.

*Improvements in Machines for Washing, Bleaching, Mordanting, Dyeing Cotton, Wool, or other Fabrics in a Raw, Spun, or Manufactured State.* C. Owens, Lower Broughton, and J. Whiteley, Salford. Eng. Pat. 4437, March 1, 1893.

The fibre is fed on to a revolving, perforated, endless table, and as it passes along is subjected to the action of water or other liquid, by means of jets placed above the table. After traversing the length of the machine the fibre is automatically passed between rollers. If necessary, a second table is provided underneath the first, in order to receive the fibre as it comes from the rollers and convey it back to the feeding end of the apparatus for a second similar treatment.

—R. B. B.

*The Production on the Fibre by the Aid of p-Amidodiphenylamine of Dye-stuffs fast to Light and Washing.* S. Pitt, Sutton. From L. Cassella and Co., Frankfurt-on-the-Maine. Eng. Pat. 1612, March 2, 1893.

This invention relates to the production of fast dyes from p-amidodiphenylamine by conjugating with diazo compounds produced directly on the fibre: such combinations being capable of production only on the fibre and not in solution. For instance, to obtain a dark blue fast to light and washing, cotton is dyed with diamine blue-black E, diazotised on the fibre with sodium nitrite and hydrochloric acid, and then passed through a bath of p-amidodiphenylaminehydrochloride.—W. E. K.

*Improvements in Apparatus for Dyeing and Washing Yarn in the Hank.* J. Rhodes, J. Rhodes, jun., and A. B. Perkins, Bradford, Yorks. Eng. Pat. 5291, March 11, 1893.

The apparatus consists of a framework, fitting inside the dye-vat, and provided with a number of cross rollers or hank-carriers, which have one end free to allow of the hanks being placed thereon. The whole framework with the rollers can be moved from one vat to another. Special rollers are also provided for keeping the yarn in a stretched condition in the vat. A feature of the apparatus, fully described and illustrated in the specification, is an arrangement of worm wheels for driving the rotatory hank-carriers.—R. B. B.

*Method of Producing Light Prints or Designs on Dark Ground, or for Producing Coloured Prints by Means of the Printing Press.* C. Vogel, jun., Gertrude a. Harz, Germany. Eng. Pat. 5397, March 13, 1893.

The print or design is produced on paper with light-coloured or white printer's ink prepared with oil, fat, or resin; and the surface is subsequently covered with water-colour, by means of rollers or other devices. As water will not mix with oil, &c., the water-colour adheres only to those portions of the surface which have not previously received an impression.—R. B. B.

*Improvements in Resists or Discharges for use in Printing or Dyeing Cotton, Wool, and other Fabrics with Aniline Black.* W. T. Whitehead, Magog, Canada. Eng. Pat. 7350, April 11, 1893.

This invention consists in the use of metallic zinc or any zinc compound (and by preference oxide of zinc) as a resist for producing white patterns on aniline black grounds. For producing coloured patterns an addition is made of a pigment and albumin, or of a coal-tar or extract colour, to the white resist (this Journal, 1893, 1627).—W. E. K.

*Improvements in Resists or Discharges for use in Printing or Dyeing Cotton, Wool, and other Fabrics.* W. T. Whitehead, Magog, Canada. Eng. Pat. 7351, April 11, 1893.

This invention consists in producing patterns in which the figure is of a shade contrasting with the ground, by printing the pattern with a resist containing zinc or a zinc compound and then dyeing the cloth with a plain colour in any well-known manner. A coal-tar, extract, or pigment colour may be added to the resist in order to vary the contrasting colour effect.—W. E. K.

*Improvements in the Production of an Indigo Vat or Bath employed in the Dyeing with Indigo.* Read, Holliday, and Sons, and H. Bindeschadler, Huddersfield. Eng. Pat. 7487, April 12, 1893.

This refers to an improvement in the hydrosulphite vat. At present sodium hydrosulphite is formed by the action of zinc on bisulphite of soda, and added to the ground indigo together with an alkali, usually lime, in sufficient quantity to dissolve the indigo white. According to this patent, instead of lime, caustic soda or potash is added in sufficient quantity to dissolve the indigo white, and also to redissolve the zinc hydrate precipitated by the first addition of alkali; thus producing a clearer vat with beneficial results.—R. B. B.

*Improvements in Dyeing Apparatus.* E. Gessler, Metzingen, Germany. Eng. Pat. 7841, April 18, 1893.

The material is wound on to a perforated spool. Into the upper part of each spool fits a regulating pipe, also perforated, and to the top of this are attached metallic rods forming a cross, from the four ends of whose arms depend hooks. These hooks are fastened into the material on the spools during dyeing. Each spool fits underneath on to a



support, which in its turn fits into a conical orifice in the false bottom of the dye-bath, and the spools thereby are firmly fixed to this bottom.

The dye-bath consists of a vessel with a false bottom, and is provided with a lid. A second large vessel or receiver, and two vacuum vessels, also form part of the apparatus, the dye-bath being connected with the receiver and vacuum vessels by suitably arranged pipes and taps. By means of one vacuum vessel the dye-bath is emptied of air, when the dye liquid flows into it from the receiver in such a manner that the liquid passes through the material from the outside, and is drawn off from the inside of the hollow spools into the second vacuum vessel. Thence it flows back into the dye-bath, passing through the fibre in the reverse direction. When these operations have been repeated sufficiently often, the spools are removed and the material dried in a centrifugal machine.

The regulating pipe ensures that all the liquid shall pass through the material. This is effected by the regulator automatically altering its length when the material is compressed on the spool, so that none of the perforations in the latter are left uncovered.—R. B. B.

*Improvements in Dyeing certain Colours on Cotton Fabrics and Yarns.* A. B. Hay and J. M. Park, Glasgow. Eng. Pat. 7919, April 19, 1893.

GREY shades are produced by fixing on the fibre a mixture of manganese and chromium oxides, brown and olive shades, by a mixture of manganese and iron oxides. For greys the cotton is impregnated with a solution containing chrome alum and manganese acetate, for olives and browns with acetates of manganese and iron. After treatment with the metallic solutions, the fibre is partially dried and passed through a weak solution of an alkaline hypochlorite at 150° F., the oxides being thereby precipitated. The colours are described as "fast and permanent."—R. B. B.

*A Process for Colouring Paper and Cardboard.* F. R. Wells, Southgate, Middlesex. Eng. Pat. 8167, April 22, 1893.

THE colouring matter is mixed with gelatin, starch, dextrin, or some other sizing material which is affected by chromium compounds, and a solution of a chromium salt or chromate. This mixture is either applied to the surface of the paper or incorporated with the pulp during manufacture. Or the colouring matter and size are first applied to the paper, which is then treated with the chromium solution.

In every case the paper or cardboard must be exposed to light or kept some time before use.—R. B. B.

*Improvements in or Appertaining to the Utilisation of a Waste or Nearly Waste Product, and the Manufacture of a Useful Gum or Gummy Compound therefrom.* J. Hanson, P. C. D. Castle, and J. H. Morrison, Liverpool. Eng. Pat. 8793, May 2, 1893.

See under XVI., page 410.

*Improvements in Yarn-dyeing Machines.* J. G. Haslam, Philadelphia, U.S.A. Eng. Pat. 18,517, October 3, 1893.

THIS invention embodies several improvements in the apparatus described in Wolstenholme's patent (Eng. Pat. 2060 of 1889; this Journal, 1889, 329). The improvements are as follows:—(1) The necessity of disconnecting the yarn frame from the lifting devices, in order to reciprocate the former in the dye-liquor, is avoided; (2) an arrangement whereby the sets of yarn sticks may be moved nearer together or further apart to suit the size of the skeins; (3) the adjustment of the yarn sticks is accomplished through the supports for the upper set of sticks, instead of the lower, so that adjustment may be brought about while the frame is lowered, and the yarn submerged in the liquor; (4) the employment of indicating devices which register the number of reciprocations imparted to the yarn frame.

In addition, certain minor improvements are described, which, together with those mentioned, are minutely set forth in the specification, and illustrated in the drawings accompanying the same.—R. B. B.

*Apparatus for Dyeing and Washing Loose and Spun Wool, Cotton, and such like Material.* E. Labhardt, Basel, Switzerland. Eng. Pat. 20,257, October 27, 1893.

Into a boiler containing the washing or dyeing liquor, a perforated basket fits exactly at the sides, but so as to leave a space below it in the boiler. The basket rests on the rim of the boiler by means of rollers allowing it to be rotated as required, and is divided into two or more compartments by perforated horizontal partitions. The liquor is brought to the boiling point and kept constantly in whirling motion by means of suitably arranged steam injectors. The action of the steam causes the liquor to pass downwards through the fibre in the basket to the lower part of the boiler, from whence it passes upwards by pipes, and thus again into the upper compartment of the basket. An injector is also arranged so that steam can be passed upwards into the basket, when necessary, in order to loosen the material contained therein.—R. B. B.

*An Improved Process of Dyeing for Producing Genuine Turkey Red and Rose Colour in Vegetable Yarns in Cops, Hanks, Carded Strips, and the like.* W. P. Thompson, Liverpool, Manchester, and London. From E. Holken and Co., Barmen, Germany. Eng. Pat. 23,559, December 7, 1893.

THE cops or hanks of yarn are oiled and mordanted with alumina in the usual manner, and the mordant fixed by means of borax. The novelty of the process consists in the alizarin dye-bath being cold and extremely concentrated, excess of alizarin being removed from the fibre after dyeing by treatment with suitable solutions (e.g. acidulated water). The depth of colour is regulated by the strength and composition of the alumina mordant. After dyeing, the yarn is oiled, and steamed under pressure, and finally cleared in a hot soap bath.—R. B. B.

*A Machine for Dyeing Hats, Caps, Stockings, and other similar Articles.* E. F. Liebt, Elberfeld, Germany. Eng. Pat. 520, January 9, 1894.

UP to the present the dyeing of these goods has been very difficult compared with yarn and piece dyeing, &c., the slightest inattention to minute details having disastrous consequences, and the expense being great.

The machine consists of two vessels, which can be used for mordanting and dyeing, or dyeing and washing. A cylinder of perforated wood or metal contains the articles to be dyed, and rotates within one vessel. The cylinder, which is constructed after the type of the paddle-wheel, is furnished internally with a number of "spokes" partly dividing it into compartments, and the rotation of the cylinder causes the articles to be continually thrown into those compartments coming in contact with the dye liquor.

By special mechanism the cylinder with the goods may be lifted out of the one vessel and placed within the other to undergo some further necessary process.—R. B. B.

## VII—ACIDS, ALKALIS, AND SALTS.

*Employment of Electrical Heat for Concentrating Sulphuric Acid.* C. Haussermann and L. Nothhammer. Chem. Zeit. 17, 1907—1908.

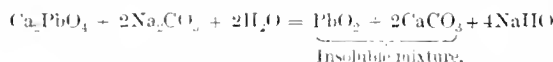
EXPERIMENTS on the concentration of sulphuric acid by means of electrically generated heat, as suggested by Bucherer (this Journal, 1894, 249), yielded very unsatisfactory results with continuous direct currents, owing to

electrolysis and consequent loss. Sulphur soon made its appearance, and sulphuretted hydrogen and other gases were evolved, the current partly passing through the acid by the shorter path. After  $2\frac{1}{2}$  hours, 42 grms. weight was lost by the acid; but the strength had only risen to 61 B., the loss being mainly due to decomposition, and not to evaporation. In another experiment under conditions again modified, considerable separation of sulphur and evolution of gases occurred, the acid itself acting as a second path to a large extent, and electrolysis consequently taking place: thus after 6 hours 190 grms. of 60° acid were reduced to 157 grms., but the concentration only rose to 62° B. It was ultimately found impracticable to diminish the resistance of the wire to such an extent as to render the branch current passing through the acid negligible, because in that case the heat developed (proportionate to resistance and square of current) would be insufficient for the evaporation; and with larger current strengths, the branch current, and consequently the electrolysis, would be also increased.

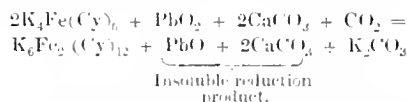
Somewhat better results, however, were obtained when alternating currents were employed; thus, under this condition, 190 grms. of 60° acid were concentrated to 65.8° B. (at 15°) with a loss of weight of 35 grms. in  $3\frac{1}{4}$  hours, the temperature rising to 286° and remaining nearly constant thereafter; the current employed was derived from a lighting supply of 110 volts and about 0.9 ampère, converted by suitable transformers into an alternating current of 6 to 7 volts and 14 ampères. No visible sulphur was formed, but a slight disengagement of gas occurred along the length of the wire. The energy consumed during the production of 155 grms. of concentrated acid accordingly amounted to  $14 \times 6.5 \times \frac{13}{4} = 296$  Watt-hours, or about 1,490 per kilo.—C. R. A. W.

*The Preparation of Ferrieyanide Salts. Preliminary Communication.* G. Kassner. Chem. Zeit. 1893, **17**, 1712.

IN 1889 the author proposed a method for the preparation of potassium ferrieyanide (this Journal, 1890, 391), based on the reaction between lead dioxide and potassium ferrieyanide in presence of carbonic acid gas, recommended by Lunge, but instead of lead dioxide the author used calcium plumbate with advantage for the conversion of ferro- into ferrieyanide salts. This compound is easily decomposed with separation of lead dioxide in the nascent state, by acids, ammonium salts, and carbonates and bicarbonates of the alkalis; in the latter case, the calcium carbonate also being made use of. By sodium carbonate at about 130° C., or under pressure, the decomposition is represented as follows:—

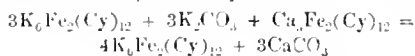


This insoluble mixture, which contains the lead dioxide in a finely-divided state, and is freed from sodium hydrate, is now caused to react with potassium ferrieyanide in presence of carbonic acid gas (on the large scale obtained from lime-kilns or coke-ovens) in the cold, according to the equation—



On evaporation of the clear liquor drawn off from the heavy insoluble residue which has settled down, the potassium ferrieyanide is separated from the potassium carbonate by crystallisation, the latter remaining in the mother-liquors.

The washed residue, consisting of lead monoxide and calcium carbonate, is employed for the regeneration of calcium plumbate. By the addition of calcium ferrieyanide, the potassium carbonate present is converted into potassium ferrieyanide, according to the equation—

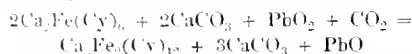


The separation takes place easily on warming, the calcium carbonate quickly precipitating and settling down, whilst the potassium ferrieyanide is obtained on evaporation in good crystals.

It is indifferent whether the calcium ferrieyanide be added before or after oxidation, but the author prefers the latter for the following reasons:—

For completely carrying on the oxidation of the ferrieyanide, carbonic acid gas is necessary, by means of which the excess of alkali from the ferrieyanide salt, is converted into carbonate, and the dioxide of lead, after giving up its oxygen, into basic lead carbonate; but since carbonic acid is only slightly soluble in the liquid, the alkali acts as a carrier of this gas, in becoming converted intermediately into bicarbonate or sesquicarbonate.

The preparation of calcium ferrieyanide is analogous to that of the potassium salt by calcium plumbate; the latter is converted into a finely divided mixture of lead dioxide and calcium carbonate, and then treated with calcium ferrieyanide and carbonic acid gas, preferably under pressure. The reaction proceeds in the cold or warm, and in concentrated solution, as follows:—



Two molecules of hydrochloric acid are now added to the precipitated residue, after running off the supernatant liquor, to remove the excess represented by one molecule of calcium carbonate; the residue can then be worked up again for calcium plumbate. The calcium ferrieyanide solution is preferably used at once for treatment of the crude potassium ferrieyanide solution as above, or if required in the solid state, it must be evaporated in partial *vacuo*, since it is somewhat unstable.

The removal of alkaline carbonate is now only necessary to obtain a pure crystallised potassium ferrieyanide. For use as an oxidising and bleaching agent, the removal of the carbonate is not only unnecessary, but its presence is actually of advantage, since by the addition of calcium plumbate, the requisite quantity of caustic alkali is thus obtained (this Journal, 1890, 108).

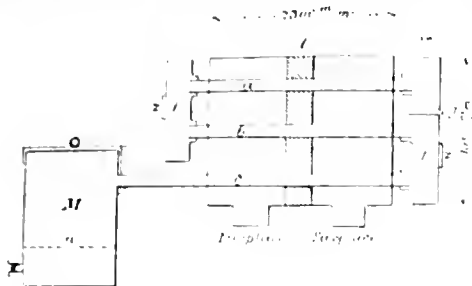
By regular regeneration of materials, the process becomes continuous.—W. B. H.

*Manufacture of Barium Salts.* Chem. Zeit. **17**, 1847—1848; **18**, 67—68.

AMONGST the various barium compounds employed in industrial operations, three more particularly are in growing demand, viz., barium chloride, sulphide, and peroxide.

*Barium Chloride* is usually prepared either by acting on *witherite* (natural barium carbonate) with hydrochloric acid, or by heating together barium sulphate, small coal, and calcium chloride, when barium chloride is formed in consequence of the double decomposition ensuing between the calcium chloride and the barium sulphide first produced by the reduction of the sulphate. This latter method is carried out on the large scale thus:—240—250 kilos. of heavy spar, ground fine (containing 93—95 per cent. of actual  $\text{BaSO}_4$ ), are intimately mixed with 160—170 kilos. of calcium chloride (containing 70—75 per cent. of  $\text{CaCl}_2$ ) and 85—90 kilos. of coal dust, and charged into the hinder part of a roaster, where it is heated up for two hours, and is then raked into the front part, where it is further heated with frequent stirring, a fresh charge being introduced into the back part of the oven, so that two charges are in simultaneously, one heating up and the other being worked. In this way five charges per shift are worked off. Instead of fused calcium chloride, the mother-liquors from potassium chlorate manufacture are sometimes used. These are usually of density about 40° B., and contain a known proportion of calcium chloride. A measured quantity is intermixed with the appropriate amount of heavy spar and coal dust to a thick pulp, which is then dried in the oven; in consequence of the extra time requisite for evaporating off moisture, only four charges per shift are worked off instead of five; only one charge, moreover, can be in the oven at once, as the stuff spreads over the whole of the sole. When the operation is finished the fluxed mass is raked

out into conical iron moulds and cooled; the product is almost black, with a grey fracture; portions of unburnt carbonaceous matter should be present, otherwise the reaction is probably incomplete; on the other hand, flakes



of undecomposed heavy spar should be absent. The structure should be somewhat porous, otherwise the subsequent lixiviation will be difficult; with a successful melt 59–60 per cent. of  $\text{BaCl}_2$  should be dissolved out, with only 0.05–0.1 per cent. of S, the rest of the mass being mostly insoluble calcium sulphide; too large a proportion of calcium chloride must be avoided, otherwise the liquors become too soon highly charged with the excess of this salt. The lixiviation is carried out in iron tanks of  $3 \times 3 \times 1.5$  metres, five or six being connected together in a system; the melt, broken into blocks the size of a head, is filled into one of these, hot water run in, and the whole heated up by steam; the strong ley thus produced usually marks  $30^\circ$ – $31^\circ$  B., and is run off, an equal volume of hot water being simultaneously run in, so as to keep the level of the liquor constant; when the ley is thus reduced to  $24^\circ$ – $25^\circ$  B. the running off is interrupted. The mud is poked over with an iron rod to feel if any lumps of solid matter are present, in which case much barium chloride might be lost through defective solution; if the mud is free from lumps, the liquor, at about  $24^\circ$  B., in the tank is run off to a different reservoir, and the tank filled with water and boiled up. The final wash waters should not mark more than  $1^\circ$  B., containing only 0.4–0.5 per cent. of barium chloride; these, together with the previous weaker liquors ( $24^\circ$  B. downwards), are used instead of fresh water to lixiviate a new batch. The stronger liquors first run off ( $31^\circ$ – $24^\circ$  B.), are pumped into a tank in which they are treated with carbonic acid (lime-kiln gases) and steam, to decompose the small quantity of barium sulphide present, the blowing being continued until a sample of the liquor no longer gives a brown precipitate with basic lead acetate solution; this, as a rule, requires about two hours; after some hours standing at rest to settle, the clear hot liquor is treated with a little hydrochloric acid to neutralise it, and evaporated in iron pans until a pellicle forms on the surface (i.e., till the density is about 30 B.); it is then run into iron crystallising pans. To avoid discolouration of crystals by iron, the evaporating and crystallising pans are coated with enamel or other protective material. The mother-liquors are worked up along with a fresh batch of liquor, and so on until the quantity of calcium chloride present rises to about 200 grms. per litre; when this is reached the mother-liquors are further concentrated alone, so as to obtain a crop of impure barium chloride crystals, the final mother-liquors being worked up along with solid chloride of calcium, &c. in the charge for the roasting oven. If anhydrous powdered barium chloride is required, the first strong liquors are boiled down in a directly-fired pan so that fine crystals of barium chloride separate; these are fished out and dried in a calcining oven with continual stirring with iron rakes, and finally packed in wooden barrels. The product contains about 95 per cent.  $\text{BaCl}_2$ , together with a little calcium chloride and water.

**Barium Sulphide** is prepared in the same kind of roasters as are in use for the chloride, with the difference, as regards materials, that calcium chloride is omitted and some binding material (such as coal tar) used instead. After some  $2\frac{1}{2}$  hours' firing the melt is raked out into well-covered iron moulds to cool, and is then packed in barrels, &c. as

rapidly as possible. By exposure to air the lumps become covered with a white layer of  $\text{Ba(OH)}_2$  and  $\text{Ba(HS)}_2$ , owing to absorption of moisture; material thus altered is worked over again with a fresh charge. The analytical valuation is effected by treating a known weight with water and hydrochloric acid, filtering and washing into a graduated vessel, precipitating a known fraction of the fluid as sulphate, and reckoning the sulphate formed into  $\text{BaS}$ .

**Barium Nitrate** is manufactured from the chloride by dissolving in a copper vessel to a saturated solution of about 32 B., and thus adding the appropriate quantity of sodium nitrate; the difficultly soluble nitrate precipitates and is collected, drained, slightly washed with cold water, and recrystallised by dissolving up in lead-lined wooden tanks of  $1 \times 2 \times 0.8$  metre to a density of 24 B.; after settling for two or three hours the hot liquor is drawn off through a lead or rubber siphon into lead lined crystallising tanks of  $2.5 \times 1 \times 0.8$  metre; the crystallisation takes two to four days, according to the time of year. The mother-liquors serve to wash the first crystals of nitrate formed from the chloride; the first mother-liquors left in the copper pans contain little but sodium chloride, and are a waste product utilised as occasion serves, e.g., for preparing sal ammoniac; the second mother-liquors, after washing the crude crystals, are evaporated so as to obtain a crop of impure nitrate crystals; this is repeated, after which the amount of barium nitrate left is too little to be worth recovering; thus the second time affords a crystallisation containing only 8.2 per cent.  $\text{Ba(NO}_3)_2$ ; the third time, one containing only 2.2 per cent.

**Barium Peroxide** is manufactured from the nitrate obtained as above, by ignition in pots holding about 16 kilos. each, a series of 24 being heated simultaneously; the salt fuses and then becomes decomposed, solid baryta being ultimately left. Next day, when the pots have cooled sufficiently, the cakes of baryta are removed and broken up into fragments, about 1 cm. square and 3–4 thick; these are filled into iron boats or gutters, about 1 metre long, two of which are placed inside a cast-iron tube capable of being heated to a dull red heat; the arrangement shown in the figure is usually employed, three cast-iron tubes, *a*, *b*, *c*, being arranged parallel to one another in a double fireplace, their ends being united by the movable socketed cross-tubes *f* and *f'*, furnished with glass spy holes, *e* and *e'*, for judging the temperature of the tubes. *f'* communicates with the chimney at *x*, a damper being placed beyond *x* to regulate the draught, and another at *g*. The tube *c* is empty, only the other two, *a* and *b*, containing the boats, filled with baryta; these are introduced when the tubes are heated to a dull red heat and kept there for 3–4 hours, a stream of purified air being drawn through all the time; the purifier is indicated by *M*, and consists of an iron chest with a tightly fitting lid, *a*, filled up to the level *n* with lumps of caustic soda, so that the air passing through is deprived of moisture and carbonic acid. Finally, the barium peroxide resulting from the roasting of the baryta is picked over to separate imperfectly oxidised lumps, &c., and packed in strong paper-lined barrels of 200 kilos. each. Its chief use is for direct bleaching, and for the preparation of hydrogen peroxide, for which purpose sodium peroxide is also coming largely into use.—C. R. A. W.

*Electrolysis of Alkaline Chlorides.* F. Oetzel. Chem. Zeit. 18, 69–70.

See under XI., page 403.

*The Detection of Mineral Acids in Vinegar by means of Rosaniline Hydrochloride.* G. Griggi. Sci. 1893, 3, 176.

See under XXIII., page 423.

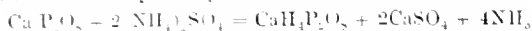
*The Detection of Mineral Acids in the Presence of Organic Acids by Colour Reactions.* E. Nickel. Chem. Zeit. 17, 1670.

See under XXIII., page 423.

## PATENTS.

*Improvement in the Manufacture of Soda and Monocalcic Phosphate.* F. Hurter, Widnes, Lancaster, and J. Omholt, Christiania, Norway. Eng. Pat. 7107, April 6, 1893.

THE residual ammonium sulphate solution obtained in the manufacture of soda from sodium sulphate by the ammonia-soda process, is evaporated to dryness, preferably in steel heat pans, and the dry salt is mixed with tricalcium phosphate (high grade native mineral phosphates being used in practice), the phosphate preferably in excess of the proportion indicated by the equation—



The mixture is heated to from 550° to 650° F. in a cast-iron horizontal retort, set in brickwork, and heated externally by direct fire, in a slow current of previously superheated steam. The ammonia evolved is led into cooling apparatus, in which a dilute solution of ammonium sulphate is collected, applicable in dissolving sodium sulphate to be treated by the ammonia-soda process. The dry ammonia is then conducted into absorbing apparatus to meet with fresh sodium sulphate solution. The contents of the retort are allowed to cool in an atmosphere of steam to about 220° F., a special inlet being provided for steam at atmospheric pressure only; the monocalcium phosphate then absorbs any water it may have lost at the higher temperature. The residue, consisting of monocalcium phosphate, calcium sulphate, and small proportions of ammonium sulphate, and unaltered tricalcium phosphate, is then withdrawn to be ground up as a manure, with addition of 10 or 15 per cent. of sulphuric acid of sp. gr. 1.60, if desired. If the mass becomes damp, it may be dried at a temperature not exceeding 212° F.—E. S.

*Improvements in the Manufacture or Production of Monocalcic Phosphate and Ammonia.* F. Hurter, Widnes, Lancaster, and J. Omholt, Christiania, Norway. Eng. Pat. 7108, April 6, 1893.

See under XV., page 409.

*Improvements in certain Apparatus for the Distillation of Ammoniacal Liquor.* F. M. Spence and D. D. Spence, Manchester, and J. Gilchrist, Lyndhurst, Manchester. Eng. Pat. 7485, April 12, 1893.

THE improvements relate to the construction of the vertical iron column containing overflow shelves projecting from alternately opposite sides used for the distillation of ammoniacal liquor by means of introduced steam. The column is made oblong in cross section, instead of square, in order to greatly increase the length of contra-current traverse between any two openings. The still is made, not only to distil off free ammonia; but for the reaction by milk of lime on ammonium salts, so that the ammonia from these also may be recovered in the same apparatus. As ammoniacal liquor usually contains both free and fixed ammonia, the shelves or diaphragms of the upper portion of the still are such in number as may suffice for the separation of the free ammonia, each shelf having a lip of sufficient height to maintain always a stratum of liquid upon it. But the first of the lower series of shelves has a much higher lip than any of those above or below, so that it may hold a considerable store of liquor, and into this receptacle a flow of milk of lime is introduced by a suitable pipe. The liquor received from the higher shelves mixes with excess of lime, and flows downwards from shelf to shelf, each lipped, like those of the upper series, having exit near the bottom by a suitable pipe. Steam is admitted, under pressure, near the bottom, and a dead-weight safety-valve is provided at the top of the still, which need not be constructed to resist more pressure than 10 lb. to the square inch. The top shelf is provided with a vertical plate near its closed end, to admit of retaining sufficient liquor to cover completely the perforated pipe, arranged transversely, into which the supply of ammoniacal liquor is led from a supply tank, having an equalising box with ball-valve. Opposite the lip of each

shelf is a manhole, closed by a lid held in position by cross bars, to admit of the free use of rakes or shovels for removing any pitchy or other matter that may accumulate.—E. S.

*Improvements in the Manufacture of Permanganates of Soda or Potash from Manganates of Soda or Potash.* J. Brock and F. Hurter, Widnes, Lancaster. Eng. Pat. 7512, April 12, 1893.

THE inventors have found that solid sodium or potassium manganate is difficult to convert into permanganate by carbonic acid gas; but that if a small proportion of water is added, as liquid or as steam, the conversion is greatly facilitated. The process is preferably conducted by spraying water, to the extent of about 20 per cent., on a layer of the ground manganate, which is meanwhile turned over or agitated. Should the powder cake, it is broken up or re-ground. The manganate is then charged into a revolving cylinder provided with breakers or stirrers, such as is described in Eng. Pat. 1213 of 1893 (this Journal, 1894, 38), and carbonic acid gas is passed in, and is rapidly absorbed by the hydrated manganate, with evolution of heat, the manganate being converted into solid permanganate, and any free alkali into carbonate or bicarbonate. The process may be modified by sending steam or a spray of water into the cylinder, instead of previously moistening the manganate; or the gas may be passed in moist, or with steam or spray.—E. S.

*Improvements in the Electrolytical Decomposition of Chlorides and Mixtures thereof, and in Apparatus for the Purpose.* F. M. Lyte, London. Eng. Pat. 7594, April 13, 1893.

See under XI., page 404.

*Improved Process for the Manufacture of Carbonate of Ammonia.* C. Raspe, Berlin. Eng. Pat. 7846, April 18, 1893.

LIQUORS containing ammonium carbonate, such as ammoniacal liquors from the distillation of fuel, bones, or the like, are freed from sulphur by agitation with suitable metallic hydroxides or with zinc carbonate. The liquor, to which sodium carbonate may be added, is then agitated with a "fatty oil" to remove a portion of the empyreumatic bodies, and is distilled in an apparatus having wide passages to obviate clogging. The ammonium carbonate vapours are passed through freshly-made, heated wood-charcoal to remove the remainder of the "empyreuma," and are condensed. "The drying of the carbonate of ammonia may also be assisted or performed by the use of drying materials, either with dry carbonates or with dry potash or soda."—E. S.

*Improvements in the Manufacture or Production of Soda Crystals.* H. R. Browne, Roselea, Lancaster, and M. Guthrie, Liverpool. Eng. Pat. 8907, May 3, 1893.

THE caustic soda liquor obtained by the electrolysis of brine, is agitated for some hours with the mixture of sodium carbonate and bicarbonate obtained in the ammonia-soda process, when the sodium bicarbonate produced in that process has been freed from ammonia and partially decarbonated by suitable heating. The proportions used of the caustic soda liquor and of the mixed carbonates are such as may result in the total formation of sodium carbonate, proportions determinable by any suitable tests. About 2 per cent. of salt cake and enough bleaching powder or liquor to oxidise any colouring matters that may be present, are added. The liquor should be of such density as to contain about 25 per cent. of sodium carbonate, and is cleared from insoluble matters by settling and decantation, and is then run into pans for crystallisation, the ordinary soda crystals ( $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ ) being obtained.—E. S.

*Improvements in and relating to the Treatment of Salt, and the Formation of Blocks thereof by Fusion.* W. Haddan, London. From La Compagnie du Sel Agglomeré, Paris. Eng. Pat. 24,194, December 15, 1893.

SALT that has become agglomerated by moisture, is crushed by apparatus consisting of unchangeable metal, mill-stone, or other materials that will not soil.

Impure or discoloured salt is purified in various ways, according to the circumstances, as by solution and decantation, washing in saturated water, or mechanical processes. If organic matter is present, the subsequent process of fusion, with or without addition of a nitrate, gives a colourless product. Drying is sometimes advisable before fusion, and may be carried out in various ways.

*Fusion or Melting.*—Small quantities of salt may be melted in movable crucibles in any system of furnaces. The crucibles are of unattackable metal, refractory earth, or porcelain. For melting salt in larger quantities, a furnace is used having crucibles fixed in it, single or multiple; or with a fixed pan; or with a movable pan. The second type of furnace, with fixed pan, is shown in the drawings below; one an elevational and sectional view; the other a plan view and section.

Fig. 1.

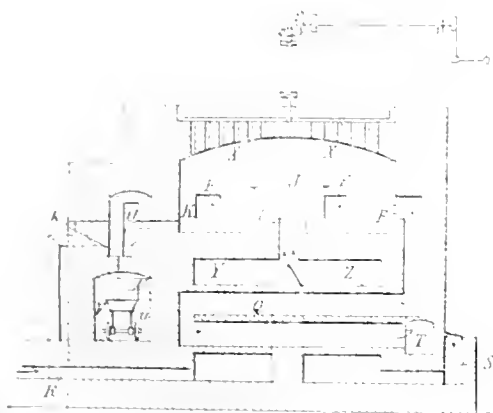
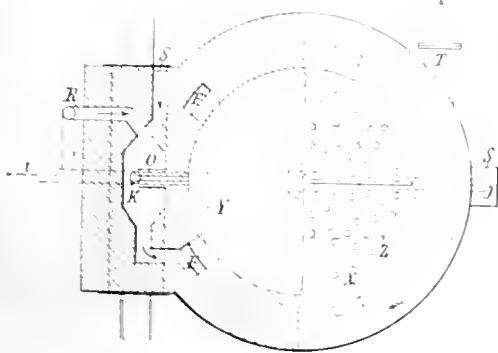


Fig. 2.



The furnaces are constructed of materials containing little or no free silica, "such as magnesia, chalk, and particularly porcelain." The fuel used is preferably gaseous. The gas arrives through the conduit R, ending at the hot gas chamber Y, air passing through the conduits S, to end at the hot gas chamber Z; the gas and air then pass together into the furnace J, and afterwards into the flues F, the regenerators Q, and, lastly, the flues T. The furnace vault is pierced by many small orifices X, through which the salt is passed by a rotary apparatus. The

running off is through orifices kept at a high temperature, as at the outlet K. If the running off is to be continuous, the overflow chute O is used. The dimensions of the orifices are adjusted to the required rate of flow. The fused salt is run into moulds U, a series of which may be mounted on wheels and circulate in the tunnel V, where they cool down slowly. The moulds may be formed of nickel, aluminium, cast iron, &c., or of porcelain or the like.

The salt blocks obtained in these furnaces have a superficial layer of solid and hard crystal surface "resisting moisture." But in some cases it is preferred to dip the blocks, while hot, in melted paraffin; or a thin coating of an alkali silicate may be applied. —I. S.

*Improvements in Apparatus for the Electrolytic Decomposition of Metallic Salts.* C. Kellner, Vienna, Austria. Eng. Pat. 24,274, December 16, 1893.

See under XI., page 494.

*Improvements in the Art of preparing Solutions carrying Salts of Zinc.* P. C. Choate, New York, U.S.A. Eng. Pat. 456, January 9, 1894.

SOLUTIONS containing zinc salts are freed from other metals by subjecting them, in a vat supplied with suitable anodes and cathodes, to a current of electricity from a dynamo electric machine or otherwise. Part of the impurities are deposited upon the cathode, and part go down as precipitates. To prevent the solution becoming acid, purified zinc oxide is added, as required. The zinc oxide for this application is obtained by subjecting crude zinc "fume" or oxide to a moderate roast in a muffle furnace at a temperature from 500 to 800 F. In case lead is present in the solution electrically treated, it is thrown down as sulphate by addition of sulphuric acid. Pure zinc may, if desired, be deposited electrolytically from the solution purified as described.

Another method of purification is adopted for solutions of crude zinc sulphate, such as those obtained by lixiviating roasted sulphur ores of zinc. The cleared solution is crystallised, and the hydrated sulphates obtained are roasted at a temperature between 500 and 900 F., the mass being stirred. The product is treated with water, and the zinc sulphate solution is cleared from insoluble matters, which are reserved for metallurgic treatment. By this process, elements more volatile than zinc are volatilised, and those less volatile are converted into compounds insoluble in water. —I. S.

## VIII.—GLASS, POTTERY, AND ENAMELS.

### PATENTS.

*Improved Means for Ornamenting Pottery Ware.* H. L. Doulton and W. P. Rix, London. Eng. Pat. 2252, February 1, 1893.

THE pottery ware is ornamented by rotating it upon the mandril of a lathe and operating upon its face with a toothed wheel caused to roll along the work and travel in a direction parallel to the axis of rotation. —V. L.

*A Porous and Absorbent Material, chiefly applicable for Tobacco Pipes.* A. H. Smith, London. Eng. Pat. 5555, March 15, 1893.

THIS material, which is called by the inventor "Catharopyr," is made by adding asbestos fibre to a thin "slip" of fine pipeclay (or steatite, or both) mixed with water. The whole is worked up to a plastic mass, which is then moulded

into any required shape and baked in a kiln at a high temperature. The resulting material is refractory to heat, and at the same time is porous or absorbent. When fouled, as for instance, by nicotine, the material can be cleansed by being heated in the fire, after which the article is as good as new.—V. C.

*Improvements in the Glazing and Enamelling of Heavy Earthenware and other Goods, and in Apparatus to be Employed therefor.* T. W. Twyford, Hanley. Eng. Pat. 6612, March 29, 1893.

SMALL articles of pottery ware are readily glazed by dipping into the liquid glaze and then setting them to drain. This cannot be done with large and heavy articles, to which the glaze is generally applied by means of a camel-hair brush, two or more operatives working at a time. This operation has to be performed very quickly, and followed up closely to prevent the edges, which set rapidly, from being "overlaid."

In the present invention the glaze is applied in the form of a spray, the workman moving the spraying apparatus from one position to another until the whole surface of the article has been coated. The principal portion of the apparatus consists of two lengths of tubing converging to a common centre, one for the conveyance of the liquid glaze, and the other for the blast.—V. C.

*Improvements in the Manufacture of Compositions suitable for Paving, Building, Pipe-jointing, and other like Purposes.* W. Sykes, London. Eng. Pat. 7499, April 12, 1893.

ONE gallon of Archangel tar or of Stockholm tar is mixed with two bushels of Portland cement, and the mixture is worked up to a plastic condition. When required for paving, hard wood sawdust is first added to the tar, and during the process of incorporation with the cement the whole is heated in a cauldron. The composition is also suitable for forming damp-proof courses or for forming bricks for building in damp soil.—V. C.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*The Composition of Portland Cement.* B. Kosmann. *Thonind. Zeit.* 1893, 17, 1140.

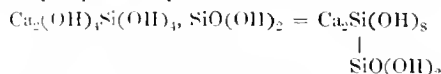
IN opposition to the views held by Le Chatelier regarding the formation of the tri-basic calcium silicate,  $\text{Ca}_3\text{SiO}_5$ , and the hydrate,  $\text{CaSiO}_3 \cdot 2\frac{1}{2}\text{H}_2\text{O}$ , and the presence of the unstable, and therefore objectionable, di-basic compound,  $\text{CaSiO}_4$ , the author puts forth the following theory:—

Silicic acid corresponding to the hydrates with one and two molecules of water being represented by the formula  $\text{H}_2\text{SiO}_3$  or  $\text{SiO}(\text{OH})_2$  (meta-silicic acid) and  $\text{H}_4\text{SiO}_4$  or  $\text{Si}(\text{OH})_4$  (ortho-silicic acid), the constitution of the constitution of the compound  $\text{Ca}_3\text{SiO}_5$  must be—



The molecule  $\text{CaO}$  is only in loose combination, hence this compound immediately splits up when brought into contact with water. The tendency of the fused silicate,  $\text{Ca}_3\text{SiO}_5$ , to dissociate is supposed to be owing to the fact of the molecular equilibrium, and therefore the limit of the capacity to react farther not having been reached before solidification takes place. The decomposition of the compound may be explained by the excess of heat, taken up during fusion, being given up on solidification. This fact of the instability of the fused silicate,  $\text{Ca}_3\text{SiO}_5$ , does not, however, prove the correctness of the supposition that the

hydrated compound,  $\text{Ca}_2\text{Si}(\text{OH})_3$ , has a deteriorating effect upon the quality of the cement. The composition of the hydrated silicate contained in set cement, which Le Chatelier takes to be  $\text{CaSiO}_3 \cdot 2\frac{1}{2}\text{H}_2\text{O}$  or  $2\text{CaSiO}_3 \cdot 5\text{H}_2\text{O}$  can only be correctly expressed by the formula—



It is in fact a hydrated ortho-silicate, to which is linked one molecule of hydrosilicic acid. This bears out W. Michaelis' theory, according to which the cause of the hardening of cement is to be found in the combination of the free hydrated silicic acid with free calcium hydroxide present.—F. M.

*The Calculation of the Fusibility of Clays.* H. A. Wheeler. *Eng. and Mining J.* 1894, 224.

IN connection with the investigation of the clays of Missouri, a large amount of experimental work has been done for determining their fusibilities and other properties. The following considerations are the outcome of this work:—

Bischof, in his "Feuerfesten Thone" (p. 71), gives a formula for calculating the fusibility of a fireclay from a chemical analysis, which is based on the statements that the pyrometric value depends on:—

1. The relation of the alumina to the fluxes (or detrimental).
2. The relation of the alumina to the silica.

From this he derives the following expression for representing what he calls the "Feuerfestigkeitsquotient," or fusibility quotient:—

$$\text{F.Q.} = \frac{\text{Alumina} \times \text{alumina}}{\text{Silica} \times \text{fluxes}}$$

The value of F. Q. he finds to be 14 for the most refractory clays (Saarau, with 1.26 per cent. of detrimental); about 9 for pure kaolins (Zettitz, with 2.00 per cent. of fluxes), 4 for Belgian fireclays (3.0 per cent. of fluxes), and 1 or less for the Rhine fireclays, which have 4 to 7 per cent. of detrimental. From Bischof's statement it follows that the more aluminous a clay is, the more refractory it will be, while the more siliceous or the more impure with detrimental, the less refractory it will be.

Whilst much alumina is indicative of purity in a clay, yet the work on Missouri clays shows no loss in refractoriness when the alumina is replaced by sand, or the clay is very siliceous, whilst every increase in the detrimental decidedly affects the fusibility, especially if the increase is in the alkalis. But the work on the Missouri clays shows that density and fineness of grain have also a very important influence on the refractoriness, and these must be considered in assessing the value of the detrimental present in a clay. Hence Bischof's formula will be totally unreliable, even in clays of similar density and fineness, on account of confusing the silica and alumina as such vital factors of fusibility, when it is mainly, if not entirely, a question of fusible impurities, density, and fineness. When clays are similar in density, and fineness, the refractoriness will be inversely as the detrimental impurities, when the latter are stated in an equation so as to exhibit their proper fluxing values, and if the expression be called the "Fusibility factor," it may be set forth in the following formula:—

$$\begin{array}{c} (\text{A.}) \\ \text{F.F.} = \frac{\text{N}}{\text{D} \times \text{D}'} \end{array}$$

where N represents the sum of the non-detrimental or total silica, alumina, titanic acid, water, moisture, and carbonic acid; D represents the sum of the detrimental impurities, or the iron (as sesquioxide), lime, magnesia, alkalis, sulphuric acid, sulphur, &c.; D' represents the sum of the alkalis, which have been found to have about double the fluxing value of the other fluxes; ferrous oxide is probably about as objectionable, but since it usually oxidises into the less fusible form of sesquioxide in the operation of burning the clay, it is not thus accentuated in ordinary coarse

clays. This formula will be found to give a fairly good comparative value of the refractoriness of clays that do not differ more than 0.2 from one another (the closer the specific gravity the more reliable the comparison) in density, and are of similar fineness of grain. When the clays to be compared differ in density and fineness, then it is necessary to modify formula A by a constant C, that will have different values depending on the density and fineness, so the formula will be—

$$(B.) \\ F.F. = \frac{N}{D \times D' \times C}$$

in which N, D, and D' will have the same values as in formula A; C = 1 when the clay is coarse grained and specific gravity exceeds 2.25; C = 2 when clay is coarse grained and specific gravity ranges from 2.00 to 2.25; C = 3 when it ranges from 1.75 to 2.00; C = 2 when the clay is fine grained and specific gravity over 2.25; C = 3 when it ranges from 2.00 to 2.25; and C = 4 when it is from 1.75 to 2.25. These values of C are only crudely approximate. Sufficient work has been done to show that formula B is the only one that can be used indiscriminately on all kinds of clay, though a very large amount of work has still to be done to arrive at more reliable values for C and the fineness, as the large number of factors involved, makes it a very complex question.

In the table of analyses given herewith examples have been selected from the 110 samples of the Missouri clays that have been completely studied, which fairly illustrate the unreliability of Bischof's formula, the quite satisfactory comparative values given by formula A on clays that are physically similar, and the much more general scope of formula B, which considers physical as well as chemical influences, though it also shows the need of further work to obtain more reliable values for the constant C. Thus, in the St. Louis fireclays, which are all obtained from the same seam, Bischof's formula gives about equal values (1.23 to 1.44) to clays of very different fusibility, and very different values (1.08 to 3.44) to clays of about equal fusibility; the Cape Girardeau kaolin (91.05 per cent. SiO<sub>2</sub> and 1.27 per cent. fluxes) is given a very inferior value for its refractoriness (0.2), though (as the analysis would indicate) it could not be affected by the highest heat of the furnace, while the easily-fusible Winona kaolin is given a higher value (1.5). Again, the Clapper (potter's) clay, which is one of the most, if not the most, refractory clay known, is assigned a value of only 3.7 by Bischof's rule, while the readily fusible Owen-ville fireclay has a value of 4. A further study of the table will show the utter unreliability of Bischof's F.F. as a means of arriving at the relative fusibility of clays.

The table shows that the value of F.F. should exceed 12 when calculated by formula A for coarse, very dense clays, or exceed 10 when using B, for a refractory clay; and the

TABLE SHOWING THE VALUE OF CALCULATED FUSIBILITIES.

Name.	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	H <sub>2</sub> O	Total N.	Alkalis D'.	Total D.	Sp. Gr.	Grain.	Fusibility, F.			Calculated Values.		
									Incipient.	Complete.	Scori- aceous.	Bischof F.Q.	Wheelers.	
													A.	B.
Above														
St. Louis fireclays:														
Christy, washed...	64.35	21.16	8.94	94.45	0.51	4.81	2.13	Fine	2200	2400	2700	1.44	17.7	13.0
„ mine run . . .	61.73	23.56	9.25	94.54	1.00	7.30	2.47	Coarse	2100	2300	2500	1.23	11.4	10.2
Over														
“Rock” or non-plas- tic fireclays:														
Pendleton .....	46.18	38.12	14.01	98.31	1.20	1.06	2.42	Fine	2300	2500	2700	16.0	28.0	18.0
High Hill.....	45.12	40.46	13.34	98.92	0.30	1.06	2.33	Very fine	2450	2550	2700	37.0	73.0	29.0
Kaolins:														
Glen Allen.....	72.34	18.94	7.04	98.28	0.42	1.89	1.80	„	2200	2400	2600	2.7	42.0	15.6
Sterling.....	57.75	27.60	11.43	96.78	0.60	3.24	„	„	2200	2400	2600	4.3	26.0	12.3
Potters' Clays:														
Guthrie.....	47.13	34.98	13.88	95.99	0.52	3.76	2.13	„	2000	2200	Above 2400	6.5	22.0	13.0
Mammoth .....	49.04	34.85	12.33	96.22	0.85	3.93	1.60	„	1800	2100	2400	6.3	20.0	11.0
Brick Clays (Loess):														
Kansas City.....	72.00	11.97	6.42	90.19	3.25	10.11	2.17	Fine	2000	2200	Over 2300	0.2	6.8	5.7
Hartwell.....	60.93	21.51	5.30	87.64	2.03	10.15	1.80	„	1850	2050	2150	0.7	7.2	5.4
Shales:														
Moberly .....	65.01	19.34	6.54	90.85	2.60	9.31	2.41	Coarse	1850	2050	2250	0.6	7.5	7.0

higher the value the more refractory it will be. It should be remembered in consulting the table that no matter how reliable a pyrometer may be—and the best of them need much care and attention—it is difficult to determine precisely what the temperature of a clay is, when it begins to change, on account of its very poor conductivity of heat; differences of over 100° F. were found within less than half an inch on the same bricklet. In burning a clay, it slowly shrinks and becomes harder as the temperature is raised above a red heat (when it begins to lose its chemically combined water), and at a certain point the granular structure of the raw clay can no longer be distinguished, and it is now found to have a hardness of 6.0 to 6.5; this is called the point of incipient vitrification (in the table), or fusibility, and it is necessary to burn clay wares to this point to develop

strength and hardness. If the temperature be raised still higher, it will become slightly harder, or 6.5 to 7.0, and at a certain point will look more or less stony or vitreous, which is called completely vitrified; at this point the clay will not soften or flow or become distorted. If the heat be still further raised the clay will finally soften, vesicles and bubbles will begin to appear as the result of gases given off by the chemical changes that now slowly take place, and finally a very spongy, scoriaceous, pasty mass will result, unless the heat be raised so high as to thinly liquefy the mass, when the gases escape and melted glass results. The first appearance of the bubbles is called the point of scoriaceous vitrification, or fusion, and shows that the clay is failing and would slowly flow or yield under pressure or abrasion. As the range in temperature is usually



from 300 to 60° F. between this first or incipient point and last or scoriaceous or falling point, with a gradual transition from one to the other, it is difficult to state with any degree of accuracy just when the clay falls, and to decide any closer than a rough approximation as to when a clay gradually passes through the above three important stages in its very slow transition from a rigid solid to a very viscous liquid. Hence the pyrometric determination of these three conditions is only approximate, and an error of 25 to 50° in the figures given in the table is quite possible.

Le Chatelier's thermo-electric pyrometer was used in making the temperature observations, which was calibrated to show changes of 25° F., and the samples, varying from  $\frac{1}{2}$  in. by 1 in. by 4 in. to 1 in. by 1 in. by 4 in. were burned in muffle and graphite crucibles that were run at different degrees of heat in coke-fired assay furnaces.

## X.—METALLURGY.

*The Condition of Gold in Quartz and Calcite Veins.* A. Liversidge. A paper read before the Roy. Soc. of New South Wales, September 6, 1893, 13—17.

GOLD occurring in a soft matrix is often distinctly crystallised, as also when it has been formed in a cavity whatever the nature of the veinstone. But the gold contained in massive quartz as a rule is not crystallised, as may be seen by dissolving the silica away by hydrofluoric acid.

—J. H. C.

*On the Origin of Gold Nuggets.* A. Liversidge. A paper read before the Roy. Soc. of New South Wales, September 6, 1893, 17—57.

THE author gives a summary of the various theories which have been put forth to account for the existence of alluvial gold other than "the old and accepted one," that it has been set free by processes of disintegration. After quoting largely from the writings of Simpson, Davison, C. S. Wilkinson, T. Cosmo Newbery, W. Skey, Brough Smyth, Professor Whitney, Professor Fgleston, and others, he gives detail of a large number of experiments made with a view to determine whether a nucleus of gold immersed in a gold-solution and in the presence of such substances as would be likely to occur in nature will increase in weight, and he concludes that gold is deposited when the nucleus is in contact not only with metalliferous sulphides and arsenides which form strong galvanic couples, but also with such substances as iron oxides, charcoal, graphite, sandstone, granite, quartz, clay, and marble, which form but weak galvanic couples with the gold nucleus. He questions whether the common assertion as to the greater fineness of nugget as compared with coarse vein gold has any foundation in fact. With fine alluvial gold there is such a difference, but this he thinks results from the removal of silver and other impurities by solution owing to the larger relative surfaces exposed.

Discussing the question of solutions of gold in natural waters, he concludes that gold is present in meteoric and mine waters (although absolute chemical proof is still wanting) because it is found in recently-formed pyrites, &c., where it must have come from solution. (E. Sonstadt has demonstrated its presence in sea-water.) Furthermore, the author urges that large nuggets could be artificially produced by following the methods used in his experiments, and believes that gold is probably being so deposited at the present day. However, he does not believe that the large nuggets have thus been formed *in situ*, although gold grains and dust may have been appreciably thus enlarged.

—J. H. C.

*Formation of Cast Iron in Zinc Muffles.* E. Jensch. Chem. Zeit. 1894, 7, 101—102.

A mass of ferruginous regulus was found in the residues contained in a zinc retort, weighing more than a kilo. after removal of cinder and clinker, &c. The temperature to which the retort had been exposed did not exceed 1,500° C. The mass contained several per cent. of sulphur, 1—2 per cent. of silicon, about as much graphite, and 84—87 per cent. of iron, the composition not being uniform throughout; hence it was essentially a mixture of pig iron with iron sulphide and oxide.—C. R. A. W.

*The Principal Smelting Reactions of Blast Furnace Slag, considered on Thermo-Chemical Principles.* A. D. Elbers. Eng. and Mining J. 1894, 297—298.

THE formation of blast furnace slag, the by-product of pig iron, begins after the charges have descended to the furnace zone in which the carbonate of lime changes to free lime, when partially reduced oxide of iron and portions of the lime unite with silica and with silicates by fritting or incipient fusion. Descending through the zone of carburisation, the fritting ingredients contract gradually to sintered or semi-vitrified masses, which even carbonic oxide gas (CO) cannot easily penetrate. In these impervious masses considerable portions of partially reduced oxide of iron remain inclosed until the impervious shell or covering melts off; hence the iron ore does not become completely reduced until it arrives at the zone of fusion.

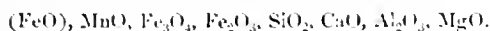
Further down in the zone of carburisation, the descending charges come in contact with the sulphurous gases that arise from the zone of fusion and from the zone of combustion. "As these gases (chiefly SO<sub>2</sub>) become reduced, sulphides form; the greater portion of the regenerated sulphur combining either with metallic iron or with lime." The lime reaction may be supposed to be the same as when sulphur and lime are heated under exclusion of air in a crucible, viz.,  $4\text{CaO} + 4\text{S} = 3\text{CaS} + \text{CaSO}_4$ . When the temperature of the reducing gases is sufficiently high, a part of the regenerated sulphur is apt to combine with glowing carbon to carbon bisulphide, which can also react on the lime ( $2\text{CaO} + \text{CS}_2 = 2\text{CaS} + \text{CO}_2$ ). Both lime reactions require, however, a higher temperature than the formation of ferrous sulphides does ( $\text{Fe} + \text{S} = \text{FeS}$ ). Hence the formation of sulphide of calcium rises with the temperature of the zone of carburisation, whereas the formation of ferrous sulphide increases when that temperature is not high enough for the formation of sulphide of calcium. Later on, still uncombined portions of the lime unite with the ash of the coal or coke, which consists chiefly of aluminous silicates intermixed with spent pyrites. The latter, at the temperature then obtaining, give off more sulphur and pass into the hearth as ferrous sulphide, excepting such minor portions as may become decomposed by coming in contact with sulphates. After collecting in the hearth, the sulphide of lime remains with the slag, whereas the ferrous sulphide—on account of its higher specific gravity—goes into the metal. When the metal is highly carburised and highly superheated, then the ferrous sulphide is apt to become decomposed by the carbon of the metal ( $\text{FeC} + 2[\text{FeS}] = 6\text{Fe} + \text{CS}_2$ ), and the resulting carbon disulphide is apt to become oxidised in passing upward through the zone of combustion, thus furnishing additional supplies of SO<sub>2</sub> for the regeneration of sulphur vapours in the zone of carburisation.

The flushed or tapped slag is apt to contain more or less of accidental ingredients that had not time to separate from it, such as ferrous sulphide, ferrous sulphate, and agglutinated particles, consisting chiefly of unreduced or partially reduced ore intermixed with carburised metal. When the hardened slag is remelted in a cupola furnace, the greater portion of this accidental ferruginous matter is apt to combine with the silicate masses.

Returning to the reactions that lead to the ultimate combination of the fritting ingredients of the descending charges, their progress can be explained as follows, beginning with the formation of simple silicates by the direct union of their constituent oxides: Nearly all of these oxides are, practically, infusible. The specific heat of those that has been

determined is, approximately, in inverse proportion to their molecular or atomic weight. Then absolute fusibility may be assumed to be in inverse proportion—and their absolute melting point, consequently, in direct proportion—to their specific heat. Their actual melting behaviour depends, however, to some extent, on the state of their molecular condensation. Magnesium oxide, for instance, is more fusible than calcium oxide, though its specific heat is considerably higher than that of calcium oxide can be assumed to be. These deviations from the general rule have, however, no practical bearing on the igneous reactions of the respective substances, inasmuch as calcium oxide fuses, with silica, at a lower temperature than magnesium oxide does, and their contingent melting point may, therefore, be assumed as approximately proportionate to their specific heat.

Adopting the latter conclusion as a general rule, the comparative or contingent fusibility of the more important constituents may be assumed to stand in the following order, beginning with the oxide having the lowest specific heat, and consequently the lowest melting point:—



As FeO does not occur in the free state, its specific heat as well as its contingent fusibility have to be inferred from those of  $\text{Fe}_3\text{O}_4$ . According to general rules  $\text{Fe}_3\text{O}_4$  must be more fusible than  $\text{Fe}_2\text{O}_3$ , and FeO more fusible than MnO. That the melting point of rhodonite ( $\text{MnO}, \text{SiO}_2$ ) is considerably lower than that of fayalite ( $2\text{FeOSiO}_3$ ) can only be due to the greater condensation of the ferrous silicate-molecules, inasmuch as the condensation of like molecules ( $2\text{FeOSiO}_2 + 2\text{FeOSiO}_2$ ) involves the absorption of heat, and consequently a proportionate increase in the molecular heat, and a corresponding diminution in the fusibility of the condensed compound.

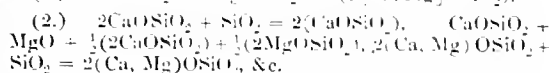
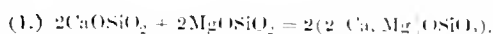
The primary fritting reactions may be defined as "the chemical combination, of molecules of two unlike substances, at a temperature that is below the melting point of either substance." As their mass becomes heated the respective substances accumulate energy according to their specific heat, the one having the higher specific heat accumulating, proportionately, more of it than the other. The excess of the energy that accumulates in the particles of the substance having the higher specific heat is proportionate to the difference in the molecular heat of the two, and can be transmitted by contact. Molecules of silicic oxide and molecules of a basic oxide unite thus, on the surfaces of contact of the respective particles, and in definite proportions, to primary silicate-molecules. As already stated, the amount of the energy that is set free by their union must be proportionate to the difference in their molecular heat, but it must also be larger than the amount of the excess that accumulated in the substance having the greater specific heat of the two, inasmuch as they combine at a temperature that is below the melting point of either of them. Hence, the specific heat of a primary silicate-molecule must be lower than that of either of its constituent oxides.

But the melting point of the most easily fusible simple silicates, of the alkalis, oxide of lead, &c., is higher than that of the respective basic oxides, whereas the melting point of a natural simple silicate of lime (Wollastonite) is considerably lower than that of its base, which is practically infusible. This seeming inconsistency can only be accounted for by assuming that the melted molecules unite, primarily, in the proportions of an equal oxygen ratio ( $2\text{CaO} + \text{SiO}_2, 2\text{Al}_2\text{O}_3 + 3\text{SiO}_2$ , &c.), inasmuch as the total amount of the energy that is set free, when the respective molecules combine in the proportion of 2:1 or 2:3, is greater than that which could be set free if they combined in equal numbers. Moreover, if they combined in the latter proportion,  $\text{CaO} + 2\text{SiO}_2$  would be more fusible than  $\text{CaO} + \text{SiO}_2$ , inasmuch as the specific heat of  $\text{SiO}_2$  is lower than that of  $\text{CaO}$ ; and if the specific heat of  $\text{CaO}$  were lower than that of  $\text{SiO}_2$ , then  $2\text{CaO} + \text{SiO}_2$  would be more fusible than  $\text{CaO} + \text{SiO}_2$ , which is not in accordance with the well-known melting behaviour of mixtures having their constituents in the respective proportions.

If, on the other hand, it be assumed that the primary silicate molecules are single silicate molecules ( $2\text{CaOSiO}_2$ , &c.), then the thermal properties of all silicates, whether artificial or natural, can be accounted for in conformity with thermo-chemical rules. According to these rules the secondary reactions of the combining oxides must then progress in the following order:—

As the fritting temperature rises, the primary silicate-molecules that have formed at the initial temperature recombine, at first, with molecules of the oxide having the lower specific heat of the respective constituents, and, in thus recombining, energy is again set free under evolution of heat. The amount of energy that is thus liberated decreases as the measure of the absorption increases ( $2\text{CaOSiO}_2 + \text{SiO}_2$ , &c.); and when it falls short of the amount that is required for the molecular work of recombination, then no further reactions can take place at the temperature obtaining, inasmuch as additional energy can only be accumulated with advancing temperature.

As the fritting heat becomes more intense, the more refractory of the basic oxides unite, with silica, to primary silicate-molecules, whereas the primary silicate-molecules that have formed at the lower temperature begin to take up molecules of the basic oxides, the specific heat of which is higher than that of the silica; and the simple silicate-molecules combine then to compound silicate-molecules, viz.:—



The latter mode of combination must also be that by which the aluminous silicates of the ore and coal unite with basic oxides, viz.:— $\text{Al}_2\text{O}_3, 2\text{SiO}_2$  ( $\text{Si}_2\text{Al}_2\text{O}_5$ ) +  $\text{CaO} = \text{CaOAl}_2\text{O}_5, 2\text{SiO}_2 = \frac{1}{2}(2\text{CaOSiO}_2) + \frac{1}{2}(2\text{Al}_2\text{O}_3, 3\text{SiO}_2)$ .

From the melting behaviour of natural silicates that have the same constituents as blast-furnace slag, it may be inferred:—

1. That the specific heat of simple silicates is, approximately, proportionate to the specific heat of their constituent oxides.

2. That the specific heat of compound silicates is, approximately, proportionate to the specific heat of their constituent simple silicates.

Applying these deductions to the melting reactions of the fritting ingredients, the following conclusions are arrived at:—

1. As the specific heat of  $\text{CaO}$  is higher than that of  $\text{SiO}_2$ , the melting down of a charge, having the respective constituents in single-silicate proportions, requires a higher temperature than the fusion of a corresponding bisilicate composition.

2. A charge that is high in alumina melts more rapidly than a charge that is low in alumina, because the specific heat of compound silicates of lime and alumina is lower than that of simple silicates of lime ( $\text{CaO}$ ) of corresponding constitution.

3. As the specific heat of magnesium oxide is higher than that of alumina, the compound silicates of lime and magnesia do not melt as readily as the compound silicates of lime and alumina.

From the melting behaviour of hardened slag, and from that of other compounds that consist of amorphous mixtures of silicates, such as glass, it must, however, be inferred that the specific heat of such mixtures is, again, approximately proportionate to the specific heat of their constituent oxides. In other words, the molecular heat of the simple silicate-molecules is lower than the sum of the molecular heat of their constituent oxides, and the molecular heat of the compound silicate-molecules is lower than the sum of the molecular heat of their constituent simple silicates; but the molecular rearrangements that take place when unlike compound silicate molecules unite, absorb just as much energy as was set free by their formation.

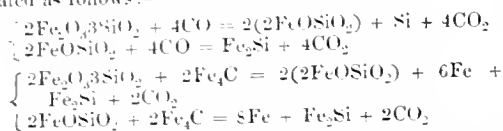
Hence, though compound silicates of lime and alumina melt more readily than simple silicates of lime, the formation of a homogeneous slag requires a higher temperature

when the contents of alumina are high than when they are low, inasmuch as the specific heat of  $Al_2O_3$  is higher than that of  $CaO$ .

It further follows that not all unlike silicates can combine with each other.

As the tritted masses melt and trickle down into the hearth, intimately intermixed with carburised metal, their ferruginous constituents become gradually reduced, and the reduction continues after they have collected in the hearth, because carburised metal passes continually through the collected silicate masses.

The progress of the respective reactions may be illustrated as follows:—



The amount of energy that is absorbed by the reduction of the silicates of iron—as above indicated—is considerably larger than that which is set free by the formation of the resulting silicides of iron (which go, of course, into the metal, where they are apt to become reoxidised by particles of unreduced ore unless the metal is high in carbon). The complete reduction of the ferruginous constituents of the slag is, therefore, only possible when the temperature of the hearth is very high and when the metal is highly carburised. But when the furnace is working well, then the temperature of the furnace is also proportionate to the fusibility of the charges. Hence the reduction of the ferruginous constituents is, practically, proportionate to the specific heat of the collected silicate masses.

The charges that are fluxed with dolomitic limestone are usually lower in alumina than the charges that are fluxed with calcite ( $CaCO_3$ ); and the specific heat of the magnesian slag that is low in alumina is higher than that of the calcareous slag that is high in alumina.

The specific heat is also lowered by an increase in the proportion of the silica; hence—as regards ultimate results—it may be said that the contents of the ferruginous constituents increase in a measure with the acidity of the slag; and for slag of the same silicate constitution: 1. That in calcareous slag the proportion of their contents is approximately in inverse proportion to the contents of alumina; 2. That in magnesian slag the proportion of their contents rises in a measure with the proportion of the alumina.

The writer has found that blast-furnace slag of the magnesian type is practically free from combined iron (in contradistinction to the mechanical admixtures heretofore mentioned) when it is approximately of single-silicate constitution, and when the contents of alumina do not exceed the proportion indicated by the molecular formula:  $12(Ca, Mg)O, Al_2O_3, 7\frac{1}{2}SiO_2$ , the relative proportions of  $CaO, MgO$ , being: 6—7  $CaO$ , 5—6  $MgO$ . As the reduction of manganous silicate requires a higher temperature than the reduction of ferrous silicate, it may also be said that the reduction of the ferruginous constituents increases in a measure with the contents of manganous oxide.

*The Nickel Industry.* J. H. L. Vogt. Stahl und Eisen, 1894, 14, 23.

With the increasing demand for nickel-steel, a further increase in the production of nickel is to be expected. In 1868 the world's production of nickel was only 700 tons, while this figure increased to 2,500 tons for the year 1890, and in 1891, in consequence of the Canadian output, to 4,500 tons for that year. The Canadian ore contains about 2.65 per cent. of nickel, and as much copper; the Norwegian containing about 2 per cent. of nickel, only a trace of copper, but more cobalt. A ton of ore in Canada costs 20 shillings, so that 1 kilo. of nickel, in the ore, costs 9½d.

The ore is submitted to the following treatment:—(1.) Open roasting. (2.) Fusion in water-cooled blast-furnaces to nickel-matte of 15—16 per cent. Ni and 15 per cent. Cu. (3.) Fusion in converter with blast to 10 per cent. of Ni. (4.) Roasting in reverberatory furnaces. (5.) Chlorinating,

roasting, and extraction of copper as chloride. (6.) Removal of iron by heating in reverberatory furnaces with iron pyrites; the nickel sulphide is then roasted and reduced to metal. The cost, inclusive of ore, amounts to 2s. 5¾d. per kilo. of nickel.

The nickel Bessemer process is carried out at three works in Canada already, and worked very satisfactorily, yet there is much room for improvement. The wealth of Norway in nickel ores, deserves more considerable exploitation, and by good work, such as is attainable through the employment of water-power for refining by electrolysis, nickel may be produced cheaper there than in Canada.—W. B. H.

*Determination of Carbon in Iron and Steel.* W. Hempel. Zeits. ang. Chem. 1894, 1, 22—24.

See under XXIII., page 421.

*The Reliability of the Principal Methods of Determining Total Carbon in Iron.* Gottig. Abhandl. d. Vereins z. Betord. d. Gewerbeleisses, 1893, 8, 321.

See under XXIII., page 421.

*Silicon Carbide ("Carborundum") for Cutting Steel.* O. Mühlhäuser. Zeits. ang. Chem. 1893, 637.

See under XI., page 402.

*Volumetric Estimation of Lead in Tin Solutions and Platings.* P. Bayrac. Jour. Pharm. Chim. 1893, 5 Sér. 28, 500.

See under XXIII., page 420.

*A Simple Method for Determining Foreign Metals in Commercial Copper.* W. Hampe. Chem. Zeit. 17, 1691.

See under XXIII., page 421.

*Decomposition of Liquids by Contact with Powdered Silica.* G. Gore. Phil. Soc. of Birmingham, 1893.

See under Analyt. and Scient. Notes, page 428.

## PATENTS.

*Processes of Separating Metals Covering Scraps, Clippings or other Pieces of Metal of all kinds.* F. E. de Fursac, J. C. Moisset, and J. B. J. Very of Orléans, France. [Under International Convention.] Eng. Pat. 3583, September 9, 1892.

The apparatus employed consists of one or more receivers having perforated walls grouped around a rotary shaft and permitting the repeated and alternate contact of the metal to be treated with the liquid and with the air. This is illustrated by drawings, but the use of any other apparatus, exposing mechanically, alternately, and completely the surfaces to be treated, is also claimed.—J. H. C.

*Coating Iron and Steel with Brass and other Metallic Alloys.* A. V. C. Fenby, and J. B. Fenby, of Sutton Coldfield, and G. Moore, Erdington, Warwick. Eng. Pat. 1906, January 28, 1893.

The articles to be coated are first thinly coated with copper, zinc, or other suitable metal by any of the ordinary methods, and then dipped in a bath of molten brass or other alloy, which is maintained at a suitable temperature, while its surface is protected by a layer of borax or other flux.

—J. H. C.

*Improvements in or relating to the Treatment of Metallic Ores.* C. G. Richardson and A. B. English, Toronto, Canada. Eng. Pat. 2119, January 31, 1893.

Ores or matters containing nickel or copper, or both combined, are finely ground, and then fused in a suitable furnace with a proper proportion of dehydrated gypsum or other suitable sulphate, so as to remove any iron present in the state of a soluble silicate, and leave the nickel and copper in the state of sulphide. The sulphurous acid given off during the fusion may be used for the preparation of sulphuric acid or such other purpose, as may be desired.

—J. H. C.

*Improvements in the Treatment of Galena for the Production of Lead, Lead Sulphate, and Lead Oxide.* A. Macdonald, London. Eng. Pat. 2883, February 9, 1893.

The galena is melted and treated in a converter by blowing air or oxygen into or upon it, whereby a large proportion of it is driven off as sulphate of lead fume, which may be collected in any known manner, the rest being left as metallic lead.

It is preferred in the first place to charge the converter with molten lead, and to blow air into this, whereby litharge is produced and the temperature is raised, when the galena may be added. In some cases sulphur is added during the blow. Any silver present is concentrated in the last portion of the metallic lead.—J. H. C.

*Improvements relating to the Purification of Metals in their Moulds, and in the Production of Dense and Flawless Castings, and to Apparatus therefor.* E. Taussig, Bohrenfeld, Holstein, Germany. Eng. Pat. 3503, February 16, 1893.

AFTER casting in the ordinary way, the metal is remelted while still in the mould by passing through it a current of electricity, the mould being placed for this purpose in a hermetically closed chamber, while the gases generated are drawn off by means of a fan or vacuum pump, so producing a more or less complete vacuum.—J. H. C.

*Improvements in and relating to the Manufacture of Zinc Oxide.* A. Gray, Glasgow. Eng. Pat. 4310, February 28, 1893.

Waste cuttings of zinc, zinc ashes, skimmings, &c. are burnt with coke in a furnace of the cupola type, having at the base a grating of either solid or hollow furnace bars, through which a blast of air or steam, or of both, is forced from a spiral tube with jets placed under the bars. Sufficient air is supplied to burn the fuel and to oxidise the zinc.

The products of combustion are drawn through a flue into a chamber, where the zinc oxide is collected by the aid of a spray of cold water, by steam, or by other means.

—A. W.

*Improvements in the Treatment of Slimes, or Finely-divided Ore containing Precious Metals.* C. Raleigh, Johannesburg, S. Africa. Eng. Pat. 5163, March 9, 1893.

To prevent settling or packing of the slimes in the process of extracting precious metals by the chlorine, cyanide, or other lixiviating process, any suitable apparatus for pulsating, vibrating, or jiggling the material under treatment by the injection of air, water, or gas is employed. The inlet for injection may be under or over the filter bed, or at the side of the vat, according to the motion required. The leaching solution may take the place of the water jet, and oxygen gas can be used with advantage to accelerate the solution of the gold or silver.

Muddy water or solution may also be used to increase the density of the leaching solution, so as to further prevent the rapid settling of the grains of ore.

The exact method of application depends on the kind of material under treatment.—A. W.

*An Improved Process for Coating or Plating Non-metallic Articles with Metal.* T. M. Ash, H. W. Gill, and L. Green, Birmingham. Eng. Pat. 5327, March 11, 1893.

ARTICLES such as paper, papier-maché, and other fibrous or textile articles, as well as leaves of plants or other natural objects, are covered with a varnish or other adhesive compound not soluble in water, with which copper or tin have been intimately mixed in a minutely divided state. After washing or partial drying the articles are immersed in a bath of nitrate of silver or other metallic bath until sufficiently coated. Or, if preferred, the object to be treated may be varnished and dusted over with the finely-divided metal before immersion in the silver bath.—J. H. C.

*Improvements in or relating to the "Thomas" Metallurgical Process.* H. Wild, Peine, Germany. Eng. Pat. 6219, March 23, 1893.

This patent refers to the duration of the overblow in the "Thomas" process. As a rule the bath is too much blown, whereby too much iron oxide is formed in the slag and rephosphorisation of the metal takes place. To remedy this the process is carried out as follows:—The first charge is blown in the manner hitherto practised; the necessary qualitative and quantitative tests are previously taken; the carbon, phosphorus, and manganese in the finished product are severally determined and the Thomas slag is analysed in regard to its proportion of iron. On the supposition that the quality of the finished product is satisfactory, comparison is made of the proportion of iron contained in these several slags with each other, and should they be found to differ, the attempt must be made still further to reduce the lowest proportion found by a reduction of the time of overblow. It is maintained that by the shorter duration of overblow there is a reduction of the waste, a smaller consumption of lime, spiegel, &c., a more even quality of metal, and a more valuable slag.—A. W.

*An Improved Process for the Utilisation of Old Articles of Tin Plate, such as Boxes, Cansisters, and the like.* A. Gutensohn, London. Eng. Pat. 6238, March 23, 1893.

THE old tins are first sorted and those with solder joints are heated in pots in a furnace to remove the solder. If the remaining pieces of tin plate are sufficiently large they are cleaned by soda, lime, or other cleaning material, and are re-tinned and passed through finishing rollers. The small pieces which cannot be again made into tin plate are freed from their coating of tin by any known method and used as scrap iron.—A. W.

*A New or Improved Process for Recovering Zinc or Spelter from Waste Galvanised Iron or Steel, and Furnaces and Appliances for the said Process.* W. Hayward, Wolverhampton. Eng. Pat. 7252, April 8, 1893.

THE waste is heated to a temperature somewhat higher than the melting point of zinc in closed or partially closed heating chambers, by which means the zinc is melted off and runs through a hole in the floor of the chamber into an open receptacle or into a vessel of water. The chamber preferred is a kind of reverberatory furnace.—J. H. C.

*Improvements in the Manufacture of Iron and Steel.* A. Sattmann and A. Homatsch, Donawitz, Austro-Hungary. Eng. Pat. 8057, April 21, 1893.

IN the specification, furnaces are described of special construction, for the carrying on, either continuously or intermittently, of the manufacture in all its stages of definitely carburised iron, from its ore. Gaseous fuel is used for heating the ores and fluxes, and for reducing and carburising the spongy iron, solid fuel for melting the spongy iron, and oxidising gases for refining the molten

carburised iron after separating the slag. These fuels are introduced through nozzles and doors according to the local requirements of the furnace. Various ways in which this can be done are described, the furnace being of the blast-furnace type with the additions near or below the bed.

—A. W.

*An Improved Amalgamating Apparatus, to be Employed either Alone or in Combination with Concentrating Apparatus or Buddles.* W. A. Green, Aberystwyth. Eng. Pat. 11,182, June 7, 1893.

In amalgamators of the circular pan and revolving muller type the pan is lined with copper, to the surface of which the mercury adheres, instead of as heretofore being entirely constructed of iron. The object of this is to prevent any of the ore creeping between the surface of the pan and the underside of the layer of mercury therein, thus escaping proper amalgamation by the action of the muller. Furthermore, instead of making the muller with a flat surface for acting upon the ore and mercury, such surface is made with a spiral rib so that as the muller revolves it causes the ore, which is fed through a central opening in it, to be slowly advanced from the centre to the outer circumference of the pan.—A. W.

*Process and Apparatus for the Extraction of Zinc.* L. V. T. Lynen, London. Eng. Pat. 16,180, August 28, 1893.

The retorts used are all connected to one or more common condensing chambers, by which means a proper regulation of temperature is facilitated. The furnace is entirely closed so that no escape of zinc vapour takes place. The retorts are mechanically charged, combustible envelopes being used for each charge to facilitate this.—J. H. C.

*Improved Means Applicable for Use in Extracting Gold and Silver from Auriferous and Argentiferous Solutions.* C. Raleigh, Johannesburg. Eng. Pat. 23,241, December 5, 1893.

The solutions are filtered through granular or permeable electrodes, which act in conjunction with a current of electricity to decompose the solutions in their passage. The precious metals are precipitated and deposited in the interstices of the filtering material, and the resultant solutions are used for lixiviating a fresh charge of ore. The electrodes are composed of fragments of loose or compressed charcoal, carbon, metal, metallic alloys, or amalgams. The precipitation may be expedited when necessary by artificial heat and the introduction of air, oxygen, or hydrogen, and either with increased or reduced pressure.—J. H. C.

*Improvements in Methods of Casting Solid Ingots of Steel.* W. P. B. Urlick, Newark, U.S.A. Eng. Pat. 23,425, December 5, 1893.

The patentee states that the hollowness of the upper end of steel castings is due to gases in the metal, evolved during the cooling process, being imprisoned by the hardened shell which first forms at the head of the metal. He overcomes this by introducing, at a suitable moment, when the metal is neither too fluid nor too pasty, a rod into the upper end of the ingot, whereby a cavity is formed which allows the gases to escape. This cavity is then filled up by pouring into it molten steel, which unites with the remainder and forms a homogeneous whole.—A. W.

*Improvements in Furnaces for Distilling Zinc, Cadmium, and the like.* F. W. Golby, London. From C. Fraenkel, of Schweidnitz, Prussia. Eng. Pat. 23,979, December 13, 1893.

In these furnaces magnesite is used instead of fire-clay for the retorts. The magnesite is formed into bricks of a peculiar shape, and of these bricks the retorts are built up. The heating is effected by the gases produced in a generator.—J. H. C.

*An Improved Process of Removing Tin from Tin Scrap or Tin-coated Metal.* T. G. Hunter, Philadelphia, U.S.A. Eng. Pat. 132, January 2, 1894.

The tin scrap is placed in a grate or basket of gun-metal or other suitable material, which is suspended in a tank, heat being applied by means of steam when necessary. The scrap is then treated with a neutral solution of sulphate of copper or other suitable sulphate until all the tin is separated in metallic form; metallic copper is also formed, and both metals fall to the bottom of the tank, together with small quantities of minor impurities. The bulk of the iron remains in the basket, and may be removed as required. The solution of sulphate of iron formed in the operation is drawn off and the mixed metallic copper, metallic tin, and other substances present are utilised in any way known to the arts.—J. H. C.

*Improvements in the Treatment of Zinc-Lead-Sulphide. Ores Carrying Gold or Silver, or Gold and Silver.* J. C. Fell, London. From The Emmens Zinc Company, New York, U.S.A. Eng. Pat. 2041, January 30, 1894.

The object of this patent is to remove the zinc existing in refractory ores containing lead, gold, or silver. The ore is crushed, roasted in an oxidising atmosphere to dull redness to convert the metallic contents to sulphates and oxides, after which the roasted material is finely pulverised and leached with water containing about 1 per cent. of ferrous sulphate. This leaching extracts the zinc sulphate present in the ore, and the ferrous sulphate precipitates any silver that may be dissolved.

The ore is then leached with a solution of ferric sulphate, containing a little ferrous sulphate, which dissolves and removes any zinc that may be left by the first leaching in the state of oxide or sulphide. The residue is again leached with water containing ferrous sulphate to wash out any liquid remaining from the second leaching, and is then a finished concentrate containing the whole of the original lead, silver, and gold. The zinc may be recovered from the liquors by crystallisation or any other convenient means.—A. W.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

*Silicon Carbide ("Carborundum").* O. Mühlhäuser. Zeits. ang. Chem. 1893, 637—646.

ACHESON discovered (this Journal, 1893, 847) that when a mixture of silica and carbon is heated to a high temperature in the electric furnace, carbon monoxide is evolved and a carbide of silicon produced. The reaction has been studied a little more in detail by the author. The mixture, which is placed in the electric furnace, contains powdered coke 100 kilos., Millington sand 100 kilos., and common salt 25 kilos. The function of the salt is purely a mechanical one. After the operation is over it cements the sand and coke particles together, and so facilitates the separation of the crystalline "carborundum." Some of the impurities in the raw materials, such as iron, calcium, magnesium, &c., are also converted into carbides; and the purer the raw materials, the more nearly colourless are the crystals of silicon carbide. There is formed at the same time a small quantity of amorphous carborundum just above the zone in which the crystalline compound is obtained.

As regards their hardness, the crystals lie between sapphire and diamond, and in the scale of hardness have the value 9.5. Used in connection with a polishing wheel, carborundum is able to cut holes in the hardest steel, and it is stated that the temper of the steel is in no wise destroyed. As an abrasive it can do the same work as corundum, in one-third to one-fourth of the time.

In order to determine the composition of carborundum, the crystals were ground up in a steel mortar and subsequently in an agate mortar, and then submitted to the

process of elutriation. In order to get samples for silicon determinations it is sufficient to allow the substance to settle for one minute before decanting. For carbon determinations, however, the carbide must be very finely divided, and for this purpose five minutes' settling are necessary. The carbide cannot be burned in oxygen, but it is readily oxidised by igniting with lead chromate. The composition of the carboreundum so obtained is given in the following table:—

	I.	II.	III.
Si.....	62.70	62.21	61.59
C.....	36.26	36.35	34.47
Al <sub>2</sub> O <sub>3</sub> and Fe <sub>2</sub> O.....	0.93	1.33	0.53
CaO.....	0.00	0.00	0.35
MgO.....	0.11	0.12	0.06

Pure silicon carbide may be obtained by igniting the very finely-divided crude material in oxygen, boiling out with caustic potash (1:3), and then with hydrochloric acid. It is afterwards treated with hydrofluoric acid and thoroughly washed. It then has the composition—

	I.	II.	Calculated.
Si.....	69.19	69.19	70.0
C.....	29.72	30.21	30.0
Al <sub>2</sub> O <sub>3</sub> and Fe <sub>2</sub> O.....	0.38	0.46	..
CaO.....	0.19	0.15	..
MgO.....	0.06	0.00	..

The specific gravity of the pure substance at 15° C. was found to be 3.22. The author has also analysed various products formed during the reaction, and also the gases evolved, and recommends that the carbon monoxide should be burned before allowing it to escape.—J. S.

#### Electrolysis of Alkaline Chlorides. E. Oetzel. Chem. Zeit. 18, 69—70.

THE electro-motive force requisite to decompose sodium chloride in aqueous solution may be reckoned thus, according to Nourrisson:—

Heat absorbed—	
NaCl = Na + Cl.....	— 364 calories.
H <sub>2</sub> O = H <sub>2</sub> + O.....	— 684 „
	— 1,048
Heat evolved—	
Na + O + H + H <sub>2</sub> O.....	+ 1,118 „
Formation of compounds of chlorine and oxygen.....	say + 60 „
	+ 1,178
Net heat absorption.....	— 470

470 calories represent  $\frac{470}{232} = 2.02$  volts.

These figures, however, are open to correction: the heat disturbance due to the formation of hypochlorous and chloric acids is a secondary result, not affecting the primary electrolysis, and is, moreover, of negative sign; in any case it should not be brought into account. The following figures more nearly represent the E.M.F. requisite for the primary decomposition:—

	Gramme Calories.
— (Na, Cl, H <sub>2</sub> O).....	— 96,510
— (H <sub>2</sub> , O).....	— 68,500
+ (Na, O, H, H <sub>2</sub> O).....	+ 111,510
Sum.....	— 53,000

53,000 gramme calories represent  $\frac{53,000}{23.067} = 2.30$  volts.

Values of 2.25–2.28 volts were obtained by the author as the actual potential difference subsisting between gas carbon electrodes whilst electrolysing common salt solution with a porous earthenware diaphragm, using currents varying between 3 and 60 amperes; practically the same values are found with potassium chloride solution. As the action goes on, caustic soda (or potash) solution accumulates round the cathode, and brine saturated with chlorine round the anode; these two liquids tend to intermix by diffusion through the porous septum, so that more or less hypochlorite is formed. This, in the neighbourhood of the cathode, acts as a “depolariser,” becoming reduced to chloride; but in the vicinity of the anode a different result is brought about, the hypochlorite being partially oxidised to chlorate and partially electrolysed directly, with production of caustic soda and hydrogen at the cathode as before, and of hypochlorous acid and oxygen at the anode. In similar fashion the chlorate produced by oxidation of the hypochlorite partly diffuses to the cathode and is there reduced, and partly becomes electrolysed, evolving at the anode chloric acid and oxygen. On the whole, the more porous the septum, and the more concentrated the caustic soda solution round the cathode, the greater the production of oxygen compounds of chlorine.

The gas evolved is never pure chlorine, but always a mixture of chlorine and oxygen. The latter is usually attributed to the electrolysis of hypochlorite, but is also due to (and probably principally caused by) the direct electrolysis of the caustic soda solution first formed, this becoming decomposed into sodium, hydrogen, and oxygen, just as the sodium chloride is split up into sodium and chlorine. The sodium thus eliminated at the cathode necessarily acts upon water there, reproducing caustic soda; so that the caustics tending towards the evolution of oxygen instead of chlorine are continually increasing in magnitude. The particular fraction of the total current which at any given moment electrolyses caustic soda rather than sodium chloride, depends on the relative richness of the solution in these compounds respectively; for equal concentrations the conductivity of caustic soda solution is greater than that of brine; moreover, the E.M.F. requisite to electrolyse caustic soda solution is only 1.5 volts, or 0.8 volt less than that requisite for sodium chloride. When the chlorides of the metals of the alkaline earths (lime and magnesia) are electrolysed, the lime (or magnesia) formed at the cathode is comparatively insoluble, and consequently is not electrolysed in the way that caustic soda solution is decomposed; accordingly the chlorine evolved is practically free from oxygen. On the other hand, when sodium sulphate is electrolysed, sulphuric acid is formed at one electrode and caustic soda at the other, both of which substances are good electrolytic conductors; so that the work of the current is divided between the three substances, original sulphate, sulphuric acid, and caustic soda.

—C. R. A. W.

#### Employment of Electrical Heat for Concentrating Sulphuric Acid. C. Häussermann and F. Niethammer. Chem. Zeit. 17, 1907—1908.

See under VII., page 391.

#### PATENTS.

##### Improvements relating to the Extraction of Metals by Means of Electricity, and to Apparatus therefor. E. Taussig, Bahrenfeld, Holstein, Germany. Eng. Pat. 3573, February 17, 1893.

THE inventor describes an arrangement in which the electric furnace, provided with electrodes of large size, is enclosed in a case which can be kept exhausted during the working process.

The moulds, into which the metal obtained is to be cast, are also exhausted during the running of the metal.

It is claimed that the metal obtained is purer, some of the impurities usually present when non-exhausted furnaces are employed being withdrawn by the pumps while in a gaseous state; and further, that the addition of fluxes, carbon, &c., is in most cases superfluous.—E. T.

*Improvements in the Electrolytical Decomposition of Chlorides and Mixtures thereof, and in Apparatus for the Purpose.* F. M. Lyte, London. Eng. Pat. 7591, April 13, 1893.

This invention is illustrated by a description of the arrangements suitable for the electrolysis of lead chloride. In a vessel of suitable mechanical strength, but not of chlorine-proof material, is melted a sufficient quantity of lead by means of a furnace arranged below. An inverted bell of some material that must be chlorine-proof, but which, as it has to withstand no great pressure, need not be mechanically strong, is placed so that its rim is some way below the surface of the molten lead. A tube passes through the top of the bell nearly to the surface of the lead, and serves for the introduction of lead chloride to form a fairly deep layer above the metal. The current is passed into the apparatus by carbon anodes dipping into the fused chloride, the molten lead forming the cathode. The metal reduced flows away by a suitable hot overflow pipe; the chlorine is led away by a pipe at the top of the bell.

The anodes are rods with a hole passing in each, from the top nearly to the bottom. In this is placed an easily-fusible metal—such as lead—which, remaining molten during the action of the furnace, makes good contact both with the carbon and with a metal rod attached to a terminal, and dipping into it. In some cases a split springy tube replaces the fusible metal. The object is to reduce the electrical resistance.—E. T.

*Improvements in or connected with Cells for Electric Batteries.* H. C. W. Emery, London. Eng. Pat. 8571, April 28, 1892.

As arrangement for removing the exciting liquid from the electrodes by changing the position of the containing cell.

The electrodes are fixed at one end of the latter, and when in use are vertical and immersed partially in the liquid. By turning the cell on to its side, however, the electrodes rest horizontally above the surface of the liquid, and are therefore preserved from waste during disuse of the cell. A partition can be used, reaching from the top nearly to the bottom, in order to prevent splashing of the liquid—though no arrangements are described for the escape of air from behind the partition.—E. T.

*Improvements in the Manufacture or Production of Soda Crystals.* H. R. Broome, Ross-lea-Lancaster, and M. Guthrie, Liverpool. Eng. Pat. 8907, May 3, 1893.

See under VII., page 394.

*Improvements in Apparatus for the Electrolytic Decomposition of Metallic Salts.* C. Kellner, Vienna, Austria. Eng. Pat. 24,274, December 16, 1893.

ADAPTED for the decomposition of salts where mercury is used as cathode. Below the surface of the electrolyte dips a vessel, which is glazed at the sides, but porous at the bottom. This porous bottom is covered by a thin layer of mercury forming the cathode. The anodes are either at the sides of the semi-porous vessel or are placed below, in which case deflecting vanes are arranged to carry the gas from the neighbourhood of the porous bottom. By this arrangement it is claimed that the mercury is kept from direct contact with the electrolyte; whilst, by keeping a suitable "reaction fluid" above the mercury, any waste of the latter is prevented, the "cation" being yielded up by the mercury directly to this "reaction fluid."—E. T.

*Improvements in Dry Batteries.* H. T. Johnson, New York, U.S.A. Eng. Pat. 24,871, December 27, 1893.

The carbon electrode used in dry batteries is made with a grooved or corrugated surface, and may be perforated or provided with numerous knobs or projections. The object in each case is to prevent loss of contact between the carbon and surrounding material in case of any shrinking or displacement of the latter, and at the same time to prevent the electrode from being easily drawn from the cell in case

of breaking or cracking of the seal. Greater contact surface is of course given at the same time, which is an advantage.—E. T.

*Improvements in or relating to the Vaporisation of Petroleum and other Liquids.* A. J. Boulton, London. From S. C. Penchen and P. Clarke, both of Toronto, Canada. Eng. Pat. 340, January 6, 1894.

See under II., page 380.

*Improvements in the Art of Preparing Solutions carrying Salts of Zinc.* P. C. Choate, New York, U.S.A. Eng. Pat. 456, January 9, 1894.

See under VII., page 395.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*Oxidation of Fatty Oils.* W. Fahrion. Chem. Zeit. 17, 1848—1850.

IN spite of various researches, the exact nature of the chemical actions taking place when oils "dry" (by the spontaneous oxidation effected by exposure to air in thin films) remains for the most part obscure. Mulder regarded the chief fatty acid of linseed and other drying oils as  $C_{18}H_{32}O_2$ , and considered the effect of "drying" to be that the glyceridic portion became wholly destroyed, whilst the supposed linolic acid,  $C_{18}H_{30}O_2$ , became oxidised, first to linonic acid,  $C_{18}H_{28}O_3$ , and then to a neutral body, linoxyn,  $C_{32}H_{54}O_{11}$ . Hazura and Bauer found the acids of the drying oils to be mixtures in different proportions of oleic acid,  $C_{18}H_{34}O_2$ , linolic acid,  $C_{18}H_{32}O_2$ , linolenic acid,  $C_{18}H_{30}O_2$ , and isolinolenic acid, also  $C_{18}H_{30}O_2$ . Of these the glycerides of the last three are not broken up by the oxidation, whilst the glycerides of saturated acids (such as palmitic, myristic, &c.), when present, are first attacked with destruction of the glyceridic part of the molecule, and liberation of free fatty acids; olein is similarly attacked to some extent, whilst linolin and linolenin take up oxygen without losing glycerol, forming a series of bodies of which the final member is the glyceride of the acid  $C_{18}H_{26}O_7$ , this substance being simultaneously a species of anhydride. The author has investigated the nature of the intermediate products so far as the acids thence obtained by saponification are concerned, the method of experimenting being to increase largely the surface exposed to the air by saturating chamois leather with the oil to be examined; after long continued exposure (in some cases for many months, partly in sunlight,) the leather was cut into fragments and treated with cold ether; the ethereal solution was repeatedly shaken with water to remove nitrogenous matters taken up from the leather and evaporated; the fatty acids obtained on saponifying the residue, were partly soluble, and partly insoluble in petroleum ether, the latter portion consisting of the products of oxidation, the former of unoxidised fatty acids. The oxidised fatty acids were readily soluble in ether, in alcohol, and in aqueous ammonia; from the latter solution fractional precipitation with barium chloride enabled a partial separation to be effected. From the results of combustions and determinations of the "iodine number," &c., of the products so obtained, the conclusions are drawn that linolic acid (cotton seed oil), furnishes a series of oxyacids where successively 1, 2, 3, and 4 atoms of oxygen are added on, forming respectively oxy-, dioxy-, trioxy-, and tetraoxy-linolic acids, the aerial oxidation action going no further than this last. Similarly, linolenic and isolinolenic acids (linseed oil) furnish a parallel series, the final product appearing to be an anhydride of hexoxylinolenic acid,  $C_{18}H_{26}O_7 = C_{18}H_{30}O_6 + H_2O$ , six oxygen atoms being added on; whilst jeconic acid (cod liver oil) gives analogous products, the final one being a double anhydride of pent-oxyjeconic acid,  $C_{18}H_{26}O_5 = C_{18}H_{30}O_4 + 2H_2O$ . As the



addition of oxygen goes on, the acids gradually become more and more solid; the original unoxidised bodies are perfectly fluid, whilst the more oxidised members are completely solid and can be powdered. The solubility in ether decreases as the oxidation progresses, whilst the opposite is the case as regards the solubility of the barium salts in water. The addition of oxygen is mostly of such a character as to form a kind of anhydride, which takes up water under the influence of alkalis, &c.; as oxidation of a glyceride progresses, the glyceridic character of the product is still maintained in the case of unsaturated acids, the original colour of the oil being maintained.—C. R. A. W.

*The Oxidation of the Fatty Oils.* W. F. Allen.  
Chem. Zeit. 17, 1453.

The oxidation of the fatty oils by the atmospheric oxygen is a very slow process, which, however, can be considerably hastened by exposing them in a very fine state of subdivision. For this purpose Vogel impregnated cotton with the oil, but the author suggests soft, porous leather as a better vehicle. The following results were obtained by cutting from a very soft sheepskin leather strips, weighing about 1 gram, each. These strips were saturated with about their equal weight of oil and, together with an unimpregnated strip of skin, suspended on brass hooks. The changes in the weights of these strips were estimated from time to time and recorded in per cent. of the weight of the oil used:—

	Leather.	Olive Oil.	Sesame Oil.	Colza Oil.	Cotton-seed Oil.	Poppo-seed Oil.	Nut Oil.	Linseed Oil.	Cod Liver Oil.
Iodine number.....	—	82.1	119.2	162.4	169.2	137.9	129.2	155.8	171.6
After 1 day .....	0	0.2	0.1	0.1	— 0.1	0.3	— 0.2	0.1	— 0.6
" 2 days .....	1.0	1.0	1.2	1.1	0.8	2.0	2.0	1.5	1.1
" 3 " .....	1.3	1.2	0.9	1.3	1.4	3.2	1.4	2.0	8.1
" 4 " .....	0.9	0.9	0.5	1.0	1.2	4.3	7.1	3.8	16.0
" 5 " .....	1.4	1.4	1.1	1.8	3.1	7.1	9.7	12.3	10.9
" 6 " .....	— 0.6	— 0.4	— 0.5	0.5	2.2	7.3	8.4	11.8	8.0
" 7 " .....	1.3	1.1	1.0	2.5	5.0	9.7	9.6	13.2	16.4
" 8 " .....	0	— 0.1	0	2.0	4.7	7.0	7.2	10.4	8.0
" 9 " .....	0.5	0.5	0.7	2.8	5.7	7.3	7.3	11.3	8.5
" 10 " .....	1.8	1.7	2.0	4.6	7.4	8.0	8.3	11.8	10.4
" 14 " .....	— 0.5	— 0.4	1.1	2.3	2.5	3.3	4.0	7.0	6.8
" 3 weeks .....	1.1	0.7	4.7	3.2	1.6	2.7	4.2	8.2	6.3
" 4 " .....	3.3	3.5	6.6	6.3	5.2	6.7	8.4	12.6	11.8
" 6 " .....	— 2.5	— 2.5	— 0.5	— 0.2	— 1.4	— 0.3	— 1.3	4.5	4.3
Maximum.....	—	0.2	3.6	2.8	5.6	8.4	9.0	12.4	9.5
Livache's test .....	—	1.7	2.4	2.9	5.9	6.8	7.9	11.3	6.4
Calculated .....	—	5.2	7.0	6.5	6.9	8.6	9.4	11.1	10.0

These figures show that even the moisture of the strips of leather impregnated with oil varies in a similar degree as the moisture of the unimpregnated strip. While in Livache's test the maximum of the increase in weight is reached after two days' exposure, it takes very much longer in this new test, the time required probably varying with the temperature. With one or two exceptions, the above figures do not agree with those obtained by Livache's test. The theoretical oxygen absorption, calculated by multiplying the iodine numbers by the factor  $\frac{16}{253} (O = 16)$ , differs widely from the actual figures, from which it is evident that the oxidation of the oils does not consist in the addition of one atom of oxygen to each pair of doubly-linked carbon atoms. Moreover, oleic acid does not suffer any oxidation at all, and finally, after the maximum of the oxidation of an oil has been attained, further exposure results in a loss of weight caused, as Mulder showed, by the formation of volatile oxidation products, such as carbonic, formic, and acetic acid.—C. O. W.

*Detection of Sesame Oil in Olive Oil.* F. Musset.  
Pharm. Central H., N.F. 1893, 14, 701.

See under XXIII., page 423.

*Analysis of Mixtures of Sperm Oil and Mineral Oil.*  
C. A. Lobry de Bruyn. Chem. Zeit. 17, 1453.

See under XXIII., page 426.

*Note on Stearin Pitch.* E. Donath and R. Strasser.  
Chem. Zeit. 17, 17.8.

See under III., page 380.

PATENTS.

*Improvements in the Manufacture of Soap Tablets and Apparatus for that Purpose.* R. H. F. Finlay, Holywood, County Down. Eng. Pat. 4581, March 2, 1893.

The soap is run direct from the pan into a moulding machine, resembling that used for moulding candles, which may be mounted on transoms, so that it may be inverted if desired. The moulds are of circular or elliptical section. When the soap has set, it is exuded from the moulds by pistons worked by a rack and pinion, then cut up in the usual way, and pressed into tablets of any desired form. The soap may be variegated in colour by inserting a core into the mould before filling with soap, then withdrawing the core after the soap is set, and filling the cavity with soap of any other colour. Or tubes, closed at one end with a valve, containing melted colored soap, may be inserted into the moulds while the soap is still fluid, the valve opened, and the tube gradually withdrawn, when the different colored soaps commingle with each other.—J. J. K.

*Plates for Oil- and other Presses.* W. C. Leechman, Colombo, Ceylon. Eng. Pat. 7039, April 6, 1894.

See under I., page 377.

*Improvements in the treatment of Linseed Oil.* J. E. and C. S. Bedford, Leeds. Eng. Pat. 7742, April 17, 1893.

The claim is for a method of, and apparatus for, manufacturing a thickened or solid material from linseed oil (either raw or boiled) by exposing it in a finely disintegrated condition, at a high temperature, to the action of air. The oil is treated in a horizontal cylindrical steam-jacketed vessel (provided with a shaft, fitted with arms or beaters) which is only half filled, and heated by means of the steam jacket, assisted by agitation, sufficiently rapid to cause a spray. Air is forced into the space above the oil, when the finely-divided sprays of oil absorb oxygen. The temperature gradually rises till 120–160° C. is attained. Samples are taken from time to time, and when the desired consistency is obtained, the mass is run into coolers.—J. J. K.

*A New or Improved Composition of Soap.* Helen Hamilton, Cornacassa, Monaghan. Eng. Pat. 8621, April 29, 1893.

The following ingredients are blended together in approximate proportions, viz.:—

	Lb.
Best white ordinary soap .....	1
Whitening .....	1½
Washing soda .....	1
And about 1 pint of pure cold water.	

—J. J. K.

*An Improved Manufacture of Oils, Fats, Wax, and other analogous substances freed from odour or flavour.* C. D. Abel, London. From *Fabriques de Produits Chimiques de Thann et de Mulhouse*, at Thann, Alsace. Eng. Pat. 9105, May 6, 1893.

The main feature of this invention is to render oils, fats, &c. absolutely odourless and flavourless, and this is done by the exclusion of air from the closed vessels in which the substances are treated. They are heated in closed vessels and the air exhausted by means of an air-pump, or an inert gas, such as carbonic acid, nitrogen, or hydrogen, is introduced. After all the air has been removed, the temperature is raised to from 110° C. to 220° C., and saturated or superheated steam is then blown in until the condensed water is free from odour. In the case of fats, such as oleomargarin, which are easily saponified by steam, an inert gas is used instead of steam to remove the odorous products. The purification may be accelerated in some instances by first emulsifying the substance with 2 to 10 per cent. of sodium bisulphite of 40° B., and afterwards washing the fat with water, before treatment as above stated.—J. J. K.

*Improvements in Soap for removing Hair from the Skin.* J. Mellinger, Baltimore, Maryland, U.S.A. Eng. Pat. 18,658, October 5, 1893. United States Pat. dated March 16, 1893.

This invention relates to the intimate admixture with soap of sodium sulphide, which acts as the hair removing agent, starch as a binding agent, and glycerin, as an emollient. The proportions of the ingredients are—

Glycerin .....	1 lb.
Tallow .....	2 "
Coconut oil .....	2 "
" Oil of ricini .....	4 "
Soda lye (35 per cent.) .....	1 "
Starch .....	1 "
Sodium sulphide .....	2 "
Oil of citronella .....	1 "
Water .....	3 pints.

—J. J. K.

*An Improved construction of Hydraulic Oil Press.* Greenwood and Bailey, Limited, and H. Lambert, Leeds. Eng. Pat. 19,864, October 21, 1893.

The features of this invention are an adjustable measurer for feeding the meal, in order that the thickness of the cakes

may be regulated, and an arrangement to ensure an even pressure throughout the pile of cakes, whereby they are more equally consolidated, and a larger yield of oil is obtained.—J. J. K.

## XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

### (A).—PIGMENTS, PAINTS.

*Ultramarine.* Jordan. *Zeits. ang. Chem.* 1893, 684–690.

The possibility of artificially producing ultramarine dates from the time when Vauquelin discovered that the blue product found in soda furnaces had the same composition as ultramarine. Gmelin was the first to publish a method for preparing ultramarine, but the first factory was started by Guimet in Tonlouse in 1828. The author gives an historical account of the development of the industry, and then proceeds to describe the modern methods of preparation.

The requisite sodium sulphide may be obtained in three ways, viz., by—

- (a.) The sulphate process.
- (b.) The soda process.
- (c.) A combined sulphate and soda process.

The raw materials which are employed in the manufacture are:—

1. *Sulphate* or calcined Glauber's salt. This should not contain more than 3 per cent. of sodium chloride, and should be as finely divided and uniform in appearance as possible. Under certain circumstances the acid sodium sulphate is used in the manufacture of ultramarine green.

2. *Soda*.—The soda should be as free as possible from chlorine, but the strength may vary from 80–99 per cent. Ammonia-soda furnishes a product with a redder shade than Leblanc-soda. The Glauber's salt may be simply ground up with the soda in the combined process, but ultramarine of a purer blue tint is obtained if the solutions of the two are previously evaporated down together.

3. *Clay*.—The suitability of any given clay for the manufacture of ultramarine depends on what sort of product is desired. If purity of tint be no object, the percentage of iron may be considerable, but in all cases, clays with a high percentage of lime should be avoided. Clay is always used in a more or less finely divided state, just as in the manufacture of porcelain, and they may either be air-dried, or anhydrous, according to the kind of ultramarine it is intended to manufacture.

4. *Silica*.—The percentage of silica in clay is often too low, especially for the preparation of blue ultramarine with a reddish tint. In such cases finely ground quartz, sand, or kieselguhr must be added. Recently, the ignited residue obtained in the manufacture of aluminium sulphate, has been used for this purpose, but on adding it to the clay, allowance must be made for the alumina which is already contained in this substance.

5. *Sulphur*.—Sicilian, German, or English regenerated sulphur may be used, but it should be as pure as possible and free from arsenic.

Powdered coal, coal tar, or the resin obtained in the preparation of oil of turpentine may be used as the *reducing agent*. The first two are chiefly employed in the sulphate, the last in the soda process.

All the raw materials should be well ground, and it is also preferable to grind up the clay with the soda before adding the other substances. The materials may be mixed in many different proportions, and each corresponds to a slight variation in the colour of the ultramarine obtained.

Generally speaking, the stability of the ultramarine, with regard to alum solution, increases by increasing the proportion of silica. The depth of the colour increases by adding more sulphur and the reddish tint becomes more

apparent by increasing the proportion of silica. A large percentage of water in the clay gives a lighter ultramarine of a pure blue. In the combined sulphate and soda process, the character of the product may also be largely varied by altering the relative proportions of sulphate and soda. In the soda process a good blue may be obtained by adding to 100 parts of clay (with silica, if necessary) at least 70 parts of 95 per cent. soda, 80 parts of sulphur, and 14 parts of resin. For a pure sulphate ultramarine, 100 parts of clay, 105-130 Glauber's salt, and 25-30 parts of coal or tar may be employed. For the combined sulphate and soda process, equal parts of the above sulphate mixture and the following soda mixture are used. Clay 100, soda 100, sulphur 60, coal or tar 10-12 parts. A darker ultramarine is obtained by adding more of the last mixture.

The burning of the various mixtures is conducted in crucible furnaces, muffle furnaces, or in retorts, and the time required varies from 30 hours to 3-4 days. If insufficient oxygen is admitted to the crucibles, &c. the mass is green. Sulphurous acid, hydrochloric acid, &c. change the green colour to the usual blue at a high temperature. The blue mass is repeatedly ground (in the wet state), washed with water, and allowed to settle. Occasionally the finest particles of ultramarine settle with great difficulty, but the addition of a small quantity of salts of the alkalis or alkaline earths facilitates the process greatly. After drying by steam the ultramarine is passed through sieves of the required fineness.

Ultramarine violet may be prepared by a variety of processes. If a mixture of chlorine and steam is passed over ultramarine blue or green, heated to 160°-180° C., the violet colour is produced along with sodium chloride. Hydrochloric acid gas and air act in the same way when led over ultramarine heated to 150°-230°.

Ultramarine red is obtained when the vapours of nitric acid are led over ultramarine violet at 130°-150°, or the vapours of hydrochloric acid at 128°-132°.

Hydrogen converts ultramarine violet at 280°-290° into a pure bright blue substance containing hydrogen.

Ultramarine varies in composition so that it is not possible to represent it by a formula. The sodium may be replaced by silver, potassium, and lithium, and the sulphur by selenium and tellurium.

The following tables give the composition of some ultramarine compounds:—

	Ultra- marine White, Hoffmann.	Ultra- marine Green, Philipp.	Ultra- marine Blue (poor in Silica), Philipp.	Ultra- marine Blue (rich in Silica), Philipp.
Na.....	21.5	17.02	15.66	14.66
Al.....	16.6	15.81	15.39	12.55
Si.....	17.0	17.51	16.87	17.29
S.....	6.1	7.91	5.69	11.38
O.....	38.1	..	..	..

	Ultra- marine Violet, Wunder.	Ultra- marine Red, Wunder.	Light Blue, Wunder.	Silver Ultra- marine, Henmann.
Ag.....	..	..	..	47.97
Na.....	11.7	8.1	11.9	1.07
Al.....	13.1	13.3	13.1	9.1
Si.....	19.4	19.3	13.7	10.09
S.....	13.3	15.2	12.7	1.75
O.....	42.1	43.4	42.0	} 9.61
H.....	0.38	0.72	0.62	
Clay residue.....	..	..	..	0.81

(See this Journal, 1890, 1137-1138.)—J. S.

## PATENTS.

*Improvements in Apparatus for the Manufacture of Metallic Pigments or Compounds, and in the Production of Lead Pigments or Compounds by Means of such Apparatus.* A. C. J. Charlier, Glasgow. Eng. Pat. 7117, April 6, 1893.

This patent refers to an improvement in the apparatus described by Hannay (Eng. Pat. 1282, 1889; this Journal 1889, 553) for the preparation of lead sulphate by the volatilisation method; and the improvement consists in the insertion of a damper between the producer furnace and the oxidising chamber, so as to prevent the pigment from being discoloured by the smoke given off from the freshly charged fuel, and in the employment of jets of steam in the furnace and chamber, which are stated also to prevent discolouration, "and the formation of slag in the oxidising chamber."—F. H. L.

*A New or Improved Paint.* W. Ward, South Shields, and J. O. Wallace, North Shields. Eng. Pat. 10,619, May 30, 1893.

This paint is claimed to be impervious to and not softened by petroleum oil, so that it may be used to protect any tanks or vessels used for storing this substance, being applied either to the fresh service, or on the top of any other kind of paint. It consists of 150 parts of wood naphtha, 50 of caoutchouc, 30 of shellac or lauri, 75 of coal-tar naphtha, 15 of "pine varnish," 20 of Stockholm tar pitch, 10 of boiled linseed oil, 30 of ground white lead, 20 of dry zinc white, and 150 of a suitable pigment, "pale celestial blue" being preferred. The mixture is made in a steam jacketed pan fitted with a stirrer, and the resulting paint is thinned with wood naphtha to a suitable consistency.

—F. H. L.

## (B.)—RESINS, VARNISHES.

### PATENTS.

*Improvements in the Composition and Treatment of Substances for Use as Glue, Putty, and Cement.* W. M. Walters, Liverpool. Eng. Pat. 3886, February 22, 1893.

One hundred parts of "resin" are melted with 2-20 parts of linseed oil, and 200-40 of a mixture of such substances as chalk, lime, plaster of Paris, fuller's earth, zinc oxide, &c.—F. H. L.

*An Improved Polishing Medium.* P. Theil and K. Schimnitz, Köpenick, Germany. Eng. Pat. 8666, April 29, 1893.

One hundred and twenty five grms. each of guaiacum and benzoin resins, 30 of shellac (preferably the "ruby" brand), 150 of linseed oil, and 30 of "benzine" dissolved in 3 litres of 94 per cent. spirits of wine, form a polish which may be employed by inexperienced persons and when applied is most durable, not being liable to exudation, when used for covering coats of oil-paint of long standing it will produce no cracks and will always prove durable and of satisfactory appearance."—F. H. L.

## (C.)—INDIA-RUBBER.

*Regenerated India rubber.* Rob. Henriques. Chem. Zeit. 17, 126°.

THE recovery of the india-rubber from golo-he- and similar articles forms an extensive branch of industry in the United States. The process by which this recovery is effected consists in the mechanical separation or destruction by means of dilute sulphuric acid, of the textile materials and the subsequent treatment of the finely ground product with steam under a pressure of six atmospheres. The steaming process seems to effect a devulcanisation by oxidation. This assumption is borne out by the fact of the regenerated india-rubber possessing, unlike vulcanised

rubber, to some extent the plastic properties of natural caoutchouc, and also by the results of an analysis, which showed the presence of a large quantity of sulphates and but little sulphur in combination with organic matter in the recovered product.

Various qualities of this regenerated or "reclaimed" rubber are met with in commerce. The price of these products, which at best represent but mediocre mixtures approaching closely in composition to the waste material from which they have been obtained is high (1s. to 1s. 6d. per kilo.), considering the quantity of real caoutchouc contained in them does not exceed 35 or 40 per cent. Their industrial value therefore appears doubtful. Three samples in the shape of black sheets, examined by the author, had a specific gravity of from 1.59 to 1.66, and contained lead oxide, calcium sulphate and carbonate varying in quantity from 12.23 to 14.02 per cent., 21.43 to 22.13 per cent., and 10.91 to 18.00 per cent. respectively, together with small quantities of ferric oxide, alumina, silica, and water. The sulphur in combination with caoutchouc amounted to from 0.71 per cent. to 2.03 per cent. Asphaltum and lamp-black, two invariable constituents of the goloshes mixture were also found to be present. Fatty oils and india-rubber substitutes could not be detected.—F. M.

*The Analysis of India-rubber Goods.* D. Holde. Chem. Zeit. 17, 1634-1635 and 1671-1671.

See under XXIII., page 425.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

*Estimation of Glue.* Papier Zeit. 19, 126.

See under XXIII., page 424.

*Examination of Glue for the Manufacture of Coloured Papers.* Th. Schlossmann. Papier Zeit. 18, 2484-2485.

See under XXIII., page 424.

*Analysis of Tanning Materials.* A Fölsing. Chem. Zeit. 17, 1770.

See under XXIII., page 426.

## PATENTS.

*Improvements in the Manufacture of Liquid Glue.* G. E. Wiese, Hamburg, Germany. Eng. Pat. 2079, January 31, 1894.

In 100 parts of water dissolve successively:—

	Parts.
Chloral hydrate.....	2
Ammonium sulphocyanide.....	5
Boric acid.....	3
Zinc chloride.....	0.5
Zinc sulphate.....	1
Glue.....	450

Stir frequently whilst mixing to prevent setting. "The chloral hydrate and ammonium sulphocyanide produce the necessary degree of fluidity, whilst the desired tenacity and elasticity are produced by the boric acid and the zinc salts."

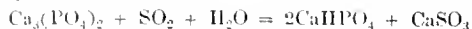
—A. G. B.

*Process for the Production of Glue and Gelatin from Bones.* J. Grillo, Neumühl-Hamborn, Germany, and M. Schroeder, Düsseldorf, Germany. Eng. Pat. 2175, February 1, 1894.

WHEN air-dried or slightly-moistened bones are exposed to a current of sulphur dioxide at the ordinary temperature, they will absorb, with the exception of those of the very

hard, tubular kind, from 10 to 12 per cent. of their weight of the gas in the course of 12 hours. If the exposure be prolonged the absorption may increase to 15 or even 20 per cent. of the weight of the bones; but this excess will be lost when the bones are exposed to the air.

An absorption of 11 to 12 per cent. calculated on the weight of the bones amounts to 16 to 17 per cent. calculated on the inorganic constituents thereof, and corresponds with the equation—



The bones, treated in this way, are easily disintegrated by water at 100° C.; the sulphite and phosphate remain insoluble whilst the gelatin passes into solution.

In practice, the bones are placed in lead-lined wooden chests or in iron cylinders and treated systematically with gases carrying sulphur dioxide. Thus, if the installation consist of eight chests, four of these may always be in use for the absorption of the gas, three in a state of rest after the gas has been absorbed to the extent of 10 to 12 per cent., to allow of chemical combination of such gas as has been "fixed by condensation," and one in course of being charged or discharged.

"After shutting out apparatus 1, apparatus 5, with fresh bone material, is connected again. The sulphur dioxide gases then pass through the apparatus 2, 3, 4, 5 in succession, whilst 7, 8, 1 are in a state of rest. Apparatus 6, which has been standing longest, is then emptied, charged with fresh bones, and, in place of apparatus 2 to be shut out, connected in the set."

When compressed liquid sulphur dioxide is available it is better to treat the bones in separate iron cylinders under pressure; 10 to 12 cwt. of the liquid will be required for every 100 cwts. of bone.

The decomposed bones are transferred to lead-lined vats, covered with water, and heated by closed or open steam; after two or three hours the liquid is changed, to avoid loss of glue substance by prolonged heating. The second liquor is mixed with the first, the third is used for extracting fresh, decomposed bones. If the bones have not been subjected to extraction with solvents whilst raw, which is the better plan, the bone-fat will here be recovered by skimming. The glue liquor will be acid in reaction and will contain a small quantity of calcium sulphite and monocalcium phosphate in solution. These must be removed in order to obtain a transparent finished product; to this end the liquor is neutralised with milk of lime, whereby the salts are precipitated and may be filtered off. The evaporation of the glue solution is conducted as usual. From 16 to 24 per cent. of glue should be obtained.

The bone residue consists of a mixture of  $\text{CaHPO}_4$ ,  $\text{CaSO}_4$  and some inner hard pieces of unattacked bone (about 10 per cent. of the bone-). By grinding under gentle pressure and sifting, this unattacked bone may be separated and returned to the sulphur dioxide vats. The finer portion of the residue is treated in closed cylinders with sulphuric acid in order to render the phosphate completely soluble and to recover the sulphur dioxide.—A. G. B.

*An Improved Process for Continuously Treating Hides in Tanning Liquors, and Apparatus therefor.* H. W. Brüning, Neumünster, Prussia. Eng. Pat. 8026, April 20, 1893.

This process is characterised by finishing the tanning process in the same receptacle in which the hides were originally suspended, the liquor flowing continuously through the receptacle (and in each vessel from below upwards) in such a direction that the strongest liquor acts on the hides which are in the last treating period of the tanning process. The object is attained by means of channels arranged in the masonry walls of the receptacles in a manner set forth in the drawings which accompany the specification.—A. G. B.

## XV.—MANURES, Etc.

*The Action of Lime, of Salts, and some Acids in Rendering Clay Flocculent.* Rob. Saehse and Arthur Becker. Landw. Versuchsst. 1893, 43, 15.

PURE china clay suspended in water is, by the addition of a few drops of lime-water, immediately converted into the flocculent modification and precipitated. The lime does not enter into combination with the clay. Two samples of soil, treated in the same manner, behaved differently from each other, inasmuch as in one case the coagulation took place instantaneously and in the other very slowly. The action of the lime might furnish a means of altering the structural condition of a soil.

Acids have the same coagulating effect. Carbonic acid, which quickly causes the fine particles of china-clay to settle, might also to some extent be the cause of the improvement of a heavy soil by humoid substances. Hydrochloric, sulphuric, and nitric acids have a rapid action upon china-clay. Hydrochloric acid, in fact, seems to surpass all other substances in this respect. Salts are less effective generally, but sodium, potassium, and ammonium chlorides were found to flocculate china-clay and one of the two samples of soil quickly, whereas the other remained unaffected.

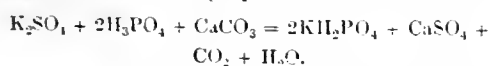
Silica in the form of infusorial earth and finely-ground quartz showed a behaviour similar to that of clay.—F. M.

*Comparative Experiments on the Fertilising Effect of various Phosphates.* O. Kelloer, Kozai, Mori, and Nagaoka. Landw. Versuchsst. 1893, 43, 1.

WHILE all phosphates (applied to non-irrigated soil) showed a distinct influence upon the three crops following the first one, the degree of this influence differed with the different phosphates. Thus superphosphate, precipitated calcium phosphate, phosphatic slag, and ground steamed bones showed a great similarity in their action. They were taken up chiefly by the first crop and their effect upon the succeeding crops became gradually less, the initial ratio of the respective manurial values remaining almost unaltered. This observation finds its explanation in the fact of the readily assimilable mono- and di-calcium phosphates suffering chemical changes in the soil, by which their solubility becomes reduced. The steamed bones and the precipitated calcium phosphate were almost identical in their behaviour, the organic substance of the former undergoing rapid decomposition in the soil, through which the effect of the phosphoric acid becomes accelerated. Raw bones, on the other hand, had less influence upon the first crop, but proved far more effective and lasting as regards the succeeding crops. In this case, again, bones not freed from fat were found superior to extracted bones. The fatty acids set free by the dissociation of the glycerides seem, therefore, to play a definite part in rendering the phosphate soluble.—F. M.

*Potassium Phosphate as Manure.* Theodor Meyer. Chem. Zeit. 17, 1267.

THE attempts hitherto made to produce potassium phosphate cheap enough to render its use as a manure possible have met with little success. A process has been worked out by the author, which is founded upon the reaction expressed by the following equation:—



In actual working this reaction does not proceed smoothly, for by the direct action of the phosphoric acid upon the calcium carbonate calcium phosphate is produced, which means a loss. The necessary operations of filtering, washing, and evaporating would also increase the cost of production. By replacing the calcium carbonate, necessary for the carrying out of the process, by so-called *craye grise* or

chalk-phosphate, not only the manufacture of cheap potassium phosphate becomes possible but also the solution of the problem of advantageously utilising the extensive deposits of this mineral, occurring near Mons, is arrived at in a satisfactory manner. It is carried out as follows:—A measured volume of phosphoric acid of known strength (about 10 per cent) is mixed with the required quantity of potassium sulphate and then the calculated amount of the finely-ground chalk-phosphate (*craye grise*) is gradually introduced. After the lapse of an hour or so the phosphate solution is filtered off and evaporated to a pasty consistency. Desiccation is completed in chambers heated to 70–80 °C. The residue left in the filter-press is without previous washing treated with sulphuric acid in order to decompose the calcium phosphate contained in it. The washings from the calcium sulphate, which forms the only refuse, are utilised in diluting the sulphuric acid for the next operation. The finished product contains from 38 to 40 per cent. of phosphoric acid ( $\text{P}_2\text{O}_5$ ) and 31 to 33 per cent. of potassium oxide, besides a few per cent. of sulphuric acid. Its value as a manure is obvious.—F. M.

*Valuation of Ground Phosphatic Slags.* E. Wrampelmeier. Landw. Versuchsst. 1893, 43, 183.

See under XXIII., page 422.

*Decomposition of Liquids by contact with Powdered Silica.* G. Gore. Phil. Soc. of Birmingham, 1893.

See under Analyt. and Scient. Notes, page 428.

## PATENTS.

*Improvements in the Manufacture of Soda and Monocalcic Phosphate.* F. Hurter, Widnes, Lancaster, and J. Omholt, Christiania, Norway. Eng. Pat. 7107, April 6, 1893.

See under VII., page 391.

*Improvements in the Manufacture or Production of Monocalcic Phosphate and Ammonia.* F. Hurter, Widnes, Lancaster, and J. Omholt, Christiania, Norway. Eng. Pat. 7108, April 6, 1893.

Dry ammonium sulphate is mixed with ground tricalcium phosphate, preferably in the form of a high grade mineral phosphate, in proportion of about 0.85 parts ammonium sulphate to each 1 per cent. of phosphate in the mineral, though it is preferred to use the phosphate in excess. The mixture is heated in a current of superheated steam in a horizontal east-iron cylinder, exposed to direct fire to a temperature of from 550° to 650° F., and the ammonia evolved is led to a condensing apparatus, in which the steam condenses with some ammonium sulphate. The ammonia gas may be utilised for any purpose, such as for the manufacture of soda by the ammonia process. The contents of the cylinder are cooled to about 220° F. by passing in low pressure steam, and are then withdrawn to be ground up as a manure. It is preferred to add 10 to 15 per cent. of sulphuric acid, to react upon any unaltered tricalcium phosphate. If the mass is damp, it may be dried at a temperature not exceeding 212° F.—E. S.

*Improved Manure or Fertiliser.* W. Brothers, Rawtenstall. Eng. Pat. 7537, April 14, 1893.

THE inventor claims the use of solid carbonates of ammonia in a crystalline or powdered form, being superior to either sulphate of ammonia or nitrate of soda, as it is less soluble, more easily decomposed as required by the plant, and the whole weight of the carbonate is utilised. For horticultural purposes it may be used in solution.—J. J. K.

## XVI.—SUGAR, STARCH, GUM, Etc.

*The Decomposition of Invert Sugar by Alkalis and the Practical Application thereof.* Priusen-Geerligs. Chem. Zeit. Rep. 1893, 299—300.

DURING the manufacture of raw sugar from the sugar cane, the presence, or formation, of invert sugar and its decomposition products is an objectionable factor, leading, as it always does, to acidity and fermentation. In order to avoid these difficulties, it is necessary to destroy the invert sugar and allied bodies present in the juice at the very outset. For this purpose the freshly-expressed juice is treated with a slight excess of lime, the excess removed by carbonic, phosphoric, or sulphurous acid, the liquid again treated at a moderate temperature with a small quantity of lime, and, finally, a little barium chloride added. Experiments on a large scale made on a sugar plantation in Java showed that by this means the amount of invert sugar in the juice could be reduced to a minimum, and its subsequent formation (from cane sugar) almost entirely arrested, providing reasonable precautions were observed as regards cleanliness of plant and risk of infection.—H. T. P.

*Note on the Influence of the Percentage of Fat in Sugar-Beet Seed, and the Size of the Latter, on the Richness of the Resulting Beets in Sugar.* P. Doerstling. D. Zucker J. 1893, 1566.

THE author cannot confirm Laskowsky's theory, according to which (1) the richness of the beet in sugar is dependent on a corresponding richness of the seed in fat, and (2) the larger species of seed yield (larger) beets poorer in sugar. Twenty-two varieties of seed and the tubers grown therefrom were examined. From the results it appears that there is no connection whatever between the weight of the beets and the percentage of sugar contained in them, on the one hand, and the size of the seed on the other; whilst the following figures show that variations in the percentage of fat in the seed do not appreciably influence the quality of the resulting beets.

*Seed Rich in Fat* (average of eight samples).—Fat, 16·6 per cent.; sugar in the beet, 14 per cent.; weight of beet, 240 grms.

*Seed Poor in Fat* (average of five samples).—Fat, 12·8 per cent.; sugar in the beet, 14 per cent.; weight of beet, 215 grms.—H. T. P.

*Compounds of Sugars with Iron.* F. Evers. Ber. 27, 474—475.

See under XX., page 415.

## PATENTS.

*Improvements in Apparatus applicable for Evaporating Liquids or Saccharine Solutions.* J. A. Morrell and J. Fromherz, both of New Orleans, U.S.A. Eng. Pat. 3170, February 13, 1893.

See under I., page 377.

*Improvements in or Appertaining to the Utilisation of a Waste or Nearly Waste Product, and the Manufacture of a Useful Gum or Gummy Compound therefrom.* J. Hanson, P. C. D. Castle, and J. H. Morrison, Liverpool. Eng. Pat. 8793, May 2, 1893.

FROM the beans of the locust or carob tree, the inventors extract a gum which they call "tragasol," which may be used to replace tragacanth, size, &c., in weaving, dyeing, and printing cloth. The kernels are split in a bean-splitting machine, the germs and pods carefully removed by aspiration and riddling, and the residue extracted with boiling water until a homogeneous mass is obtained, which is then

put through a centrifugal machine. The liquid may, if necessary, be evaporated to a thick paste, and it will keep for 10 days without the addition of any antiseptic. The specific gravity is about the same as that of water. Care must be taken during the manufacture to prevent the gum coming in contact with either iron, steel, brass, or copper, as it would be discoloured by them.—F. H. L.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*Raw-Grain Conversion in the Brewery.* A. Gordon Sakauon. Trans. Inst. of Brewing, 7 [2], 1893, 31—47.

PRIOR to the year 1880—when duty was levied on the malt, instead of, as at present, on the wort—the use of raw (unmalted) grain was entirely prohibited. Since the restriction has been removed, however, raw grain has come into considerable prominence, chiefly owing to its cheapness as compared with malt. For instance, the average cost of 1 lb. of extract from good barley, prepared rice, and maize, is 5d., 4d., and 3½d. respectively. Apart from cost, another advantage consists in the almost absolute freedom of these malt adjuncts from mould, &c. Malt, as a necessary consequence of the mode of its production, always contains more or less mould, which, if present in excess, exerts a pernicious influence on the resulting beer. In this connection the author has made experiments to determine the minimum percentage of moisture which must be present in rice or maize in order that mould can develop, and finds it to be about 20 per cent. If the moisture be much below this amount no mould-growths appear, even after prolonged incubation of the grain at 100° F. Since the water in rice and maize does not usually exceed 12—14 per cent., it follows that the brewer should rigidly reject all samples containing the least trace of mould. There is one disadvantage attending the use of raw grain, which applies more particularly to maize, and arises from the comparatively large amount of oil existing in that cereal. The oil in maize averages 3—4 per cent., and sometimes attains 5 per cent.; and, like other similar oils, it is most readily decomposed, especially when heated to a temperature exceeding 200° F., into products (free fatty acids) of a most offensive character. This oil, if retained in maize to any extent, would absolutely prevent the latter from being used in large proportions by the brewer. There is little doubt that the "greyness," which at the outset characterised maize beers, was due to the presence of finely-divided oil. The oil of rice, although smaller in amount, is also objectionable. Smetham has described these oils, and the author quotes some of his figures (this Journal, 1893, 848). Fortunately the oil, being chiefly contained in the germ and husk, may be readily removed by mechanical means and reduced to 0·5 per cent. (in the prepared grain). The author suggests that this figure should be adopted by brewers as the maximum permissible amount.

As regards flavour, it is interesting to note that although the starches of maize and rice do not apparently differ chemically from barley-starch, yet the beers produced from them have a distinct character of their own. Rice imparts a dry flavour and the beers clarify readily, whilst maize yields a full, round, smooth flavour, very suitable for "running" beers.

The form of prepared grain mostly used in this country is that known as "flakes." In its manufacture the seed is crushed between suitable rollers and carefully graded until all the broken starchy particles attain as nearly as possible the same size. At the same time, by an exhaust suitably arranged, the liberated germs, husk, and meal, which are much lighter than the starch-particles, are separated. The product thus obtained, known as "grits," is gelatinised by means of steam. It is of importance that this operation be not conducted too quickly, otherwise there is a risk of vitrifying the outside of the grits, and of thus preventing or retarding the satisfactory rupture of the starch cells.

After gelatinisation the grits are flattened out into the familiar form of flakes by being passed between steel rollers, and finally dried on heated floors. It has been suggested that high drying at this stage would be beneficial because tending to the production of dextrin; but this advantage is counterbalanced by the fact that high drying leads to vitrification and consequent loss of extract.

As regards the percentage of raw grain that it is possible to use, general experience shows that 20—25 per cent. is about the limit, and even then great care must be taken to mix the material thoroughly with the malt grist. If appreciably more be employed the excess is simply not dissolved, and leads to bad drainage and other evils. Many attempts have been made to devise methods permitting of the admixture of a largely increased percentage of raw grain with the malt, but so far with little success, at least in this country. The most perfect process and apparatus at present known is that designed by Billings (this Journal, 1892, 628 and 833), and, according to the author, by means of this process 50 per cent. of raw grain (grits) may be incorporated with the malt without the slightest injury attaching to the resulting beer.

In the discussion which followed the reading of the paper, the main points raised related to the Billings apparatus. Going into the question of flavour, the author stated (in agreement with another speaker) that the beers produced

by its means compare favourably as regards flavour with ordinary beers. He further laid stress on the fact that in Billings' process the maize is not boiled as in the older methods, and that consequently no fear need be entertained on the score of the decomposition of the maize oil into objectionable flavouring bodies. As regards extract, the author admitted that the results at the outset were rather poor, owing to gross errors in manipulation, but that at present excellent results were obtained. Concerning the influence of raw grain processes on yeast, the author stated that from worts produced in Billings' apparatus perfectly healthy and vigorous fermentations were obtained, and that the appearance of the yeast under the microscope was all that could be desired.—H. T. P.

*Note on the Composition of Hops; and the Influence of Hops on Wort.* L. Aubry. *Zeits. f. d. ges. Brauw.* 17, 1894 [1], 1—2; [2], 9—10; [3], 17—18.

A RECORD of investigations carried out at the experimental brewing station of Munich. Analyses of wort before and after boiling with hops are given, from which it appears that the composition of the wort, especially as regards the total and albuminoid nitrogen, is practically unaltered by the treatment with hops. The analyses of various samples of hops are given in the following table:—

Source.	Moisture.	Percentages on Dry Substance.							
		Alcoholic Extract.	Benzene Extract.	Water Extract of Sample previously extracted by Alcohol.	Total Nitrogen.	Water-soluble Nitrogen.	Tannin.	Resin.	Water Extract.
1. Posen.....	8.08.	45.50	30.10	14.20	2.11	0.69	5.32	19.46	25.88
2. Saaz .....	8.29	46.21	33.00	16.28	2.35	0.75	5.84	19.15	27.04
3. Spalter Stadt .....	8.48	48.76	33.27	13.79	2.05	0.73	5.31	22.20	26.55
4. Württemberg .....	9.26	49.57	36.19	12.09	1.96	0.64	4.37	26.10	23.47
5. Hops of commerce ....	7.51	43.48	32.64	14.50	2.09	0.63	4.89	21.64	22.49
6. Welnzsch.....	8.17	47.62	34.52	13.63	2.13	0.69	4.89	24.31	23.39
7. Altmark .....	8.72	39.07	26.41	15.79	2.47	0.73	4.40	17.92	21.14
8. English .....	7.63	44.39	27.45	11.15	2.75	0.81	4.43	5.62	25.76
9. Hops of commerce ....	8.35	42.00	30.85	14.52	2.26	0.75	4.02	19.81	22.15

The above hops were also examined after the resins had been extracted by petroleum ether. The results show that only minute traces of tannin and nitrogenous bodies are dissolved during such treatment.

*Ripe and Unripe Hops.*—It is well known that practical brewers ascribe various objectionable effects to the use of unripe hops. Investigation proved that, chemically, there is no difference whatever, either before or after fermentation, between worts prepared under identical conditions with ripe and unripe hops respectively. The matter resolves itself simply into a question of aroma and flavour. Unripe hops yield very little aroma, and impart a harsh flavour to beer.

*Russian Hops* are daily coming into greater prominence, and compete favourably as regards quality with the home-grown article. A sample (1891 crop) yielded the following figures on analysis:—Moisture, 9.38 per cent.; benzene-extract, 31.06 per cent.; alcoholic extract, 49.66; water-extract, 28.80; tannin, 5.33 per cent.; total nitrogen, 2.46 per cent.

The remainder of the paper is devoted to the description of a number of fermentation experiments, in which worts hopped with different brauds of hops were employed. The various results obtained are tabulated. The general conclusion to be drawn from them is, that, other conditions

being equal, the quality of the hops used, exerts no influence on the attenuation and chemical composition of the resulting beer.—H. T. P.

*The Carbohydrates of Yeast.* F. Salkowski. *Ber.* 27, 497—502.

THE liquid obtained by boiling yeast with water, although possessing no reducing action, is shown by the author to contain at least two carbohydrates. One of these—a gum—is precipitated by Fehling's solution in the form of a copper compound; the other remaining in solution yields with iodine the coloration peculiar to glycogen, &c. The former appears to be derived from the contents of the yeast-cells, the latter from the cellulosic tissue. The author has for the present more particularly studied the gum, the method adopted for its isolation being as follows:—Pressed yeast (1 part) is boiled for half an hour with 3 per cent. potash solution (10 parts), allowed to cool and settle, and the clear portion decanted off and heated on the water-bath with Fehling's solution (1½ parts). The bluish tenacious mass which separates in a lump is quickly removed from the liquid, slightly washed with water, ground up with water, and redissolved by the cautious addition of hydrochloric acid. From the resulting turbid solution the gum



is precipitated by adding 3-4 volumes of 90 per cent. alcohol. The precipitate may be purified by re-solution in water and reprecipitation by a large excess of absolute alcohol, and should be finally dehydrated by trituration with absolute alcohol, followed by ether. Thus obtained, the gum forms a white, friable, non-hygroscopic powder, having, when dried at  $110^{\circ}\text{C}$ ., the composition  $\text{C}_{12}\text{H}_{16}\text{O}_{11}$ . It is very soluble in water to a bright pale yellow solution, which when strong is very viscous, and leaves on evaporation a residue greatly resembling gum arabic. The gum is dextro-rotatory— $[\alpha]_D = 90^{\circ}10$  in about 2 per cent. solution.

A weak solution of the gum yields no reaction with neutral or basic lead acetate, ferric chloride, or tannin. Barium hydrate produces at first a turbidity, then a stringy precipitate. Ferric chloride, or basic lead acetate, in conjunction with ammonia, yield a jelly-like mass or precipitation. The reaction with Fehling's solution is so delicate that one part of gum in 5,000 of solution may be precipitated by its means.

On heating with acids the gum is converted into a slightly dextro-rotatory, fermentable, reducing sugar. The yield of gum from pressed yeast appears to be about 2 per cent., equal to 6.9 per cent. calculated on the dry matter.

—H. T. P.

### PATENT.

*Improvements in the Preparation of Malt and in Apparatus therefor.* R. Genge, Hamburg. Eng. Pat. 24,548, December 20, 1893.

THESE improvements relate to the pneumatic system of malting, and are claimed to effect the supply to the grain of cooled air of a constant temperature, and also to maintain a constant degree of humidity within the drum, while allowing for the removal of the carbonic anhydride formed during germination. The air supplied to the drum passes through a tube, around which a system of tubes, containing a cooling medium, is arranged. These tubes also circulate within the drum, and are there claimed to effect the maintenance of a constant degree of humidity by condensing moisture from the air and re-communicating it to the grain. The removal of the gaseous products of germination is attained by perforating the circular wall of the drum and placing round it a series of flaps, which, as the drum rotates, are so arranged as to close the holes in the then upper parts of the drum walls, and to leave open those in the lower parts.

—J. G. W.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY AND WATER PURIFICATION, DISINFECTANTS.

### (A).—CHEMISTRY OF FOODS.

*The Changes Produced in Bread by Mould.* Hebebrand. Landwirthschaftl. Versuchsst. 42, 421-427.

THE changes which bread undergoes during the growth and development of the mould fungus involve principally the consumption of the carbohydrates. Since no evolution of nitrogen in the form of ammonia seemed to occur, it would appear that a proportionate increase of albuminoids must have taken place in the mouldy bread. Nevertheless, it must be observed that a considerable portion of the nitrogen will then no longer exist as albumin, but in the form of decomposition products thereof.—W. S.

*Margarin rendered Conspicuous by Admixture of Phenolphthalein.* F. Hart. Chem. Zeit. 17, 1908-1909.

SOXHLET has recommended that all margarin should be compulsorily impregnated by the manufacturer with phenol-

phthalein; whilst, on the other hand, it has been urged that this admixture would render the substance uneatable in a week. The author finds that this is not the case, addition of from 0.002 to 0.006 part of phenolphthalein having no injurious effect of any kind on 50 parts of margarin, even after keeping six weeks; the samples were perfectly eatable, and gave a red coloration on shaking with soda in a test tube. Moreover, quantities of phenolphthalein varying from a centigram. to 0.2 gram. taken at breakfast produced no evil result of any kind, although somewhat larger doses (0.3 to 0.5 gram.) produced slight diarrhoea. The quantity of phenolphthalein recommended by Soxhlet to be added to margarin (to facilitate detection of adulteration of butter) would only amount to 0.3 gram. in 13.6 lb. (German) of material.—C. R. A. W.

*The Poisonous Action of Martins Yellow, and its Detection.* D. Vilali. Boll. Chim. farmac. 1893, 32, 738.

See under XXIII., page 423.

*Note on some Modifications of the Reichert Meissl Method of Detecting Margarine in Butter, and a New Method for the Determination of the Reichert-Meissl Number.* C. Bunte. Chem. Zeit. 18, 204-206.

See under XXIII., page 427.

*New Method of Estimating Pentosans in Vegetable Substances.* E. Hotter. Chem. Zeit. 17, 1743.

See under XXIII., page 424.

### PATENT.

*A New or Improved Treatment or Preparation of Cream and its Employment in the Manufacture of Butter or for other Purposes.* C. A. Mountfort and J. Prior, Wellington, New Zealand. Eng. Pat. 5527, March 14, 1893.

WHEN butter is made for transport in a cold or frozen state to long distances, salt has to be used, and at the end of the time required for transport it is in a mealey or crumbling condition, and not likely to find a ready sale. The inventors find it preferable to transport cold or frozen cream instead of frozen butter. It is found that the butter made from the frozen cream after its transport is indistinguishable from butter made with the same quality of cream in the fresh state.—V. C.

### (B).—SANITARY CHEMISTRY AND WATER PURIFICATION.

*Treatment of Municipal Refuse in Podewil's Faecal Extract Factory at Augsburg.* Chem. Zeit. 18, 68-69, and 102.

THE possibility of dealing with faecal matters so as to make a profit out of the resulting manure, depends largely on the system of collection; small vessels (tubs) usually receive only a portion of the urine, and little or no household slops, whilst larger receivers (cesspools) often contain the latter to a large extent; hence, whilst the estimated value of the faecal matters from towns where the tub system is adopted is as high as six or eight marks per cubic metre (1,000 kilos.), that in cesspool towns falls to only four or six marks. As regards quantity of excrement per head, the usually received statistics on this point are widely incorrect; the results of the measurements at Augsburg indicate that the individuals from whose excrements the statistics were deduced must have eaten a good deal more than the average population consumes per head in Germany and Austria—a point of much importance in all calculations respecting the treatment of excrementitious matter.

The system adopted at Augsburg consists in mixing the faecal matter with acid and drying for several hours at 120° C. in closed rotating vessels, so that the resulting solid matter issues as a dry powder, whilst the evolved vapours are condensed by a 15-fold supply of river-water to a fluid which has no evil effect on fish, and is not perceptible a few yards lower down stream than the point of inflow. No nuisance of any kind exists; the factory is only two kilometres (1½ miles) from the centre of the town, and is in the vicinity of dwelling-houses, large shops, a school, and other residences.

The same system, with modifications, is also in use for disposing of carcases and abattoir refuse, including blood and similar materials; the closed vessels are made stronger, so that by the aid of high-pressure steam the temperature can be raised to 160° C.; fatty matters are thus melted out and utilised as such, whilst the residue is dried and pulverised whilst still in the same chamber, thus involving a great improvement on other forms of apparatus where treatment with high-pressure steam, drying, and grinding, are three separately conducted operations. It is not practicable to prepare any useful glue from such materials, excepting when clean bones only are used; in all other cases a gelatinous meat broth is obtained, only capable of use in manure making. Podewil's apparatus dries up all such extracts along with the more solid portions of the carcases, producing a readily marketable powdered manure; whilst fat and condensed vapours are the only other products; since all the operations are conducted throughout without exposure to air, any unpleasant smell is practically avoided, and no removal or handling of products at intermediate stages is requisite. In addition to 12 years' experience at Augsburg, the system has also been adopted on the large scale at Hamburg; and smaller arrangements are also in use in Munich and Barmen.—C. R. A. W.

*The Determination of the Reducing Action of Organic Substances contained in Water upon Permanganate of Potassium.* A. Zega. Chem. Zeit. 18, 2.

See under XXIII., page 427.

*Decomposition of Liquids by contact with Powdered Silica.* G. Gore. Phil. Soc. of Birmingham, 1893.

See under Analyt. and Scient. Notes, page 428.

## PATENTS.

*Improvements in or connected with the Purification of Sewage and Analogous Foul or Impure Liquids.* J. J. Meldrum and T. F. Meldrum, both of Manchester. Eng. Pat. 6423, March 27, 1893.

THE inventors claim as chief improvement the purification of sewage by forcing it into and through the air in the form of a fine spray or rain. Before aerating, the sewage may also undergo a preliminary purification by means of iron salts or similarly acting materials; or it may be charged with sulphureous acid.

The projection of the liquid in the form of spray or rain may be effected by applying centrifugal force. The sewage is forced, under suitable pressure, to nozzles or discharge pipes, which are provided with internal twisted blades or screws having one or more deep threads, or are rifled or equivalently formed, by which the liquid is given a circular or gyratory movement as well as a forward motion, causing it, after leaving the nozzle or discharge pipe, to fall apart or be distributed in the form of fine drops or spray.

The inventors describe four other means of attaining the desired spray. Very bad sewages, particularly if not previously chemically treated, must be subjected to aeration more than once.—L. de K.

*A New and Improved Method of Connecting Joints in Sanitary Earthenware and other Articles.* A. and F. G. Johnson, Hanley, Staffordshire. Eng. Pat. 9765, May 16, 1893.

See under I., page 378.

*Improvements in Apparatus for Sterilising Water.* N. Yagin, St. Petersburg, Russia. Eng. Pat. 9808, May 16, 1893.

THE inventor claims as improvement the continuous production of boiled cooled water, by means of a boiler in combination with a cooler, the boiler being composed of an upright closed vessel having passed through it a tube, the lower end of which is connected with a lamp, so that the heat of the latter is conveyed through the flue to the surrounding water. The cooler is constructed of a long upright chamber, containing tubes through which the boiler water is made to flow in one direction, while the water supply to the boiler is made to pass through the chamber surrounding the tubes in the contrary direction. The cooler may be readily taken to pieces. The apparatus is also provided with a regulator in conjunction with a steam coil, the pressure of steam in which controls the action of the regulator upon the discharge of water from the boiler.

As the water cools down under continued pressure, most of the air which has been expelled by the boiling will become reabsorbed, and the water, on storage in vessels, will not assume an unpleasant flavour.—L. de K.

## (C.)—DISINFECTANTS.

### PATENTS.

*An Improved Disinfecting Oven.* O. Baumbach, Copenhagen, Denmark. Eng. Pat. 813, January 13, 1893.

THE claim of the inventor is that the apparatus consists of two steam-tight chambers, one inside the other. The articles to be disinfected are placed on a perforated shelf in the inner chamber and heated by compressed steam circulating in the outer chamber. Condensation water free from dirt may, if desired, be admitted through perforations in the bottom of the inner chamber, and its supply and pressure regulated as required.

The faults of previous inventions are (1) that the articles are heated by direct contact with the steam, and hence are soaked with condensation water, and (2) the rust and oil of the apparatus itself soils the article under process.—L. de K.

*Improvements in or connected with Disinfectants.* P. Molyneux, Bow, and G. R. Potts, South Shields. Eng. Pat. 6953, April 1, 1893.

BLACK sea oil, creosote, caustic soda, resin, and "albo-carbon" (naphthalene) are mixed in various proportions, the resulting solution constituting a liquid disinfectant. The sediment from this solution is mixed with slaked lime, and powdered, forming a solid disinfectant.—J. G. W.

*A New Antiseptic Material.* S. Pitt, Sutton, Sarrey. Eng. Pat. 7038, April 5, 1893.

THIS consists of a 40 per cent. solution of formaldehyde to which the name "formalin" is given. It is claimed to possess a more powerful antiseptic action than corrosive sublimate or carbolic acid, and to have but a small poisonous effect on the human organism. It is stated to have a considerable antiseptic power, even when diluted in the proportion of one part to several hundreds of water. It is preferably prepared by passing air through methyl alcohol, and then over gently heated copper gauze, the surface of which is more or less oxidised. The mixture of air and gaseous formaldehyde is then passed through water, which will dissolve about 40 per cent. of the formaldehyde.

—J. G. W.

*Improvements in the Preparation of Antiseptics.* S. Pitt, Sutton, Surrey. Eng. Pat. 8429, April 26, 1893.

THE commonly used antiseptics, when applied in surgery in the form of dilute aqueous solutions, are stated by the inventor to have the disadvantage of coagulating albuminous solutions, and so being rendered inert as regards disinfecting purposes. To avoid this it is proposed to dissolve these antiseptics—viz., the various phenyl bodies employed and inorganic and organic silver salts—in a solution of either of the following organic bases: ethylenediamine and its alkyl derivatives; the bases obtainable from mono-chlorhydrin, dichlorhydrin, epichlorhydrin and ammonia, and also piperazine. These organic bases are claimed to be neither caustic nor toxic, and to produce a solution of the antiseptic which will not coagulate albumen.—J. G. W.

*Improvements in or connected with the Rolls of Machinery for Mixing and Calendering India-Rubber or other Materials.* A. G. Brookes, London. From J. E. Davis, Lynn, Mass., U.S.A. Eng. Pat. 21,095, November 7, 1893.

See under 1., page 378.

*Apparatus for the Production of Ozone from Phosphorus for Disinfecting Purposes.* S. F. Kattenhoy, Berlin. Eng. Pat. 24,709, December 22, 1893.

WITHIN a glass vessel, provided with a cover pierced with several holes, are suspended two or three small glasses by means of an adjustable rod. These glasses are provided with inlet holes at the bottom, and are arranged to carry pieces of phosphorus and to keep them just above a solution of potassium permanganate in 10 per cent. sulphuric acid. They are each covered by a small cap, which allows of the entry of air through one small opening and of the outlet of the phosphoric acid (?) produced, by a second and somewhat higher opening, and, at the same time, prevents ignition of the phosphorus taking place. The ozone produced during the oxidation of the phosphorus escapes into the air of the room to be disinfected through the perforations in the cover.—J. G. W.

## XIX.—PAPER, PASTEBOARD, Etc.

*Parchment Paper.* By a Practical Man. Papier Zeit. 18, 2717, 2750, and 3117.

IN the manufacture of parchment paper there must be absolute purity of raw material, entire freedom from lime, soda, alumina, chlorine, &c., and the web must be as spongy as blotting paper, without any sizing whatever. Its composition varies with the different strengths required, the thicker the parchment paper the softer, looser, and more permeable the raw material must be, whereas for medium and thinner qualities, it should be somewhat harder and more resistant. For thick paper weighing from 75 to 150 grms. per square metre, it is advisable to use cotton half-stuff, to allow, after bleaching, to remain for some time in the draining-boxes, to submit it to the same treatment as blotting paper, and, if necessary, to give it another bleach. Careful treatment of the finished pulp in the Hollander is also of importance. The thicker the web, the finer the pulp must be, whilst for thinner sorts the fibre should not be too short. In the machine the thicker varieties should be handled, so to speak, without pressure, whereas the thinner varieties must be subjected to slight pressure in order to enable them to withstand the parchementing operation. For certain qualities of thicker paper, in particular for osmosis parchment, as used in the sugar factories, an addition of 30–50 per cent. of linen pulp is necessary.

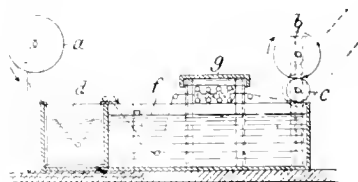
In the thinner sorts, weighing 30–45 grms. per square metre, soda and sulphite wood-pulp have been employed for

many years with good results. Mechanical wood-pulp is totally unfit, but a certain amount of bleached straw-pulp is not objectionable.

In the process of parchementing, two receptacles are required—one, on a higher level, for reducing the concentrated sulphuric acid to 56° B.; the other, on a lower level, for the reception of the cooled acid ready for work. The latter must be thoroughly mixed before using, and the vessel containing it closed with a lid. Temperature is of great importance and should not exceed 18° C., nor should the strength of the acid fall below 56° B. Care must also be taken to use a perfectly dry web in one piece, to reduce the proportion of acid to a minimum, and to avoid folds during the running.

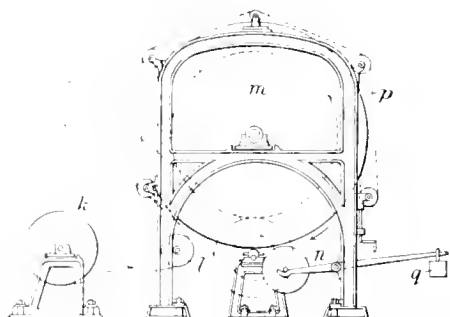
Fig. 1 is a section of the parchementing apparatus. From the roller *a* the web passes into the lead-lined trough *d*

Fig. 1.



containing the acid, and next into the water trough *f*. It is then partly washed on both sides by the jets issuing from the perforated tubes in *g*, and finally, after passing round the roller *c* is wound up on *b*. Thorough washing being indispensable, each machine is provided with two or more systems of jets similar to the one shown in the drawing. After

Fig. 2.



washing, the parchment, unless it be required hard, is passed through a solution of glycerin and soda crystals and again rolled up, when it is ready for drying. The softer the paper is required, the more glycerin must be used. The dyeing of the paper, when requisite, must precede the glycerin treatment.

Owing to the fact that it contracts more than ordinary paper, parchment paper is more difficult to dry. Fig. 2 shows a simple form of machine. From the roller *k* the web runs over roller *l* on to the drying cylinder *m*, and winds itself up again on *n*, which is pressed against the drying cylinder by the lever weighted at *q*. In this, as in the previous operation, great skill and experience are required on the part of the workman directing the machinery.—E. G. P. T.

*Examination of Glue for the Manufacture of Coloured Papers.* T. Schlossmann. Papier Zeit. 18, 2484–2485.

See under XXIII., page 424.

*The Dyeing of Paper in Pulp.* H. Falke. *Farb. Zeit.* **5**, 97—100 and 113—116.

See under VI., page 389.

#### PATENTS.

*A Process for Colouring Paper and Cardboard.* F. R. Wells, Southgate, Middlesex. Eng. Pat. 8167, April 22, 1893.

See under VI., page 391.

*Improvements in the Manufacture of Cellulose.* C. Kellner, Vienna, Austria. Eng. Pat. 21,275, December 16, 1893.

WHEN wood is boiled under high pressure, the resulting cellulose is capable of being well bleached, but is deficient in strength; on the other hand, when boiled under low pressure, the resulting cellulose has considerable tenacity, but cannot easily be bleached. To obviate these disadvantages, the wood is boiled under high pressure in the cellulose apparatus, and the resulting cellulose, which is therefore capable of being easily bleached, is diluted with a considerable amount of water and treated in apparatus, such as a rag engine, opening machine, or separator, in order to detach the fibres from each other, and cause them to become intermingled. Hitherto they have been conducted direct to the paper-making machine, but according to the present invention the fibrous mass is first thickened into a pulp by removing surplus water, and afterwards subjected to the action of a beating apparatus, such as that of a mill-course or of a ball-mill. A mass is thus produced which, when thinned with water, is suitable for treatment in the paper-making machine, and which, besides being tenacious, is also capable of being easily bleached.

—E. G. P. T.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*The Manufacture of Oxygen from Calcium Plumbate.* G. Kassner. *Chem. Zeit.* **17**, 1242.

NOR only the ease with which this compound dissociates on application of heat and takes up oxygen again from the air, constitutes, as accepted by Le Chatelier, its valuable properties for the preparation of that gas and its partial superiority over the barium peroxide used in Brin's process, but the further possibility of obtaining as a by-product pure carbonic acid, and effecting a saving in fuel and reduction in wear and tear of apparatus.

The method of using the plumbate consists in exposing it, in the shape of porous lumps, slightly damped with steam, to the action of washed combustion-gases at a temperature below 100° C. Carbonic acid is readily absorbed, and a mixture of lead peroxide and calcium carbonate results. The oxygen is expelled from the saturated product in a retort maintained at a red heat, preferably with the assistance of superheated steam. With the gradual rise of the temperature and the continued action of the steam, carbon dioxide is also set free, and is collected separately. The mixture of oxygen and carbonic acid produced at the intermediate stage of the process, likewise furnishes pure oxygen when exposed to the absorbing action of calcium plumbate. The regeneration of the plumbate is effected by the action of air blown into the retorts after the expulsion of the carbonic acid.

In Peitz's patented process, pure carbonic acid is used, by the aid of which the oxygen is expelled at a red heat.

—F. M.

*The Chlorination of Ethyl Alcohol.* M. Altshul and Victor Meyer. *Ber.* **26**, 2756—2759.

THE mixture of products obtained in the manufacture of chloral, consisting of chloral alcoholate, chloral hydrate, chloral anhydride, and trichloroacetal, when subsequently decomposed by sulphuric acid and distilled over calcium carbonate leaves an oil behind in the residual calcium chloride which is volatile with steam. This oil, 2–3 kilos, of which were obtained from 1,000 kilos, of crude chloral, boils between 60–168° C., and yields three fractions on rectification. The first, boiling between 60–90° C., consists of chloroform, which most probably results from the decomposition of trichloroacetic acid in the distillation with steam, since it is not contained in the crude chloral. The second fraction, from 90–140° C., has not yet been examined. The third, from 110°–168° C., contains the ethyl ester of dichloroacetic acid, trichloroethyl alcohol, and probably dichloroethyl alcohol also. The chlorinated alcohols, which are only formed in very small quantity, result in all probability from the direct substitution of the ethyl alcohol, whilst the chlorinated acids are to be regarded as being formed by the oxidation of the corresponding aldehydes and will naturally be converted into their esters in the presence of alcohol.—C. A. K.

*Compounds of Sugars with Iron.* F. Evers. *Ber.* **27**, 474—475.

IRON saccharates similar to those described by Schmidt (*Arch. de Pharm.* 1888, 137), but approximately constant in composition, and free from alkali, may be thus obtained. A solution of 120 grms. of cane-sugar in 1 kilo. of ferric chloride (sp. gr. 1.28) and 2 kilos. of water is quickly stirred into a slight excess of a solution of sodium hydrate (7½ per cent.). The mixture is at once largely diluted with water and the precipitate separated, preferably by means of a filter-press, and washed, first with sugar solution (1:1000), then with water. The precipitate, when dried at a low temperature, forms a red-brown crystalline powder, and appears to be a chemical compound of sugar and ferric oxide (48–49 per cent. Fe), for the former is not removed even by boiling water. This iron saccharate dissolves slowly in a hot, moderately strong solution of cane sugar leaving only a small insoluble residue of ferric hydrate. The solution, on evaporation, yields a brown, amorphous, hygroscopic mass of soluble iron saccharate, which retains its solubility in water (and 90 per cent. alcohol) indefinitely. The soluble saccharates thus obtained differs from the preparations containing alkali, in so far that the iron is not precipitated from its solution by sodium acetate. Moreover, no precipitation occurs when the compound is added to a solution containing hydrochloric acid, salt, and peptone; nor is the iron thrown out, even when a great part of the sugar is removed by dialysis. It is important to note that in the above respects, the preparation in question in no way differs from the alkali-free saccharates made according to Athenstaedt's process (*Ger. Pat.* 52,082). The insoluble and soluble compounds of iron with maltose resemble the corresponding preparations of cane-sugar and may be similarly obtained.—H. T. P.

*Hyoscine.* O. Hesse. *Annalen*, **277** [3], 304—306.

RAEHLMANN believes he has discovered in "Scopolamine" an alkaloid, possessing advantages over hyoscine for inducing *mydriasis*. The author strongly maintains, and indeed demonstrates, that scopolamine is nothing but hyoscine. The conclusions he arrives at, the proofs of which he gives in the text, are:—

1. Hyoscine, discovered by Ladenburg, has in the pure form the composition  $C_{17}H_{23}NO_4$ .

2. Hyoscine yields on decomposition at 60–100° C. as basic product, only *osine*  $C_{11}H_{13}NO_2$ .

3. Scopolamine is really hyoscine, and the scopolamine arising by its decomposition is nothing but osine.

4. Scopolamine hydrobromide is no new mydriatic, but only hyoscine hydrobromide, known since the year 1881.

—W. S.

*The Melting Point of Cocaine Hydrochloride.* Cincónine, &c. O. Hesse. *Annalen*, **277** [3], 308—309.

Messrs. Böhringer and Sons have pointed out to the author that cocaine hydrochloride has no melting point, since it first decomposes and then the *residue* melts (this *Journal*, 1894, 172). On heating for 31 minutes at  $160^{\circ}$ — $161^{\circ}$  C., after 15 minutes the salt caked together, and after 25 minutes intumescence set in. After 31 minutes complete fusion had taken place. The author practically confirms this observation, but considers that since at  $185^{\circ}$ — $186^{\circ}$  C. the intumescence merges itself sharply into the fusion throughout the whole mass,—this point and the precise behaviour thereat, taken together, may be safely adopted in testing the quality of a sample of the hydrochloride. In such case, the cocaine salt should be quickly heated to  $175^{\circ}$  C., and the temperature then be slowly raised.

He also points out that cinchonine, generally considered as melting at  $252^{\circ}$ — $253^{\circ}$  C., also decomposes at that point of temperature, and hence, after a time, changes its melting point. Tropine platino-chloride likewise, instead of melting at  $198^{\circ}$ — $201^{\circ}$ , will melt at  $186^{\circ}$ — $187^{\circ}$ , if heated constantly for a few minutes at this latter temperature.

—W. S.

*The Atropine Alkaloids.* O. Hesse. *Annalen*, **277** [3], 290—300.

RESEARCHES by the author on the belladonna root had demonstrated, that it sometimes contains hyoscyamine alone, though very frequently small quantities of atropine also, and occasionally a new alkaloid, atropamine (this *Journal*, 1891, 158). Schütte furthermore discovered very small quantities of hyoscyne (*Arch. de Pharm.* **230**, 709). Merck maintains that atropamine is identical with the apo-atropine previously prepared by Pesci (*Gaz. Chim. Ital.* **11**, 547; **12**, 287), and he has succeeded in converting his apo-atropine into belladonnine just as the author had done when operating on atropamine (Merck's *Jahresber.* 1892, 3). The principal remaining difference between the two bases, is that whilst apo-atropine with ammonia yields an intense violet colour, is crystallisable, and yields tropine by its decomposition, atropamine exhibits no coloration with ammonia, is uncrystallisable, and by decomposition, after previous conversion into belladonnine, yields no tropine. However, Merling (*Ber.* **17**, 381) and Merck have likewise obtained tropine by the decomposition of belladonnine.

The apparent confusion, if not contradiction, thus engendered, led to further researches by the author, and as result he *firstly* confirms Merck's observation that apo-atropine is really identical with "atropamine" (this *Journal*, 1893, 861). *Secondly*, he demonstrates a closer connection between apo-atropine and hyoscyamine which does much to explain the fact of the occasional appearance of the former in the belladonna root. *Thirdly*, that hyoscyamine becomes converted into atropine, apo-atropine, and belladonnine under conditions more or less existing in the respective method of manufacture. Hence, the possibility that in course of manufacture not only apo-atropine is obtained as a by-product, but also belladonnine. *Fourthly*, whereas during the change of hyoscyamine into atropine and apo-atropine, the tropine group remains intact, this group suffers, during the further change into belladonnine, a change of position, which results through the change of properties of the new base produced. The author's result with belladonnine is now only contradictory of that of Merling, whereby tropine is produced by the decomposition of belladonnine. The author can only explain this on the supposition that the bellatropine on long boiling with alcoholic baryta solution is probably reconverted into tropine. Purified belladonnine obtained in the manufacture of atropine, heated to  $140^{\circ}$  C. with eight parts of fuming hydrochloric acid, yielded only traces of tropine, but much bellatropine. Atropine, on heating with hydrochloric acid at  $140^{\circ}$  C., yields tropine but no bellatropine. Thus, then, the conversion of atropine into belladonnine depends upon a conversion of the tropine residue into a bellatropine residue, and consequently

apo-atropine bears the same relation to tropine that belladonnine bears to bellatropine. Kraut, indeed (*Ber.* **26**, 1084), by 16 hours' heating of tropine with fuming hydrochloric acid, seems to have actually obtained bellatropine.

—W. S.

*Pereira Alkaloids.* O. Hesse. *Annalen*, **277**, 300—302.

THE investigation of Pereira hark demonstrated that it contained three alkaloids—Geissospermium,  $C_{19}H_{24}N_2O_2$ ,  $H_2O$ ; Percirine,  $C_{19}H_{24}N_2O$ , and a base which separates from its ethereal solution in granular crystals. The latter is more soluble in ether than geissospermium, and remains therefore in the mother-liquors when geissospermium is crystallised from ether. It is soluble in concentrated nitric acid, producing a purple-red coloration, and in so far resembles both geissospermium and percirine. The "geissospermium" of Freund and Fauvet (this *Journal*, 1893, 618) seems to be identical with this alkaloid. The author, to avoid confusion, now terms Freund and Fauvet's "geissospermium," the "new alkaloid," and commences by observing that it is considerably more soluble in ether than geissospermium. Equally concentrated solutions of both alkaloids in dilute sulphuric acid, agitated with excess of ammonia and equal volumes of ether, yield—

- (a) Geissospermium at once in small needles, terminating in domes;
- (b) "The new alkaloid" only after some minutes, and crystallised either in rhombic plates exhibiting gradually other faces, or else in granular crystals with many faces.

Geissospermium crystallised from alcohol or ether, contains a molecule of water of crystallisation, which it loses at  $100^{\circ}$  C., and agrees with the formula  $C_{19}H_{24}N_2O_2$ .

The "new alkaloid" contains no water of crystallisation, and has the formula  $C_{22}H_{28}N_2O_4$  (Freund and Fauvet). Geissospermium melts at  $160^{\circ}$  C., whilst the new alkaloid melts at  $189^{\circ}$  C.; moreover the former dissolves in concentrated nitric acid with a purple-red colour, becoming paler after a few hours; whilst the latter yields the same colour, which does not become paler. Geissospermium dissolves in sulphuric acid containing ferric oxide in solution, with a beautiful blue colour, whilst the "new alkaloid" forms a colourless solution. Similarly, both alkaloids behave with sulphuric acid containing molybdic acid, but the colourless solution of the "new alkaloid" on heating becomes intensely purple-red. The former with hydrochloric acid, yields an uncrystallisable salt easily soluble in water or hydrochloric acid; the latter gives at once beautiful crystals of hydrochloride. The geissospermium hydrochloride gives in aqueous concentrated solution with potassium sulphocyanide, an amorphous precipitate, whilst the hydrochloride of the "new alkaloid" gives a milky precipitate, consisting of white needles concentrically grouped together. Geissospermium easily dissolves in dilute sulphuric acid, and yields a normal salt, crystallising in delicate white prisms. The "new alkaloid" is but little soluble in such an acid; at the same time the undissolved portion is converted into sulphate, only with difficulty soluble at ordinary temperatures in dilute sulphuric acid. The sulphate forms granular crystals, dissolving in the mother liquor on heating, and recrystallising on cooling.—W. S.

*The Alkaloids of the Bark of the Pomegranate Root.* G. Ciamician and P. Silber. *Ber.* **26**, 2738—2753.

THIS paper contains the continued investigations of the authors on the alkaloid "Pseudo-pelletierine," first isolated by Taurer, from the bark of the pomegranate root. It is proposed to substitute the name "*Granatoline*" for the above, so as to allow of a more simple nomenclature for its derivatives. The base is closely related to tropine, and yields a series of corresponding derivatives, as is shown by the following table:—

Granatoline.....	$C_{11}H_{15}NO$	Tropine.....	$C_8H_{13}NO$
Granatoline.....	$C_{11}H_{15}NO$	Tropamine.....	$C_8H_{13}N$
Granatamine.....	$C_{11}H_{15}N$	Hydrotropine.....	$C_8H_{13}N$
Granatamine.....	$C_{11}H_{15}N$	N-hydrotropine.....	$C_7H_{13}N$
Nor-granatoline...	$C_{10}H_{13}N$		

These bodies result successively by the reduction of granatoline; granatoline yields a hydriodide corresponding to that of tropine. Granatoline is regarded as the homologue of tropine, but the hitherto ascertained facts regarding the former are too incomplete to allow of a constitutional formula being assigned to it. The properties of granatoline are given in previous papers (Ber. 25, 1698, and 26, 156; this Journal, 1892, 705); in the present communication those of the above derivatives are described. The examination of the other alkaloids associated with granatoline especially pelletierine, are now under investigation.

—C. A. K.

*Compounds of the Citral (Geraniol) Group.* F. Tiemann and F. W. Semmler. Ber. 26, 2708—2729.

*Citral*, the aldehyde contained in lemon oil and in oil of lemon-grass, is an almost colourless liquid, slightly soluble in water, readily in alcohol and ether and which distils at 120°—122° C., under a pressure of 23 mm. It is best isolated from the oil by means of its crystalline double compound with acid sodium sulphite. Citral is optically active; on reduction it yields the alcohol geraniol, which is optically inactive. Closely related to geraniol are a number of optically active alcohols having the same formula,  $C_{10}H_{18}O$ , all of which yield citral on oxidation. These include *coriandrol*, the alcohol of coriander oil; *linalool*, from linaloe oil (from *Bursera delpechiana*); and *rhadinol*, the alcohol obtained from rose-oil by Eckart (Ber. 24, 4209; this Journal, 1892, 265). The relationship of these alcohols are discussed. The oxime, hydrazone, anilide, and nitrile derived from citral are described. The last yields geranic acid,  $C_{10}H_{16}O_2$ , upon treatment with alkali, which acid is also formed by the action of moist silver oxide upon citral. When citral is oxidised at 0° C. with chromic acid, only a minute quantity of geranic acid is obtained, methylheptylene ketone,  $C_8H_{14}O$  and an acid, which yields the ketone on distillation, being the essential products. Geraniol oxidised under similar conditions yields the same products. The remainder of the paper deals with a number of cyclic compounds, obtained by the action of sulphuric acid upon geraniol derivatives and which bear upon the constitutional formulae of citral and of geraniol.—C. A. K.

*Determination of Oil of Turpentine in Alcohol.*

J. Perl. Chem. Zeit. 17, 1851.

See under XXIII., page 427.

*The Estimation of Saccharine in Presence of Salicylic Acid.* Hairs. J. de Pharm. d'Angers.

See under XXIII., page 424.

## PATENT.

*A new Medicinal Preparation for the Curative Treatment of Leprosy, Cancer, Eczema, and other allied Diseases.* W. Watson, London. Eng. Pat. 8493, April 27, 1893.

This preparation, for internal use, consists of a decoction of an Indian plant, *Farnaria parinflora*, known in the Punjab as "Papperah," either in the dry or fresh condition.

—J. G. W.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

### PATENTS.

*Improved Silver Haloid Emulsions for Photographic Purposes.* W. H. Prestwich, Tottenham. Eng. Pat. 5517, March 14, 1893.

This invention relates to sensitive silver emulsions for coating glass, films, or paper for photographic purposes, the improved emulsion, in the case of negatives, preventing solarisation and halation; in the case of transparencies imparting a ground-glass effect; and in the case of prints on paper or opal glass, producing a matt surface. These results are obtained by using, in conjunction with gelatin, a proportion of an amylaceous substance such as starch, arrow-root, corn or rice flour, say 3 parts of gelatin to one of starch.—E. G. P. T.

*Improvements in Processes of Making Transparent Photographs on Glass.* C. A. Allison, London. From F. B. Forster and G. G. Rockwood, both of New York, U.S.A. Eng. Pat. 6501, March 28, 1893.

This process consists in printing the positive image in a metallic oxide from the negative upon a medium which has been surfaced with a mixture of the materials constituting the sensitive film and said metallic oxide; then transferring said print to, and developing it upon, a temporary support; then transferring the print and rendering it insoluble upon the surface of a transparent glass plate; then coating the print with a vitreous flux which fuses at a temperature between 600 and 700 F.; and lastly, subjecting the glass plate bearing the print and its flux, to a degree of heat sufficient to fuse the flux.—E. G. P. T.

*An Improved Method of Preparing Pigment Paper for Photographic Printing.* W. White, Paddington. Eng. Pat. 824, January 13, 1894.

By this method the usual operations of the carbon or pigment printing process may be performed in any ordinary apartment and in ordinary daylight. A paper or other flexible material is selected, for supporting the pigment and gelatin compound, which is opaque or suitably coloured, so as to transmit only non-actinic light. The supporting tissue is coated with the pigment compound and dried. The sensitising is performed in the usual way, and when sufficiently flaccid, the paper is taken out and laid on a sheet of zinc, or other smooth and opaque material, the excess of solution being removed by a squeegee; the whole is then placed aside in a current of air to dry.

—E. G. P. T.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*A New Method of Estimating Yellow (Ordinary) Phosphorus.* Julius Töth. Chem. Zeit. 17, 1244.

See under XXIII., page 420.

### PATENT.

*An Improved Igniting Composition for Matches.* J. M. Meissner, Vienna, Austria. Eng. Pat. 6775, March 30, 1893.

This invention essentially consists in employing aluminium as an ingredient of igniting composition for matches. The composition consists of a suitable adhesive substance and potassium chlorate, potassium, barium, or strontium nitrates, and finely pulverised aluminium; with or without the addition of iron filings or granular aluminium.—W. M.

## XXIII.—ANALYTICAL CHEMISTRY.

## APPARATUS, ETC.

1. *New Thermometer for High Temperatures.* E. C. C. Baly and J. C. Chorley. Ber. 27, 470—471.

THE appearance of this instrument is precisely that of an ordinary mercurial thermometer, but instead of mercury it contains the liquid sodium-potassium alloy. The boiling point of the alloy is about  $760^{\circ}\text{C}$ ., whilst its freezing point is  $-8^{\circ}\text{C}$ ., and hence the thermometer charged with it possesses the wide range of  $-8^{\circ}\text{C}$ . to  $650^{\circ}\text{C}$ . For the sake of convenience, however, the graduation only commences at  $200^{\circ}$ , so that the ordinary dimensions of a mercurial thermometer are not materially exceeded. To this end the tube has a bulbous expansion blown upon it, at a distance of about 12 cm. above the bulb forming the alloy reservoir. The glass is of the most infusible quality ("Resistenz Glas"), capable of withstanding a red heat. The space above the alloy is filled with pure nitrogen under a pressure such that when the reservoir is red hot, and thus somewhat soft, the interior pressure will be about equal to the external atmospheric pressure. Thus, alteration in the form of the reservoir is avoided.

The thermometer is not graduated until it has withstood successfully the application of a red heat for 30 hours. The fixed points, for the higher graduation, are established by immersing the instrument in the vapours of boiling sulphur and other substances. Only the reservoir and 9 cm. of the tube are heated, since the coefficient of expansion of the alloy increases with the temperatures. In this way the error of the unprotected thread is compensated. Thus, then, in using the thermometer, not more than 9 cm. of the tube should be exposed to the heat, and special care should be taken that the bulbous expansion, already referred to, is not heated.—W. S.

## Simple Extraction Apparatus. W. Büttner. Zeits. ang. Chem. 1893, 634—635.

THE author describes an extraction apparatus which can be easily made in the laboratory, and which possesses the additional advantage that only one cork is required. The apparatus is represented in the accompanying figure. The outer tube consists of an ordinary rather thick-walled test tube, 150—160 mm. long, and 30—35 mm. wide, contracted slightly about 60—70 mm. from the bottom. In the upper part is placed a somewhat narrower test tube, and into this dips a siphon, as shown in Fig. 1. The siphon is made by

Fig. 1.

Fig. 2.

Fig. 3.



drawing out a piece of glass tubing before the blow-pipe until its bore is about 1.5—2 mm., and then bending it as shown in Fig. 2, or preferably as in Fig. 3, which allows of its easy introduction into the inner test tube.

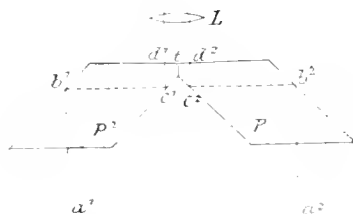
The substance to be extracted is placed in a cartridge of filter-paper, which fits loosely into the inner tube. The extract is obtained in the lower part of the wide tube, in which it may be evaporated to dryness and weighed.—J. S.

## A Colorimeter with Lummer-Brodhun Prism System.

H. Küss. Zeits. f. anorg. Chem. 5, 1893.

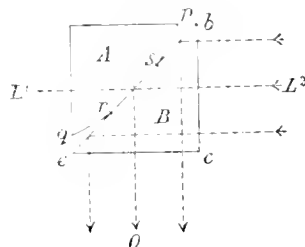
IN colorimetric investigations generally, the object is to determine as accurately as possible the point when two solutions, &c., being compared, are equal in point of colour and brightness. In practice, the solutions contained in cylinders, &c. are viewed simultaneously, side by side, by transmitted light, in the field of a lens or telescope. The delicacy of the method depends largely on the degree of proximity of the two areas to be compared, and is greatest when the boundary between them is a "mathematical" line. The contrivance chiefly used for this purpose (Fig. 1),

Fig. 1.



consisting of two reflecting prisms  $p^1$  and  $p^2$  and an eyepiece  $L$ , suitably placed above the cylinders, has been generally found somewhat unsatisfactory, owing to the fact that the line of demarcation ( $t$ ) between the two halves of the field has an appreciable width, thus impairing the accuracy of the results. This difficulty may be overcome by employing the modified prism system (Fig. 2) devised

Fig. 2.



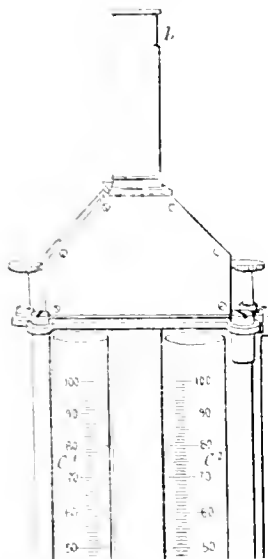
by Lummer and Brodhun for photometric purposes. It consists of two prisms, an ordinary total reflecting prism  $B$ , and a spherically-faced prism  $A$ , of which only the central portion  $rs$  is ground flat and placed in optical contact with  $B$ . An eye placed at  $O$ , receives from the direction  $L^1$  only the direct rays passing through the area of contact  $rs$ ; from the direction  $L^2$  only the rays reflected by the surfaces  $er$  and  $sb$ , and will therefore, in general, perceive a sharply defined, bright or dark, elliptical spot, surrounded by a uniform field. When the direct and reflected rays are of equal intensity, the illumination of the field is uniform all over, and the central spot completely disappears. The general arrangement of the apparatus for colorimetric purposes is indicated in Figs. 3 and 4. Two subsidiary prisms,  $p^1$  and  $p^2$ , are employed, which reflect the light from the cylinders,  $C^1$  and  $C^2$ , containing the liquids to be compared, into the component parts of the main prism,  $A$ ,  $B$ , from which it passes into the eye-piece  $L$ .



Fig. 3.



Fig. 4.



Concerning the delicacy of the new colorimeter, it appears that, for photometric purposes, the Lummer-Brödhun prism system is about three times as sensitive as the ordinary arrangement, in which the central line of the field is not eliminated. —H. T. P.

#### PATENT.

*Improvements in the Method of and Apparatus for Determining the Density of Gases.* G. Frères and M. Meslans, both of Paris, France. Eng. Pat. 70, January 1, 1894.

See under I., page 378.

(Also this Journal, 1894, 64.)

#### INORGANIC CHEMISTRY.—QUALITATIVE.

*Paranitrophenol Sodium as Acid Indicator in Testing Boiler Feed Water for the Presence of Magnesium Chloride.* A. Goldberg. 12. Ber. d. naturw. Gesellsch. z. Chemnitz, 1893, 68.

EXPERIMENTS on the large scale having proved the usefulness of this indicator in checking the efficiency of the water purification process by means of caustic soda, the author made some trials on a small scale with a view of ascertaining the applicability of this method to the preliminary testing

of boiler feed-water. The object of the test is to find out whether the water is likely to acquire an acid reaction when heated in the boiler under pressure.

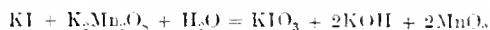
A tube made of wrought-iron, by boring out a solid block, and another of copper closed with a gun-metal screw-cap, were used in the experiments. The iron tube at first proved unreliable, a solution of the indicator in distilled water being decolorised at a temperature of from 150 to 160° C. After repeated use, however (and consequent formation of a protective layer of oxide), solutions of the indicator 1 to 50,000 and 1:100,000 remained intact at 165° C. The presence in the water of  $\frac{1}{100,000}$  and even  $\frac{1}{1,000,000}$  part of magnesium chloride caused complete decolorisation. The results obtained with the copper tube were different. A 1:50,000 or 1:100,000 *p*-nitrophenol-sodium solution remained unaffected after the addition of a small quantity of magnesium chloride (from 0.1 to 1 gm. per litre) and heating to 180° C. The hydrochloric acid which may have been formed by dissociation, appears to have recombined on cooling. No change of the colour was observed after the addition to the water (with or without MgCl<sub>2</sub>) of various neutral soluble reducing agents and of nitrates. The simultaneous presence of some organic substances, such as sugar, glue, &c., however, caused a reduction, which also took place in the pure solution of the indicator after the introduction of metallic zinc, iron, or powdered spathic iron ore.

It may be concluded from these results that the iron-tube method is a reliable means of testing boiler feed-water with the indicator; that the changes the *p*-nitrophenol sodium might suffer in the boiler itself by secondary reactions, are insignificant; and ultimately, that water causing decolorisation of the indicator in the copper tube may be considered to possess a pronounced tendency to become acid. —F. M.

#### INORGANIC CHEMISTRY.— QUANTITATIVE.

*Preparation of Pure Potassium Iodate for Volumetric Analysis.* M. Groger. Zeits. ang. Chem. 1894, 1, 13.

THE salt can be conveniently prepared by dissolving 40 grms. of pure potassium permanganate in hot water, adding 20 grms. of potassium iodide dissolved in a little water, and heating the mixture on a boiling water-bath for 20–30 minutes, then dropping in alcohol until the red colour due to excess of permanganate is discharged, and filtering the product. The reaction which occurs is represented by the equation—



The filtrate, alkaline with caustic potash, is treated with acetic acid until it has a perceptibly acid reaction; it is then concentrated to about 50 cc. and allowed to cool. The mother-liquor is poured off the granular crystals of potassium iodate which have formed, and the salt is then repeatedly washed with strong alcohol and allowed to dry. The acidification of the filtrate from the oxidised liquid is essential, since, unless this precaution be observed, six or seven crystallisations are necessary to free the iodate from caustic potash, which adheres obstinately to it. The product is thereby reduced to 40–50 per cent. of the theoretical amount, whereas when acetic acid has been used 90–95 per cent. of the calculated quantity of potassium iodate is obtained. Free alkali cannot be removed by washing with alcohol, as some potassium carbonate is always formed by contact with the air, and, being insoluble in alcohol, remains with the iodate. Potassium acetate, on the other hand, is readily removed by treatment with alcohol. The potassium iodate prepared in this manner is perfectly pure, has a neutral reaction, and remains colourless when acidified with dilute sulphuric acid and allowed to stand. It can be used for standardising thiosulphate and acids in the manner described by the author (Zeits. ang. Chem. 1890, 285). —B. B.

\* The quantities given in the article quoted are ten times those that should be used, the omission of a decimal point having occurred.

*S. Ium Thiosulphate for Iodometry.* C. Meineke. Chem. Z. it. 19, 33—34.

Pure sodium thiosulphate forms a convenient means of standardising iodine solutions and titrating iodine; but although the salt is easily obtained sufficiently free from saline impurities, it generally contains a slight excess of water, probably enclosed in the crystals as mother-liquor; so that the quantity of actual  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$  present is only 98—99 per cent. It can be entirely dehydrated *in vacuo* over sulphuric acid ( $\text{Na}_2\text{S}_2\text{O}_3 = 99.9—99.95$  per cent.); but the product is extremely hygroscopic, so that it cannot be kept in ordinary stoppered bottles without alteration. Drying the powdered crystals by pressure in blotting-paper does not remove adherent moisture as thoroughly as rubbing in a mortar with strong alcohol, sucking off the liquor, washing with absolute alcohol or ether, and drying in filter-paper; the product thus obtained from a pure salt free from sulphate may be kept for years without alteration; thus a sample containing, in 1888, 99.90—99.91 per cent. of  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ , gave, in 1891, 99.94 per cent.; in 1892, 99.93 per cent.; and in 1893, 99.92—99.94 per cent. Precipitation of a concentrated aqueous solution with alcohol gives crystals enclosing mother-liquor, so that only about 99 per cent. of true pentahydrated salt is present.

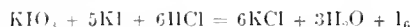
—C. R. A. W.

*The Estimation of Iodine in the Presence of Bromine and Chlorine.* M. Groeger. Zeits. f. ang. Chem. 1894, 52.

ALKALINE iodides, in a neutral or alkaline solution, are completely oxidised into iodates by the action of potassium permanganate, whilst bromides or chlorides suffer no alteration under these conditions. Reinize proposed to utilise this reaction for the estimation of iodine in the presence of bromine and chlorine, by titration with permanganate, according to the equation—



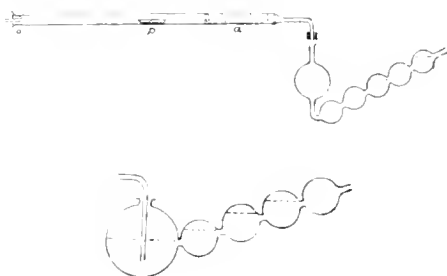
MacCulloch showed this method to be deficient, owing to the precipitate of potassium manganate, formed during the process, and containing less oxygen than that required for manganic peroxide. The author proposes to estimate the amount of iodic acid formed by this oxidation iodometrically. For this purpose, the substance to be analysed is weighed, dissolved, and diluted to a definite volume with water. An aliquot part of this solution, containing at most 50 mgrms. of iodine, is heated on the water-bath in a flask, excess of permanganate solution (1 : 25) added, and this excess, after prolonging the heating for some minutes, at the end of which the strong red coloration of the liquid should have been maintained, is then removed, by the addition of a few drops of alcohol. After the subsidence of the precipitate formed in the liquid, the latter is filtered and the residue on the filter well washed. All the iodine is now contained in the filtrate in the form of potassium iodate. After cooling, 0.5 gram. of potassium iodide is added, and the solution is acidulated with hydrochloric acid. Iodine separates out according to the equation—



and is then estimated by means of 1/10 N-thiosulphate, using starch solution as an indicator. 1 cc. of the thiosulphate represents 2.109 mgrms. of I contained in the titrated solution. The trustworthiness of the method was demonstrated by applying it to solutions which had been beforehand standardised gravimetrically. Presence of ammonia vitiates the results owing to the formation of nitrites during the process of oxidation. In this case it is necessary to expel the ammonia before oxidising by boiling the solutions with a small excess of caustic potash. Too great an excess of caustic alkali should be avoided as preventing the complete reduction of the permanganate. The method may also be employed for the assay of commercial iodine, which frequently contains chlorine, bromine, and cyanogen.—C. O. W.

*Valuation of Fuels.* F. Fischer. Zeits. ang. Chem. 1893, 677—679.

The author points out that it is only in cases where coal or coke is used for reducing or smelting purposes that the estimation of the total sulphur is important. In other cases it is sufficient, and better, to determine the amount of sulphur which is converted into sulphurous or sulphuric acid on combustion. For this purpose he recommends the apparatus shown in the figures.



A weighed quantity of coal or coke (0.5—1.0 gram., according to the amount of sulphur contained in it) is placed in the combustion tube in the platinum boat *p* and ignited in a current of oxygen introduced at *o*. The other end of the tube *a* is packed with asbestos or pumice stone, which is kept at a red heat during the operation. The system of bulbs contains 10—20 cc. of a pure solution of peroxide of hydrogen, which serves to oxidise the sulphurous to sulphuric acid. After the combustion is at an end, the contents of the bulbs are transferred to a beaker and titrated with deci-normal potash solution. The second figure shows another system of bulbs, in which the drawn-out end of the combustion tube dips into the peroxide of hydrogen. By weighing the platinum boat afterwards, the amount of ash contained in the coal may be determined.

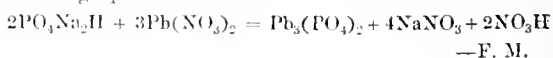
The author also points out that many thermo chemical calculations may be much simplified by making use of the number 22.3 (the volume in litres at standard temperature and pressure occupied by a gramme-molecule of gases and vapours). Reference is also made to one of Niehl's high-temperature thermometers, which enables temperatures to be measured up to 560° C.—J. S.

*A New Method of Estimating Yellow (Ordinary) Phosphorus.* Julius Toth. Chem. Zeit. 17, 1244.

The method is intended for the purpose of ascertaining the quantity of phosphorus in mixtures containing also phosphoric acid, and consists in the extraction with carbon bisulphide, precipitation of the phosphorus in solution by means of silver nitrate, oxidation of the silver phosphide formed with dilute nitric acid, and precipitation of the phosphoric acid in the usual manner.—F. M.

*Volumetric Estimation of Lead in Tin Solutions or Platings.* P. Bayrac. Journ. Pharm. Chim. 1893, 5, Sér. 28, 500.

A STANDARD solution of di-sodium phosphate is employed in this method. Lead phosphate being soluble in mineral acids, sodium acetate is added to the solution to be tested, to counteract this effect. An excess of di-sodium phosphate is added, which is titrated back with a standard solution of lead nitrate, potassium iodide being used as an indicator. The reaction taking place in the process is expressed by the following equation:—



—F. M.

*A Simple Method for Determining Foreign Metals in Commercial Copper.* W. Hampe. Chem. Zeit. 17, 1691.

THE following method will be found much quicker than that previously described by the author. 25 grms. of the copper are placed in a large beaker with 200 cc. of water, 100 cc. of pure concentrated sulphuric acid, and 45–46 cc. nitric acid of sp. gr. 1.210. The solution proceeds rapidly and quietly on heat being applied, and when all the copper is dissolved, about 200 cc. of water are again added, to prevent crystallisation on cooling. The solution is clear except when lead, antimony, or bismuth are present; in such cases the solution must be filtered, and the precipitate separately analysed. The clear solution is heated to 40° C., and a brisk stream of sulphurous acid is passed through it, which quickly reduces the small excess of nitric acid present; this stream should be continued until the solution smells strongly, when, if silver be present, turbidity will arise, owing to a partial precipitation of metallic silver, which may be disregarded when no "wet" silver determination is to be made; or it may be rendered complete by adding a few drops of HCl and setting aside for 24 hours in a 2-litre flask. The mixture of Ag and AgCl is then collected on a small filter, which is treated with hot fuming nitric acid, and the silver is subsequently determined as chloride in the usual way. Such determinations have been found to agree very well with the results of dry methods.

The solution containing suspended particles of metallic silver, or the filtrate from the last operation, as the case may be, is now treated, while cold, with chemically pure sulphocyanide of copper, while a stream of SO<sub>2</sub> is again passed through it. The sulphocyanide must not, however, be in excess; indeed it is better to leave a little copper in solution since it does not interfere with the subsequent operations. A solution of known strength is therefore used, 500 cc. of which will precipitate 25 grms. copper, and this is added by degrees, so as to leave a very little copper in solution. The gas-leading tube is then withdrawn, water is added until the flask is filled up to the mark, and the whole is well mixed and left to rest for a time, when it is filtered through a dry filter into a dry beaker. From the filtrate, a known volume, say 1,800 cm., is taken, the SO<sub>2</sub> is driven off, and sulphuretted hydrogen is passed with the usual precautions so as to precipitate the metals of the arsenic group, which, when present, may be separated and separately determined by the ordinary methods. The same may be said of the following precipitate of nickel, iron, &c., by means of sulphide of ammonium.

In calculating the analysis, the volume of the copper sulphocyanide precipitate must be taken into account. When the copper present amounts to 25 grms. it occupies a space of 15.983 cm., leaving 1984.017 cm. for the true volume of solution in the 2-litre flask. Details are given of the method of calculation and of comparative tests, giving very close results, and the author states that analyses formerly occupying five or six weeks can now be performed in about eight days.—J. H. C.

*The Reliability of the Principal Methods of Determining Total Carbon in Iron.* Göttig. Abhandl. d. Vereins. z. Beförd. d. Gewerbeleisses. 1893, 3, 321.

THE author arrives at the following conclusions:—

I.—The direct estimation of carbon by combustion of the iron in a current of oxygen is, in no case, to be recommended. The method nearly always yields low results, chiefly owing to the difficulty experienced in recognising when the process is completed; and partly because the oxide of iron formed occludes particles of carbon, which thus escape combustion.

II.—The wet combustion process—with chromic and sulphuric acids—may be directly applied (*i.e.*, without previous separation of the carbon) in every instance. There is, however, a possibility of small quantities of hydrocarbons being formed, especially when the sulphuric acid employed is dilute. It is advisable, therefore, to use a good excess of chromic acid (12–15 times the weight of the iron), and to include a tube of glowing copper oxide and a drying apparatus before the absorption bulbs. This is especially necessary in the case of samples containing

much combined carbon. In practice the method is advantageously applicable to graphitoid or manganous irons (Spiegeleisen, ferromanganese). Indeed, for this purpose, the author condemns all dry combustion processes, since the graphite, even when previously separated from iron, is exceedingly difficult to burn. In the case of samples only available in the shape of coarse grains or large pieces, it is well to dissolve out the iron prior to combustion. For this purpose Weyl's electrolytic process is generally suitable, the electrode being surrounded by fine platinum wire-gauze in order to prevent loss of carbon. For the estimation of total carbon in iron free from graphite, the chromic acid method is most convenient when only single determinations are to be made; but when a series of samples are to be analysed, Wohler's chlorine process is preferable. A number of samples (8–10) may be simultaneously treated with chlorine, the residues being afterwards burnt in a stream of oxygen.

III.—In methods involving the solution of the iron in copper sulphate, &c., there is no need to separate the precipitated copper from the carbon, if the latter is to be burnt in the wet way. Otherwise this must be done, for the same reason as under I.

As regards the estimation of graphite in iron and steel, the author makes the following observations:—

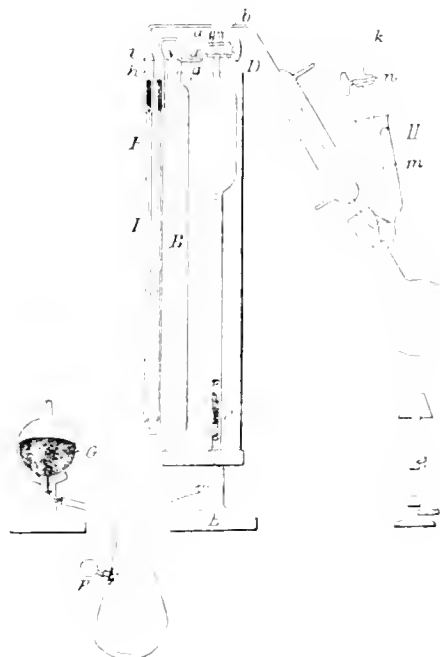
1. Thorough boiling of the material with nitric or hydrochloric acid, renders subsequent washing of the residue with potash, alcohol, and ether unnecessary, and gives better results than when the boiling is omitted and washing resorted to.

2. Compared with hydrochloric acid, the use of nitric acid easily leads to low results.

3. Nitric acid is to be preferred to hydrochloric acid, because by its means a residue free from combined carbon is more readily obtained.—H. T. P.

*Determination of Carbon in Iron and Steel.* W. Hempel. Zeits. ang. Chem. 1894, 1, 22–24.

THE method of determining carbon in iron by solution in chromic acid mixture is imperfect, from the fact that hydrocarbons are evolved. This defect can be removed by the addition of mercury. 0.5 gm. of the iron is treated with 2.3 grms. of mercury and a mixture of chromic acid, sulphuric acid, and water in a partial vacuum, and the atmosphere thus obtained, consisting of air, oxygen, and CO<sub>2</sub>, is drawn over into a gas-analysis apparatus, and the CO<sub>2</sub> determined. The apparatus used is shown in the figure.



A is a measuring tube of a capacity of 150 cc., for the reception of the gas to be analysed; B is a correction tube to save calculations for alterations of temperature and pressure; and C is a manometer connected by one limb with the gas burette and by the other with the correction tube. The burette and correction tube are enclosed in a common water-jacket. A round-bottomed flask at the right of the figure serves for the solution of the iron; it is provided with a condenser and a stoppered funnel. The following reagents are required:—(1.) *Chromic Acid Solution*.—Commercial chromic acid is generally sold in bottles luted with paraffin wax, particles of which may obtain access to the interior of the bottle and cause an error. The acid should therefore be prepared by dissolving 300 grms. of potassium bichromate in 500 cc. of water and 420 cc. of strong sulphuric acid. When solution is complete the mixture is allowed to stand for 12 hours, the clear solution is poured off, from the potassium bisulphate, which crystallises out, and this salt is washed with 10–12 cc. of water. The solution is warmed to 80–90° C., and treated with 150 cc. of strong sulphuric acid and sufficient water to redissolve any chromic acid that may separate. The solution is concentrated until a crystalline crust forms on the surface, and is then set aside for 12 hours, after which the chromic acid which separates is collected on a platinum filtering-cone by the aid of the pump. Two further crops of crystals may be obtained on again concentrating. The temperature of concentration should be moderate lest the chromic acid be attacked by the sulphuric acid. 100 grms. of the chromic acid thus prepared are dissolved in 300 grms. of water and 30 grms. of sulphuric acid of sp. gr. 1.704, the resulting solution having a specific gravity of 1.2, which should not be exceeded, as otherwise too much oxygen will be given off during the solution of the steel.

(2.) *Sulphuric Acid*.—This should have a specific gravity of 1.704 at 16° C., corresponding with a content of 78 per cent. of  $\text{H}_2\text{SO}_4$ . It is prepared by mixing 1,000 cc. of the strongest sulphuric acid (not necessarily pure) with 500 cc. of water and 10 grms. of chromic acid. The mixture is heated for an hour on the sand bath. The burner is then removed and air blown through the liquid for five minutes to expel any  $\text{CO}_2$  that may have been formed. Seeing that the acid is somewhat concentrated by the heating it is diluted with water to the right specific gravity. The strength should be exact within 1–2 per cent.

The actual analysis is carried out by weighing 0.5 grm. of iron very exactly into the solution flask, adding 2.3 grms. of mercury from a small pipette, exhausting the flask by connecting *k* with the water pump, placing 30 cc. of the chromic acid solution in the funnel *o*, and allowing it to run into the flask by cautiously lifting the tube *m* from the seat into which it is ground at the bottom of the funnel. The contents of the flask are heated to boiling point by a small flame, and 120 cc. of the sulphuric acid prepared as described above allowed to run in through the funnel, and the boiling continued for 30 minutes. Connection with the gas burette is made when sufficient  $\text{CO}_2$  has been evolved to prevent any risk of mercury being sucked back into the solution flask. This is usually the case after the addition of the sulphuric acid. At first only  $\text{CO}_2$  is given off, but as the temperature rises oxygen is also evolved. When the reaction is complete the burner is removed, the funnel filled with distilled water, and allowed to run gently into the flask, until the whole of the gas has been driven over into the burette. Should there not be enough gas to reach to the graduated portion of the burette air is admitted until the level of the mercury has fallen to the proper point. The determination of the  $\text{CO}_2$  is then proceeded with in the usual way.—B. B.

#### *The Determination of Phosphorus in Coal and Coke.* J. Lynchenheim, Swedeland, Pa. Eng. and Mining J., 1894, 222.

In connection with the manufacture of special Bessemer pig, containing 0.030 per cent. or less of phosphorus, it was found that the phosphorus in anthracite varied so greatly that it was necessary to sample and analyse each truckload. This, of course, in addition to routine work,

involved a considerable amount of extra trouble, and some experiments were made, with the view of cutting down the time and labour of the determination to a minimum. As there seemed to be no question that all the phosphorus is to be found in the ash, the old method of fusing 5 grms. of coal with 10 grms. of sodium carbonate, and 40 grms. of potassium nitrate was not tried. It involved too much attention in preventing the fused mass from overflowing the crucible. The author concludes: "I had been under the impression that fusion of the ash or residue was necessary in all cases; but wishing to ascertain exactly what proportion was dissolved out by hydrochloric acid, and what remained in the residue, I tried the experiment of treating the ash exactly like an ore, transferring it to a 5-inch crucible; adding 40 cc. of strong  $\text{HCl}$ ; evaporating to 10 cc.; then adding 40 cc. of nitric acid (specific gravity 1.42) and evaporating until all brown fumes had passed off and the solution contained about 20 or 25 cc. The nitric acid solution has a strong tendency to "bump;" and care must be taken to keep the contents of the crucible in motion. The results showed that the phosphorus was almost completely soluble in hydrochloric acid and only a trace remained in the residue. This, then, was a solution of the problem by which results could easily be obtained in from an hour to an hour and a half, with little labour and attention on the part of the operator. It is just as satisfactory and gives just as concordant results as the longer methods.

"In all this work we have been using a very simple arrangement, by which the combustion of the carbon was considerably hastened. In fact, it was found much more satisfactory than the large fusion crucibles we had previously employed. Its comparative cheapness enabled us to run six analyses at a time, and also did away with the expensive necessity of laying in a supply of large crucibles. It consists of a boat 2 inch square and  $\frac{1}{2}$  inch deep, made from 0.002 platinum foil. Care should be taken in making the boats that the corner flaps fit tightly, so that none of the ash will be lost by getting into the interstices. A tripod, Erdmann chimney, and two pieces of thin platinum wire complete the arrangement. The wires should be so bent as to be about  $\frac{1}{2}$  inch below the top of the chimney. The heat applied for the first five minutes should be a low red, in order that none of the coal shall be lost in the escape of the volatile matter. After that the gas should be turned on full, and a bright red heat maintained. It is not necessary that the sample should be ground very finely. By constant stirring we have effected the combustion of the carbon in half an hour, and with an occasional stirring it should never take more than an hour."

#### *Valuation of Ground Phosphatic Slags.* E. Wrampelmeyer. Landw. Versuchsst. 1893, 43, 183.

A MICROSCOPICAL examination of the sample reveals the presence of natural phosphates through the light colour and the rounded outline of the single grains. In suspicious cases the loss of weight on ignition, the specific gravity and the solubility in water are ascertained. These tests may be followed by the determination of the solubility in citric acid according to Loges and Petermann.

For the determination of the specific gravity the use of a small strong glass vessel of from 8 to 10 cc. capacity is recommended. This is filled with the powder by gentle knocking until no further consolidation takes place. The subsequent operations do not require description.

The amount of soluble matter is estimated in 2 grms. of substance, previously well ignited (but not fritted) in a platinum dish, by the loss of weight brought about by repeated boiling with water and washing on a filter until the filtrate no longer shows an alkaline reaction or leaves a stable residue on evaporation.

Loges' method of estimating the phosphoric acid soluble in citric acid is best carried out in the following manner:—1 grm. of the sample is digested for 12 hours at a temperature of from 50 to 70° C. with 150 cc. of a 5 per cent. solution of citric acid, 100 cc. of water are then added; the mixture is boiled for 1 minute and after cooling made up to 500 cc. Of the filtered solution 250 cc. are reduced by

evaporation to about 50 cc. mixed with 13 cc. of 5 per cent. citric acid solution and neutralised with ammonia. The phosphoric acid is precipitated by the addition of 25 cc. magnesia mixture, and one-third of the total volume of 10 per cent. ammonia, allowing the liquor to stand for at least two hours.—F. M.

### ORGANIC CHEMISTRY.—QUALITATIVE.

*The Poisonous Action of Martins Yellow, and its Detection.* D. Vitali. Boll. chim. farmac. 1893, 32, 738.

MARTINS yellow, often used as a food-colouring, may be detected in the urine by the following methods:—

1. The urine, slightly acidified by hydrochloric acid, is shaken up with ether. A portion of the ethereal layer is shaken with caustic potash solution, the alkaline solution then acidified with hydrochloric acid and warmed, some strands of white wool, free from fat and mordanted with alum solution, are now placed in it. The wool is coloured yellow in the presence of as little as  $\frac{1}{1000000}$ th grm. of the colour.

2. A second portion of the ethereal solution is evaporated to dryness, a drop of potassium cyanide solution added to the residue, when a red coloration is obtained.

3. Another portion of residue by evaporation of the ethereal solution is mixed with potassium bisulphate, heated to redness in a glass tube, and the residue dissolved in water. A paper moistened with Griess's reagent and placed in the solution is coloured a beautiful violet.

4. A solution of Martins yellow or urine containing it, gives with cobalt chloride and a little caustic potash, a fine green pigment.

5. With stannous chloride and a trace of ammonia, Martins yellow gives a white precipitate, which becomes rose-red by subsequent treatment with ammonia.

For the recognition of Martins yellow in the stomach, intestines, &c., these are finely cut up, acidified slightly with hydrochloric acid, and digested for some hours at 40°–50° with absolute alcohol. The liquid is filtered, evaporated at a low temperature, made alkaline by caustic potash, filtered, acidified by hydrochloric acid, and shaken out with ether. The ethereal solution is then examined as above.

A dog, on being given daily doses of Martins yellow, beginning with 0.02 grm. and increasing by this amount each day, passed urine containing this colouring matter on the second day, showed poisoning symptoms on the eighth day, and died on the fifteenth day. After death, the colouring matter was found in the abdominal organs, especially the liver; examination showed hemorrhagic intestinal inflammation, and venous infiltration of brain cuticle.

Other trials on a human subject with large doses, produced yellowness of skin and unpleasant sickly feeling which soon passed away; small doses over an extended period produced no effect whatever.

The results obtained confirm Cazaneuve's observations. The small quantity that may be found in foods is shown by these experiments to be quite harmless.—W. B. H.

### *The Colour Reaction of Hydrazine Sulphate with Lignin and some Aldehydes.* E. Nickel. Chem. Zeit. 17, 1243.

THE author has compared the reaction of the most simply constituted hydrazine  $\text{H}_2\text{N}-\text{NH}_2$  with that of phenyl hydrazine, recently described by him (this Journal, 1893, 869). A cold solution of hydrazine sulphate applied to wood produces a distinct bright yellow coloration varying in intensity with different species of timber. By the action of 20 per cent. hydrochloric acid, the fully developed yellow is converted into an orange, the intensity of which gradually increases.

It having been pointed out that the colour reactions of wood with phenols and the corresponding nitrogenous compounds are owing to its aldehydic nature, the study of the reactions of hydrazine with aldehydes and ketones next suggested itself. Vanillin and cinnamic aldehyde have

both been held responsible for the colour-reactions of wood. The behaviour of hydrazine sulphate however furnishes an additional proof of the fallacy of the conclusions derived from a few colour reactions as regards the nature of a substance tested in this manner. Hydrazine sulphate solution added to an aqueous or slightly alcoholic solution of vanillin produces a strong yellow coloration, but no precipitate is formed until after the addition of hydrochloric acid. Even a continued action of the hydrochloric acid does not, as in the case of wood, change the yellow into orange. Piperonal, *p*-hydroxybenzaldehyde and salicylic aldehyde behave similarly, the last mentioned showing only a very faint yellow.—F. M.

### *The Distinction of Birch-tar from Pine-Tar.* E. Hirsch-olm. Pharm. Zeits. Russl. 1893, 32, 657.

A COMPARATIVE study of these preparations, either one or the other of which is official in the various pharmacopœias, has resulted in the following facts:—

*Birch-tar*, sp. gr. 0.920–0.945 at 20° C. When shaken with water (1:10) an almost colorless extract is obtained, possessing an acid reaction, and coloured green by the addition of ferric chloride. 5 cc. of the extract treated with 2–3 drops of aniline and 4–6 drops of hydrochloric acid, give a yellowish mixture. One volume of birch-tar mixed with 20 vols. of petroleum ether and filtered, yields a pale brown filtrate, which is not coloured green when shaken with an equal volume of copper acetate solution (1:1,000).

*Pine-tar*, sp. gr. 1.02–1.15 at 20° C. The aqueous extract is yellowish, acid in reaction, and is coloured red by ferric chloride. With aniline and hydrochloric acid an intensely red mixture is obtained which yields up its colouring matter to chloroform. The petroleum-ether extract of the tar assumes a green tint when shaken with copper acetate solution. One volume of pine-tar dissolves to a clear liquid in 9 vols. of 90 per cent. alcohol. If the solution be turbid, the presence of birch-tar, kerosene, kerosene residues, &c., is indicated.—H. T. P.

### *The Detection of Mineral Acids in the Presence of Organic Acids by Colour Reactions.* E. Nickel. Chem. Zeit. 17, 1670.

IN this test the colour reaction obtained with phloroglucinol on pine-wood shavings in the presence of free mineral acids is made use of. A shaving or splint of pine wood or bamboo wood is immersed in the acid to be tested, to which a liberal addition of phloroglucinol has been made, and finally the whole is boiled for a short time in order to completely dissolve the phloroglucinol. The colour reaction, as a rule, soon becomes perceptible, and is particularly pronounced when bamboo wood is employed, although in this case from 12 to 24 hours may elapse before the coloration reaches a maximum.—C. O. W.

### *The Detection of Mineral Acids in Vinegar by Means of Rosaniline Hydrochloride.* G. Griggi, Selmi, 1893, 3, 176.

ONE drop of a solution of rosaniline hydrochloride (25 grms. fuchsin in 100 cc. of 90 per cent. alcohol) is added to 1 cc. of vinegar contained in a shallow porcelain dish. In the case of pure vinegar, the fuchsin retains its reddish-violet colour; but in presence of small amounts of mineral acids (1 per cent.) the liquid assumes a dirty yellow tint. Addition of alkali restores the original colour of the reagent.—H. T. P.

### *Detection of Sesame Oil in Olive Oil.* F. Musset. Pharm. Central. H. N. F. 1893, 14, 701.

A SMALL piece of dry phosphorus, weighing about 0.1 grm., is dissolved, by heating in a water-bath, in 10 grms. of the oil contained in a 15 g. bottle. The cork or stopper with which the bottle has been closed is, after cooling, replaced

by a loose plug of cotton wool. By allowing the bottle to stand for some time the phosphorus becomes oxidised, the acid is formed, attracts water and run down the sides in narrow streaks.

If the olive oil is pure, milky streaks will be observed after the lapse of 24 hours, which after 48 hours will be found to have settled at the bottom in the shape of a narrow, perfectly clear ring of yellowish-brown colour. An admixture of 10 per cent. of sesame oil causes the streaks, which after 48 hours all show a distinctly yellow tint, to require almost 8 days to settle. The ring at the bottom consists in this case of two well-defined layers, a lower one of pure oil and an upper opaque and deeply colored one. With 20 per cent. of sesame oil the streaks are yellow at once and do not settle completely, while with 50 per cent. a brown ring is formed on the surface from which descend brown streaks.

Two cc. of ammonia gently added to the oil after the formation of a ring cause the latter if sesame oil be present to assume within 24 hours a more or less deep brown colour, whereas with pure oil it remains colourless.—F. M.

#### *Examination of Glue for the Manufacture of Coloured Papers.* Th. Schlossmann. *Papier Zeit.* 18, 2484—2485.

THE importance of good glue in the manufacture of coloured papers and of a simple and rapid method of estimating the value of the many qualities, which vary in price from 30 to 100 marks per kilo., will be sufficiently obvious. Absolute methods the author rejects, and recommends comparison with some approved standard quality. If a tablet of glue be covered with cold water and allowed to stand it gradually becomes softer, and a considerable increase of volume is observed. The larger the amount of water taken up in a given time, the better the quality. The moment when the tablet begins to break up must also be carefully noted. Lower qualities become slinky after 12 hours, whereas good parchment or leather glue continues to swell and to take up water for twice and thrice the space of 24 hours without breaking up. The water remains clear, containing only minute quantities of gelatin in solution, the tablet feels tough and (this is especially to be noted) there is no trace of bad smell. Inferior qualities, on the contrary, after soaking for a brief period begin to dissolve and emit an offensive odour; the water is turbid and yellowish, and the tablet breaks up on the slightest touch.

The test should be made as follows:—Two strips of the sample to be tested and of the standard are selected of as nearly as possible equal size, say 150 mm. by 50 mm., and by means of a red hot wire a hole is bored in each, about 20 mm. from the upper edge and as nearly as possible in the middle. Two graduated 1,000 c.cm. glass cylinders, 65 mm. in diameter, are filled with clean cold water exactly to the zero point, and a weighed strip is suspended in each by means of a stout brass wire with a hook at one end and a loop at the other, a glass rod being passed through the latter and resting on the top of the cylinder. The experiment should be started in the evening and the observations begun at the end of 12 hours. The strips are raised and allowed to drain back into the cylinder, and the amount of water absorbed is read off. The other points referred to above are also carefully noted, and the observations are repeated every 12 hours until the moment arrives when it is no longer possible to withdraw the strip, which is either cut through by the brass wire or entirely falls to pieces. By a comparison of the percentages of water absorbed, and of the other phenomena observed during the experiment, the relative solubility, the amount of odour emitted, &c., an accurate estimate can be formed of the value of the sample submitted to examination. A good sample, after 2 × 24 hours in summer, and 4 × 24 hours in winter, should be firm to the touch, almost insoluble in water, and above all, should emit no ill odour.—E. G. P. T.

### ORGANIC CHEMISTRY.—QUANTITATIVE.

#### *Estimation of Glue.* *Papier Zeit.* 19, 126.

THE adhesiveness of glue is in proportion to the gluten which it contains, but the chemical estimation of the latter is, in the author's opinion, of doubtful value. On the same grounds he rejects the processes based on the absorption of water (see adjoining column), and on the degree of stiffness possessed by a jelly produced by dissolving in water a certain amount of glue and allowing it to set, and recommends as the only trustworthy method that which consists in sticking pieces of wood together and measuring by means of corresponding weights the force required to pull them asunder. The wood must be thoroughly dried. Pieces 50 cm. long and 5 cm. square section are prepared, cut through the middle with a fine saw, and the cross-grained surfaces stuck together again. The glue to be tested, say 250 grms., is softened for six hours in 1½ litre of water, dissolved, and evaporated until the solution weighs 900 grms. After standing three days the test pieces, resting on each end, are weighted in the middle, 5 kilos, being added every minute until fracture ensues. Good glue should sustain a weight of at least 75 kilos.

It is a common practice with joiners to subject their solution of glue to prolonged heat in the belief that the adhesive power is thereby increased. Experiments with regard to this notion indicate that it is a delusion. In fact, the adhesive power of a glue is actually reduced after six hours boiling, from 250 to 100.—E. G. P. T.

#### *New Method of Estimating Pentosans in Vegetable Substances.* E. Hotter. *Chem. Zeit.* 17, 1743—1745.

IF a solution of furfuraldehyde is heated with hydrochloric acid and an excess of pyrogallol in a closed tube at 100° C. for a few hours, a condensation product is obtained which weighs about 1,974 times as much as the furfuraldehyde employed. On this fact the author bases his method for determining the amount of pentosans in vegetable substances.

From 5—10 grms. of the substance containing the pentosan are distilled with hydrochloric acid of sp. gr. 1.06 until the distillate no longer shows the furfuraldehyde reaction with paper impregnated with aniline acetate. The distillate is then made up to 400—500 cc. with hydrochloric acid of the above strength, and an aliquot portion of it, containing about 0.08—0.1 gm. of furfuraldehyde (usually about 20—30 cc.) taken for the estimation. An excess of pyrogallol is weighed off into a test-tube, which is then constricted near its open end before the blowpipe. After the above aliquot fraction of the solution has been introduced it is sealed off at the narrow part and heated for two hours at 100°—110° C. The precipitate which forms is collected on a weighed filter, washed, dried at 103° C., and weighed. The weight of the precipitate divided by 1,974 gives the amount of furfuraldehyde contained in the quantity of liquid employed. From this the percentage of pentoglucose in the substance is then calculated.

As certain by-products, which vitiate the results, are formed on distilling with hydrochloric acid, the author suggests that perhaps it would be advantageous to distil under diminished pressure. Operating in the way described he found 50—52 per cent. of furfuraldehyde in arabinose and xylose instead of 64 per cent.—J. S.

#### *The Estimation of Saccharine in Presence of Salicylic Acid.* Hairs. *J. de Pharm. d'Angers.*

100 cc. of the liquid to be examined (beer, wine) are acidified with hydrochloric acid, treated with a slight excess of bromine water, and violently agitated. After a short time, the almost insoluble precipitate of bromosalicylic acid is filtered off, the excess of bromine removed by a current of air, and the liquid shaken out with ether. The ethereal extract, after evaporation with addition of a few drops of sodium bicarbonate solution, leaves a residue in which saccharine may be detected by its flavour; or, after fusion with potash, by testing for salicylic acid.

—H. T. P.

*The Determination of Nitrogen in Benzene, Pyridine, and Quinoline Derivatives by Kjeldahl's Method.* M. Krüger. Ber. 27, 609—613.

THE application of Kjeldahl's process to difficultly destructible bodies, such as aromatic hydrocarbon derivatives, alkaloids, &c., has been more or less successfully attempted. Most of the suggested modifications of the method tend towards increasing the oxidative power—by the addition of metallic oxides, &c.—of the acid; nevertheless, in all cases, the bulk of the oxidation has to be done by the sulphuric acid. The author modifies the process by relegating the oxidation of the substance from the very beginning to some powerful oxidising agent, and employs for the purpose chromic acid, or preferably, potassium bichromate. The details are as follows:—0.2 to 0.8 gram. of the substance is dissolved by gently warming in 20 cc. of strong sulphuric acid in a bulb flask of 150 cc. capacity, and allowed to cool. Powdered potassium bichromate is then added in small quantities, and more or less quickly according to the violence of the reaction, the oxidation being assisted after a short time by placing the flask in a water-bath, which is gradually heated to boiling. The total amount of bichromate used should be in excess of that required by theory for the oxidation of the substance (generally 15—20 parts to 1 part of substance). Finally, when the evolution of CO<sub>2</sub> has ceased, the flask is heated for 10 minutes over a small naked flame, allowed to cool, and the distillation of the contents proceeded with as usual. The numerous test

analyses given are excellent. Attempts to apply the method to nitro-, nitroso-, azo-, and similar bodies proved unsatisfactory.—H. T. P.

*The Analysis of India-Rubber Goods.* D. Hilde. Chem. Zeit. 17, 1631—1635 and 1670—1671.

IN a former paper (this Journal, 1893, 463) the author proposed to estimate free oils in india rubber articles, by means of extraction of the finely-subdivided samples with ether-alcohol (1:3). If the extraction is effected with the solvent warm, the process is very tedious, owing to some of the other constituents of the india-rubber articles also slowly dissolving, in consequence of which even a four times repeated extraction is not sufficient to completely exhaust the samples. For the extraction of the free oil alone, however, treatment with the cold solvent was found to succeed in one operation. About 1 gram. of the finely-divided substance is placed on a filter in a funnel, and extra ted with 50 cc. of ether alcohol. The filtrate was collected in a weighed Erlenmeyer flask, the solvent distilled off, the flask dried in an air bath and subsequently weighed again. The weight of oil so obtained requires, however, a correction, owing to the solvent action of the ether alcohol upon the caoutchouc itself, of which it dissolves on an average 5 per cent., the observed maximum being 7 per cent. The working of the method will be seen from the following results:—

Nature of India-Rubber Article.	Nature of the Added Oil.	Quantity of Oil Added.	Per Cent. of Oil found.		Total Extract.	Ether-Alcohol Employed.	
			Percentage of Extract to be Deducted for Pure Rubber.				
			7 Per Cent.	5 Per Cent.			
		Cc.			Per Cent. 6.3	Cc. 39	
Sample I. :— Finely ground white eraser india-rubber.	....	0	..	..	7.0	} 64	
					3.7		
					7.1		
					6.8		
					3.1		
	Refined colza oil.....	22.1	21.1	22.1	21.1	39	} 60
		12.9	12.5	14.1	18.4		
		18.5	18.5	21.2	23.2		
		26.8	26.9	28.4	12.0		
	Cotton-seed oil.....	59.8	58.8	59.7	61.7		
Sample II. :— India-rubber stopper broken up into small coarse fragments.	....	0	..	..	2.8	} 21	
					3.1		
					3.1		
					2.5		
					3.7		
	Refined colza oil.....	17.0	} Much too little oil, i.e. total extract.		12.5	} 20	
		15.8			14.6		
		19.0			15.5		
		15.7			11.1		12.9
		26.2	22.5	24.2	28.0	61	
Sample III. :— Refined colza oil ....	5	..	..	4.1	} 61		
	26.7	22.2	23.8	17.6			
White tubing reduced to coarse granular fragments.	Sample IV. :— Cotton-seed oil.....	0	..	..	3.9	} 60	
					5.3		
		29.3	36.7	37.7	10.9		
		60.2	57.7	58.6	60.8		

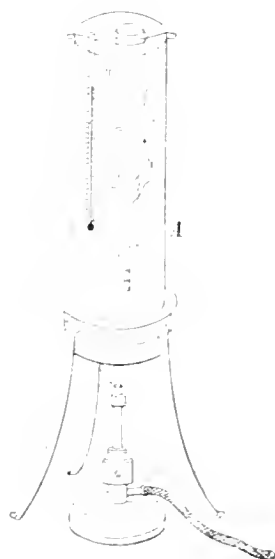
White tubing reduced to coarse granular fragments.



In a former paper (this Journal, 1893, *loc. cit.*) the author showed that the india rubber substitutes prepared from fatty oils by means of sulphur chloride, yield only traces of sulphur to ether-alcohol, so that the above process offers a means for the separation from india-rubber of the fatty oils in the presence of such substitutes. Should any rubber sample also contain mineral oils, resin oil, &c., which are also soluble in ether-alcohol, the further quantitative examination of the extract by well-known methods becomes necessary.—C. O. W.

*The Estimation of Indigotine in Indigo.* C. Donath and R. Strasser. *Zeits. f. ang. Chem.* 1894, 49.

1 GRM. of the finely ground indigo is weighed out in a weighing flask, and mixed therein with four times its volume of moderately fine, dry pumice stone sand. This mixture is then placed in a spacious Soxhlet extractor, on the bottom of which is firmly secured a layer of asbestos fibre, reaching above the opening of the lateral siphon (see Fig.), and covered with a thin layer of pumice stone sand. The weighing flask, after being emptied into the extractor, is also rinsed with pumice stone sand. The indigo is then extracted with water and hydrochloric acid, a small quantity of the latter being added through the condenser as soon as a sufficient quantity of water has accumulated in the extractor. The extraction of the glutinous matters is complete after 15 minutes' boiling, and the indigo is then washed with hot water, which is poured direct into the extractor. The extractor is then adjusted to the neck of a flask half filled with a mixture of four parts of alcohol and one part of ether, and with this mixture the indigo is extracted until the brownish-red colour of the indigo-red has altogether disappeared, the liquid running from the extractor being perfectly colourless, or at most only exhibiting a faint blue coloration. The extractor, with contents, is now completely dried at from 100° to 110° C. The delivery end of the siphon of the extractor is then closed with an asbestos plug, the contents of the extractor are completely covered with concentrated sulphuric acid, and the extractor, by means of copper wires, is now suspended in an air-bath (see Fig.). This air-bath consists of the copper cylinder A closed at the bottom and placed on a tripod. After heating the apparatus for from 1½ to 2 hours to 80° C. the sulphonation of the indigo may be relied upon as completed. The extractor is then left to cool, subsequently the plug removed from the siphon and distilled water poured into the extractor. The indigo-sulphonic acid formed is thus siphoned off and collected in a litre flask, the last traces of



dye may be removed from the asbestos by washing with boiling water. The contents of the litre flask are allowed

to cool, and the whole is then made up to 1,000 cc. 100 cc. of this solution are diluted with 400 cc. of water and titrated with a solution of permanganate, 1 cc. of which corresponds to 0.0099139 gram. of indigotine. Four estimations of a Bengal indigo showed the percentage of indigotine in four experiments to be 54.53, 54.13, 54.62, and 54.73 per cent. respectively.—C. O. W.

*Analysis of Tanning Materials.* A. Folsing. *Chem. Zeit.* 17, 1770—1771.

THE gravimetric method of estimating the tannin in a tannin is open to the objection that a number of other substances, such as metallic salts, phenols, alcohols, picric acid, &c., are precipitated by hide, and therefore included in the difference between the total solids and the solids not tannin.

Schreier's method of valuing a tanning material, not applicable to extracts, is devised for the examination of raw materials with simple appliances. It consists in introducing 50—100 grms. of the ground material into a flask, together with 1 litre of water at 15°; the flask is corked and shaken intermittently during 24 hours. At the end of this time the liquor is filtered through paper, and its specific gravity is taken by means of a special hydrometer; comparison with published tables shows the percentage of tannin which would be contained in a liquor from the same class of material and of the same specific gravity.

The colour and turbidity of extracts may be compared by measuring 10 cc. of each into a litre cylinder, adding water to the mark, shaking, and allowing the cylinders to stand before a window for half an hour, when a comparison may be made. In the case of a solid extract 5 grms. should be weighed out and dissolved in 10 cc. of water in a dish on the water-bath; the sample may then be treated as described above. Equality of temperature is important in making the comparison.

The author's colorimetric method consists in placing 0.5 gram. of the sample and of a sample of the same species of bark of known tannin-content, in each of two "colour glasses." Equality of water content is necessary, so that each sample must be dried at 110° before being weighed. Half a litre of water is added to each glass, and then a strip of textile material, which is subsequently dyed with madder on the water-bath. The dried strips are then compared. The quality of the shade will indicate which specimen is the poorer in tannin. Extracts must be prepared for this test by weighing 2 grms. into a litre flask, making the solution up to the mark, and transferring 100 cc. to the "colour glass," containing half a litre of water.

The introduction of hydrogen dioxide as a bleaching agent for leather is noteworthy. Foelsing's electrical tanning process differs from others in that the hides are kept at rest and the liquor in motion; it is alleged to have proved of service in tanning heavy leather.—A. G. B.

*The Oxidation of the Fatty Oils.* W. Fabrian. *Chem. Zeit.* 17, 1453.

See under XII., pages 404 and 405.

*Analysis of Mixtures of Sperm Oil and Mineral Oil.* C. A. Lobey de Bruyn. *Chem. Zeit.* 17, 1453.

PURE sperm oil contains at least 25 per cent. of the higher primary alcohols, which are insoluble in water, and can be separated from the solution of the saponified oil in alcohol by means of petroleum ether (this Journal, 1892, 134—145). In applying to such an oil Holde's test for the detection of mineral oil in sperm oil (boiling a few drops of the oil with the solution of a small piece of caustic potash in a few cc. of alcohol) and diluting the soap solution so obtained with water, the higher alcohols remain for a considerable length of time dissolved in the soap solution, so that this test can be used for the detection of mineral oil in sperm oil.

Sperm oil may be adulterated with seed oil, such mixtures possessing the same specific gravity as pure sperm oil. Mixtures of this kind are analysed according to Allen and Thomson's method, by heating 5 grms. of the oil with a solution of 2 grms. of caustic soda in 25 cc. of alcohol, but it is advisable to boil for some hours previous to evaporation. The residue obtained on evaporation is dissolved in water, and the unsaponifiable matter extracted by twice shaking the solution with petroleum ether. The latter is distilled from a weighed flask. For the separation of the mineral oil and the higher alcohols in the residue obtained after distillation, it is boiled for half an hour with twice its volume of acetic anhydride. On cooling, any mineral oil present separates out completely, the higher alcohols remaining in solution. The two layers are separated, the mineral oil is distilled off, washed with dilute alkali, dissolved in petroleum ether, and after drying the latter with calcium chloride it is distilled off. Pure mineral oil remains behind. The acetic anhydride separated as above from the mineral oil, contains the higher alcohol in the form of esters. These may be obtained by diluting the acetic anhydrides with water, when the esters of the higher alcohols separate out, forming an oily layer upon the surface of the water. Amagat and Jean's refractometer also appears to prove very useful in testing the purity of sperm oils. — C. O. W.

*Note on some Modifications of the Reichert-Meissl Method of detecting Margarine in Butter, and a New Method for the Determination of the Reichert-Meissl Number.* C. Bunte. (Chem. Zeit. 13, 201-206.)

THE author has investigated Kreis's method of saponifying butter-fat by means of sulphuric acid, in order to determine the influence of various factors, such as the concentration of the acid employed, temperature, &c., on the resulting "Reichert-Meissl" numbers. The figures obtained are given in a series of tables, as also, for the sake of comparison, the Reichert-Meissl values obtained in the ordinary way. From these it appears that:—

1. Small variations in the initial temperature of the butter-fat cause considerable differences in the R.M. value.  
2. When Kreis's directions are strictly adhered to, the figures obtained fall considerably below the truth, in spite of the sulphurous acid which is *always* formed and which of course raises the results.

3. In Kreis's method, therefore, saponification is incomplete, the degree of completeness depending on the concentration of the acid and the temperature of the butter.

4. Taking also into account the difficulty of preparing strong sulphuric acid of a particular specific gravity, Kreis's method must be entirely condemned.

The author finds, however, that by slightly modifying the process, reliable figures may be obtained which compare well with those yielded by the Reichert-Meissl method. The details are as follows:—5 grms. of the butter-fat to be examined, contained in an Erlenmeyer flask (1,000 cc. capacity) are heated to 100° C. in a drying chamber. 10 cc. of sulphuric acid (1.8355 sp. gr.) are then added, the flask shaken until the butter is dissolved, and placed for 10 minutes on a water bath maintained at 30°–32° C. 150 cc. of water are then added with constant agitation, the liquid titrated with strong permanganate solution until the red colour remains permanent for a few moments, and finally distilled in the usual way. It is essential that the above directions be carried out to the letter. The sulphuric acid should be exactly of the strength indicated (to the third place of decimals). (This Journal, 1893, 872 and 952.)—H. T. P.

*The Determination of the Reducing Action of Organic Substances contained in Water upon Permanganate of Potassium.* A. Zega. Chem. Zeit. 18, 2.

THE author has found the methods of Kubel and Schulze defective in the case of volatile organic substances, and proposes instead the following:—50 cc. of the water to be tested are placed in a globular flask with a capacity of about 100 cc. 5 cc. of the usual solution of permanganate are added and 5 cc. of dilute sulphuric acid (1 vol. acid to

2 vols. water). The mixture is placed on a boiling water bath for 20 minutes and the remaining permanganate titrated as usual with oxalic acid.—J. H. C.

*Determination of Oil of Turpentine in Alcohol.* J. Perl. Chem. Zeit. 17, 1851.

FOR certain purposes alcohol is denatured with oil of turpentine. The quantity of oil present in a given sample may be determined by distilling a known quantity over quickly, and determining how much water must be added to the distillate to produce permanent turbidity. Alcohol actually containing 0.5 per cent. was thus found to contain 0.11 when valued by comparison with undistilled mixtures of alcohol and oil of turpentine; when the distillate was collected in four nearly equal fractions, the first indicated 0.5, the second and third 0.14, and the last 0.1 per cent., average = 0.44.—C. R. A. W.

## ANALYTICAL AND SCIENTIFIC NOTES.

### *International Standards for the Analysis of Iron and Steel.*

At the World's Congress of Chemists in Chicago, a sub-committee of the original committee on international standards for the analysis of iron and steel, was appointed to consider the subject of standard methods.

This sub-committee consists of Dr. C. B. Dudley, chairman, Messrs. A. A. Blair, W. P. Barba, P. W. Shimer, and T. M. Brown. It has recently held a meeting and has decided to recommend standard methods in iron and steel analysis, to be used as the basis of commercial transactions. The sub-committee fully appreciates the fact that these methods, to have the highest value, should be in facility and in time of execution such that they will readily recommend themselves for daily use in iron and steel works.

To further this end, the sub-committee wishes to have the co-operation of the iron and steel chemists of the country, and to ask them for a brief outline of the processes or methods they use and prefer for the determination of different elements in iron and steel, and for such other information and suggestions as they think will aid it in the work before it. The sub-committee recognises the fact that it will add immensely to its efficiency and value if the iron and steel analysts of the country will take a personal interest in it, and aid it by their counsel and active influence.

They are therefore requested to send to the chairman of the sub-committee, as soon as convenient, such an outline as they may deem sufficient to fully describe their practice. It is suggested that they follow the general plan here indicated, by answering the following questions, which may be referred to by number to save unnecessary trouble:—

1. What general method do you use for the determination of phosphorus in iron and steel?

2. What special precautions do you consider necessary to make this method reliable?

3. What precautions do you take to prevent the interference of arsenic?

4. What factors do you use in your calculations?

5. What variations do you introduce in the case of iron ores or slags?

6. Do you use the same method in pig iron and steel, and do you consider the results equally reliable?

7. Do you ever examine the residues insoluble in acid, in pig irons, or iron ores, and do you ever find phosphorus in them?

8. Are all your determinations made by the same method, or do you check your work by reference to another method, and if so, what method do you use for this purpose?

9. How many determinations do you make a day in your laboratory under ordinary circumstances?

10. What do you consider the greatest length of time necessary to obtain a result, permissible in your work?

The sub-committee begs that chemists will send at the earliest possible moment as full replies to all or any of the above questions as they conveniently can, and assures them

that in making use of any details that may be original, they shall have full credit. They will likewise be furnished with copies of the various reports. Replies should be sent to Dr. Chas. B. Dudley, Chairman, Sub-committee, Altoona, Pa.

*Colloidal Lead Chloride.* A. van de Velle. Chem. Zeit. 17, 1908.

On mixing together two solutions, one containing 379 grms. (1 molecule) of sugar of lead, the other 117 grms. (2 molecules) of sodium chloride per litre, a slimy precipitate forms consisting of a mixture of amorphous lead chloride and crystals of the same substance. Excess of one or other salt does not alter the result but the weaker the solutions the more the crystalline predominates; on filtering, the liquid passes through turbid, whilst what remains on the filter becomes crystallised on washing with water. Potassium, ammonium, calcium, or bromium chloride may be used instead of sodium chloride; hydrochloric acid, however, forms only a crystalline precipitate. Basic lead acetate or lead propionate, formate, or lactate may be used instead of sugar of lead, but lead nitrate, chlorate, or perchlorate only yield fine crystals.—C. R. A. W.

*Electrical Phenomenon observed in the Preparation of Salipyrine.* D. Schrijnen. Pharm. C.-H., N.F., 1893, 14, 727.

The crystalline product obtained by allowing the oily liquid to cool, which results from the treatment on the water bath of antipyrine with salicylic acid and water and subsequent evaporation of the latter, emits when crushed in a mortar a fine bluish-white light, the peculiar crackle of electric sparks being at the same time noticeable. On grinding the substance in the dark these sparks may be seen in profusion. The electricity is supposed to be generated by the friction of the crystal surfaces against each other.—F. M.

*Measurement of Radiation of Light by means of the Radiometer.* N. Techn. Journ. Prakt. Chem. 47 (1893), 568—583.

With the object of finding whether Crookes's radiometer could be employed in measurements of diaphaneity (diaphanität) towards radiant energy, the author first investigated a method of determining accurately the rate of rotation of the vanes of the radiometer.

The radiometer was enclosed in a metal case blackened on the inside and open in the direction of the radiation. The case was also provided with a side-tube (also blackened on the inside), through which the observer could examine the vanes. When a rotating vane came into a certain position with reference to the source of light and the observer's eye, a flash of light was seen. With the aid of a chronoscope the instant at which this occurred was noted. A suitable number of vane-transits were observed and counted, and the time which had elapsed was again determined with the aid of the chronoscope. The method is susceptible of considerable accuracy, and was first employed in testing the ratio between the number of vane-transits in a given time when the source of light was (a) at a distance of 20 cm., (b) at a distance of 40 cm., from the radiometer. In three successive experiments the ratio of (a) to (b) was found to be 3.95, 3.99, and 4.00.

This is exceedingly near the theoretical value of 4 : 1, and shows that the rate of rotation of the radiometer-vanes is a correct measure of the intensity of radiation.

The author appears subsequently to have become aware that Crookes had already shown this, although in a somewhat different manner.

In endeavouring to measure the diaphaneity of bodies (glass plates, liquids in flat troughs, &c.), the author had either to rely upon the absolute constancy of some source of light, or else to devise a method which should be quite independent of this. The method adopted consisted in using two independent radiometers, which were first placed at such distances from the source that they rotated at the

same speed. The plate or solution to be examined was then introduced in front of one of them, and by readjusting the distances the rates of rotation could again be equalised. Some of the results obtained are given below, the diaphaneity of water being in all cases taken as 100 :—

(1.) Saturated aqueous solutions exhibit only slight differences in diaphaneity.

Water.....	100.00
Sodium chloride.....	103.75
Oxalic acid.....	98.79
Citric acid.....	101.37
Boric acid.....	99.69
Borax.....	99.14
Sugar.....	101.29
Alum.....	99.06
Tartaric acid.....	99.89
Ammonia.....	99.62

(2.) The diaphaneity of compounds of the fatty series increases with increasing molecular weight.

(3.) In the case of aromatic compounds the converse is true—

Benzene.....	128.62
Toluene.....	126.32
Xylene.....	124.95

(4.) Some isomeric compounds differ greatly in diaphaneity, e.g., acetic acid and methyl formate, ethyl acetate and butyric acid, methyl butyrate and valeric acid; whereas others only differ very slightly, e.g., propyl- and isopropyl-alcohol, butyl- and isobutyl-alcohol.

(5.) The diaphaneity of carbon bisulphide (141.70) exceeds that of glass (140.77); and it is remarkable that carbon tetrachloride (145.77) is more diaphanous than air (142.26).—D. E. J.

*Decomposition of Liquids by Contact with Powdered Silica.* G. Gore. Phil. Soc. of Birmingham, 1893, 9 [1], 1—24.

A SOLUTION of an acid, alkali, or salt, having no chemical action on pure precipitated silica, was well shaken up in a bottle with 50 grains of the silica, and after 16 hours' standing it was found that the film of liquid adhering to the powder contained a higher percentage of the dissolved substance (acid, alkali, or salt) than the supernatant solution itself in bulk. The amount of solid thus abstracted from the solution varies with the kind of powder employed, its degree of fineness, the character of the dissolved substance, the ratio of the powder to dissolved substance, the nature of the solvent, the proportion of solvent to powder, the degree of dilution of the solution, and to a certain extent to the temperature. The union ensues quickly and prolongation of immersion has but little influence. Finely precipitated silica possesses the property in the highest degree, and alkaline substances are most affected. With very dilute alkaline solutions more than 80 per cent. of the dissolved substance was abstracted by the silica. The results throw some light undoubtedly upon :—

(a.) The purification of water by sand filtration.

(b.) The retention of the alkaline constituents of soils, in a greater degree by the silica than by the alumina.

(c.) The great losses incurred by the gold extractor (MacArthur, Forrest process) through the action of the silica of the pulverised quartz upon the weak potassium cyanide solutions employed for the extraction of gold and silver. It is a case of simple adhesion of the cyanide to the silica (see also p. 339).—W. S.

## New Books.

**ODOROGRAPHIA.** A Natural History of Raw Materials and Drugs used in the Perfume Industry, including the Aromatics used in Flavouring; intended for the Use of Growers, Manufacturers and Consumers. By J. CH. SAWYER, F.L.S. Second Series. London: Gurney and Jackson, 1, Paternoster Row (Successors to Mr. Van Voorst). Brighton: W. J. Smith, 43, North Street. 1894. 15s.

THIS forms the second volume of the handbook bearing the above title, the notice of the first volume having appeared in this Journal, 1893, page 71. It is of similar size and appearance, and contains 74 illustrations of various plants, &c., bearing essences, oils, &c.

The number of pages of subject-matter in this volume is 523, the final two pages being occupied by a table representing the "Solvent Power of Essential Oils" and a Table of Weights and Measures, &c. The work commences with a Preface, a Table of General Contents, a List of Illustrations, and another of Principal Works referred to, with Abbreviations.

The whole work is divided into five sections. SECTION I. commences with Nutmegs and Mace, Sassafras, Laurus Penzance, Pimento, Myrtle, and ends with Mint, Chamomile, Eucalyptus, and Odorous Waxes. It occupies pages 1 to 346.

SECTION II. treats of Ferment Oils, Ferments, Organic Ethers, Bouquet of Wines, Brandies, &c. Theory of Compound Odours, Oil Secretions, &c. Pages 347—376.

SECTION III. Emphysematic Oils and Aromatic Products of Destructive Distillation, including Russia Leather, Casearilla, Caffene, Furfural, &c. Pages 377—394.

SECTION IV. Addenda to Volume I. Vanillin, Musk Substitutes, Rosemary, Cloves, Mignonette, Rose, Lavender, Geranium, Bergamot, Violet, and Orris, &c. Pages 395—457.

SECTION V. Descriptive of Plants yielding purely Floral Perfumes. Pages 459—520.

The work ends with an Alphabetical Index.

**A HANDBOOK OF GOLD MILLING.** By HENRY LOUIS, Assoc. Roy. School of Mines, &c. London and New York: Macmillan and Co. 1894. 10s.

8vo. volume bound in cloth, containing frontispiece representing a Stamp Mill in the sixteenth Century, Preface, Table of Contents, Subject-matter, covering 477 pages, an Appendix of 16 pages, and an Alphabetical Index. The text is illustrated with upwards of 118 well-executed wood-engravings. The subject is subdivided for treatment in the pages of this work, as follows:—I. Mode of Occurrence. II. Physical and Chemical Properties of Gold. III. Physical and Chemical Properties of Mercury. IV. Alloys and Amalgams of Gold. V. Primitive Methods. VI. General Arrangements. VII. The Stamp, Tappet, Stem, Head, Shoe, Cam Shaft, &c. VIII. Frames, Guides, Hoisting Gear, Water Supply, Ore Feeders, &c. IX. Other Crushing Machinery. Huntington Mill. X. Amalgamation, &c. XI. Principles of Concentration. XII. Treatment of Concentrates, Amalgamation, Chlorination, Cyanidation, &c. XIII. Cleaning-up, Treatment of Amalgam, &c. XIV. Modes of Treatment, Cost of Milling, Returns. XV. Sampling and Assaying of Ore, Tailings, Concentrates, and Bullion.

**THE GAS ENGINEER'S LABORATORY HANDBOOK.** By JOHN HORNBY, F.I.C. London: E. and F. N. Spon, 125, Strand. New York: Spon and Chamberlain, 12, Cortlandt Street. 1894. 6s.

THIS little work is dedicated by the author to his chief, Mr. John Methven, of the Beckton Works. Part I. treats of various preliminary operations in general quantitative work. II. Describes the methods of estimating gravimetrically, certain pure substances, and typical of the more

ordinary analyses conducted in gasworks. III. General account of operations connected with volumetric analysis, followed by some simple volumetric processes applicable to gas-testing. IV. Describes the more complex determinations required in gasworks, some gravimetric, some volumetric. Methods of Analysis of Coal, Fresh and Spent Purifying Material, Ammoniacal Liquor, Firebricks and Clay, Tar, Crude Gas and Purified Gas, &c., are described. V. Technical Gas Analysis, &c. The work is illustrated with 63 engravings, and numerous tables are given. An alphabetical index is supplied. The book contains 296 pages of subject-matter inclusive of appendix containing the tabular matter.

**TABELLÄRISCHE ÜBERSICHT ÜBER DIE KUNSTLICHEN ORGANISCHEN FARBSTOFFE UND IHRE ANWENDUNG IN FÄRBEREI UND ZEIGERIECK.** Von Dr. ADOLF LEHNE. Herausgeber der Farber-Zeitung. Vierte Lieferung. Berlin: Verlag von Julius Springer. 1894. London: H. Grevel and Co., 33, King Street, Covent Garden.

THIS beautifully illustrated work has now reached the issue of Part IV., which contains dyed and printed samples illustrating colours No. 180—219, commencing with Benzo-brown G and ending with Pyronine G. (This Journal, 1893, 633.)

**TEINTURE ET IMPRESSION.** Par M. PRUD'HOMME, Ancien Élève de l'École Polytechnique. Paris: Gauthier, Villars et Fils, Quai des Grands-Augustins 55. G. Masson, Éditeur, Libraire de l'Académie de Médecine, Boulevard St. Germain 120. London: H. Grevel and Co., King Street, Covent Garden. 1894. 3 francs.

SMALL 8vo volume, in cardboard cover, and containing 187 pages of subject-matter, a bibliographic list, and a table of contents.

In the text the following subjects are treated of:—I. Fixation of Colouring Matters. II. Mordants. III. Fixation of Mordants. IV. Garancine and its Artificial Substitutes. V. Indigo. VI. Aniline Black. VII. Albumin Colours. VIII. Aniline Colours. IX. Azo Colours. X. Reserves and Discharges (*enlevages*). XI. Alterations of the Fibres of Tissues. XII. Fastness of Colour.

**GRUNDRISS DER PHARMACOGNOSIE.** Von F. A. FLÜCKIGER. Zweite, mit Berücksichtigung technisch wichtiger Pflanzen bearbeitete Auflage. Berlin: R. Gaertner's Verlagshandlung. Hermann Heyfelder, Schönebergerstr. 26, S.W. London: H. Grevel and Co., King Street, Covent Garden. 7s. ; or bound, 8s.

OCTAVO volume, bound strongly in cloth, and containing Preface, Table of Contents, subject-matter covering 309 pages, and an Alphabetical Index. The principal division of the subject-matter is into two branches. I. Drugs obtained from the Vegetable Kingdom; and II. Those derived from the Animal Kingdom. The first receive a thoroughly scientific and complete classification in the table of contents, according to the natural orders to which they belong. A second table of contents then follows, in which the matters are classified according to their chemical and physical properties, as is usually done. *First Class.* Vegetable Matters without Organic Structure. I. Gums and Gummy Matters. II. Sweet Substances. III. Resin mixed with Gum. IV. Resin with Etheral Oils and Gums. V. Resin with Considerable Amounts of Etheral Oils. VI. Resins. VII. Balsams. VIII. Etheral Oils. IX. Fats and Waxes. X. Milk Juices and their Constituents. XI. Extracts and Colours. *Second Class.* Organic Matters of the Vegetable Kingdom. XII. Powders. XIII. Galls. XIV. Non-powder Vegetable Organs. Animal Matters and Products. A. Entirely Animal. B. Parts of Animals. C. Products from Animals.

## Trade Report.

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

(From the Board of Trade Journal.)

#### VICTORIA.

##### Tariff Changes.

By an Act assented to on the 13th October, the specific duties on sugar of all kinds are doubled. It is provided that, dating from the 14th September 1893, a rebate of 3s. per hundredweight was to be allowed to manufacturers on all sugar used in the manufacture of ale, beer, cordials, syrups, jam, jellies, and preserves, and on all sugar and glucose used in the manufacture of confectionery, such rebate to be allowed only to licensed or registered manufacturers under regulations to be approved by the Governor in Council, and in respect of sugar and glucose upon which the increased duty under this Act has been paid. The following are the new duties leviable upon sugar:—

Sugar, not being the produce of sugar-cane, 12s. per cwt.  
 Sugar, the produce of sugar-cane, 6s. per cwt.  
 Sugar, the produce of sugar-cane, and refined in Victoria in a bonded warehouse, 3s. per cwt.  
 Glucose, 6s. per cwt.  
 Molasses, refined, 6s. per cwt.  
 Molasses, refined, in bond, 4s. per cwt.  
 Molasses, unrefined, 2s. per cwt.  
 Candy, 4d. per lb.

#### THE ALCOHOLIC STRENGTH OF IMPORTED SPIRITS.

The West India Committee of Billiter House, London, E.C., having requested that a statement should be inserted in the *Board of Trade Journal* of the method adopted in the Customs department of ascertaining the true alcoholic strength of spirits to which sweetening, colouring, or other matter interfering with the free action of the hydrometer has been added, the Board of Customs state that they have found that the correct strength of spirit, such as rum, in which colouring or sweetening matter is present, cannot be ascertained by Sykes's hydrometer without first distilling the spirit, and that, under the law as it now stands, the Customs resort to distillation in the first instance for the purpose of ascertaining the true alcoholic strength of such spirits by Sykes's hydrometer.

#### THE NEW SPIRIT DUTIES.

The increased spirit duties proposed by the Chancellor of the Exchequer in his Budget Bill came in force on April 1<sup>st</sup>. Should the Bill fail to pass and the duty remain unchanged, any excess paid will be returned by the Government. The following resolution was adopted by the House of Commons on Monday night, and went into immediate effect:—That, in addition to the duties of Customs now payable on spirits, there shall be charged and paid the duties following (that is to say):—

	£	s.	d.
For every gallon computed at proof of spirits of any description except perfumed spirits.....	0	0	6
For every gallon of perfumed spirits.....	0	0	10
For every gallon of liquors, cordials, mixtures, and other preparations entered in such a manner as to indicate that the strength is not to be tested.....	0	0	8

And the duties of Customs on the articles hereinafter mentioned, being articles of which spirits are a part or ingredient, shall be proportionally increased, and shall be as follows:—

	The lb.	£	s.	d.
Chloral hydrate.....	The lb.	0	1	4
Chloroform.....	"	0	3	3
Collodion.....	The gallon	1	6	3
Ether acetic.....	The lb.	0	1	11
Ether butyric.....	The gallon	0	16	5
Ether sulphuric.....	"	1	7	5
Ethyl iodide of.....	"	0	14	3

The excise duty on spirits distilled in the United Kingdom is increased by a separate resolution.

The advance on chloral hydrate and acetic ether amounts to 1d. per lb.: on chloroform, to 2d. per lb.; on iodide of ethyl, to 8d. per gallon; on butyric ether, to 9d. per gallon; and on sulphuric ether and collodion, to 1s. 3d. per gallon. —*Chemist and Druggist.*

### EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

#### BALATA PRODUCTION IN SURINAM.

In his report on the trade and commerce of Surinam during 1893, Mr. E. W. Smith-Delaconr, Her Majesty's Consul at Paramaribo, gives the following particulars of the production of balata in Surinam:—

One of the most valuable forest products of Surinam, and which has been considerably exported, is the caoutchouc-like substance known as balata. Balata gum holds the first place among the substitutes for gutta-percha. It is obtained from the minussops balata. The balata gum combines in some degree the elasticity of caoutchouc with the ductility of gutta-percha, freely softening and becoming plastic, and being easily moulded like gutta-percha. The tree which produces this is widely distributed over the colony, even in parts so far in the interior as to have been hardly visited as yet; and the industry, now that experience has taught how it can be carried on without the wasteful extermination of the tree, should have a positive future value. Better means of access and of working are required before the expenses of production form the proper proportion of the value produced.

Balata is principally exported to the United States of America, small consignments also reaching Holland and Great Britain.

The total production in 1893 was estimated at 113,018 lb., valued at 6,347*l.*, as compared with 265,976 lb., valued at 15,070*l.* in 1892, showing a decrease in quantity of 152,958*l.*, and in value of 8,723*l.*—(*No.* 1329, *Foreign Office Annual Series.*)

#### PRODUCTION OF BANANA-MEAL IN SURINAM.

Mr. E. W. S. Delaconr, Her Majesty's Consul at Paramaribo, in a report to the Foreign Office, dated the 15th February, states that a company has lately been established at plantation "Susannasdaal" in the colony of Dutch Guiana, for the purpose of reducing into meal the large quantities of bananas and plantains grown on cocoa estates in the district. The banana trees are originally planted as shade for the young cocoa trees, and to such an extent that much of the fruit is wasted. Machinery and plant have been imported to the value of about 2,000*l.*, and a small amount of meal has already been produced and sent to Holland.

The company anticipate that a large quantity of banana meal for bread-making will be sold to the estates on which British Indian immigrants are engaged.

Mr. Delaconr encloses the following analysis of the Surinam banana:—

Analysis of banana-meal:—	
Moisture.....	11.44
Ether extract.....	0.30
Alcohol extract (80 per cent.).....	5.94
(of which, reducing sugar 1.18 per cent.)	
(of which, saccharose 0.08 per cent.)	
Cold water extract.....	5.33
Fibre.....	1.11
Ash.....	3.04
Starch.....	59.35
Albuminoids.....	5.25
Digestible fibre and other undeterminable carbohydrates	8.15
Total.....	100.00

From this it will be seen that the food value of the meal is minute, and should be compared rather to the potato than to a cereal.

In the face of this analysis, it is difficult to believe, says Mr. Delacour, that the banana-meal enterprise will prove remunerative, especially when it is remembered that so soon as the cocoa trees are sufficiently strong to bear the sun, the cultivation of the banana tree as shade will be no longer necessary, and the supply will consequently become limited. — *Board of Trade Journal*.

#### THE UNITED STATES DWYWOOD INDUSTRY.

*U.S. Consular Reports. March 1894, 438—450.*

The report includes statements with regard to the dyewood industries of Haiti, Mexico, Colombia, and Jamaica, compiled by the United States Consular officers in those places.

##### *Haiti.*

The port of Cape Haitien has for several years exported more logwood than any other port in the world. A notable decrease in the export of logwood has taken place during the last few years. A maximum was reached in 1881, when 157,322,650 pounds were exported, while in 1892 the amount was only 39,766,320 pounds. The logwood-producing forests are being rapidly destroyed, and, should the present rate of destruction continue, Haiti will cease to export logwood in 15 years, although much remains at present in districts remote from public roads.

##### *Mexico.*

Mexico produces many dyewoods, the best known being logwood, brazil wood, and fustic. These grow in profusion along the coast, and more sparingly in the interior. The principal markets are England, United States, Germany, France, Spain, and Russia. The export of 'Palo Moral' or fustic amounts to 9,000,000 kilos, a year. Notwithstanding the reckless way in which the trees are cut down, the supply is said to be almost inexhaustible. The Government has placed an export duty on logwood of 1.50 dol., and on fustic of 75 cents per 1,000 kilos.

In the district of La Paz are found two little-investigated dyewoods known as "lombay" and "torote," which yield red colonies.

##### *Colombia.*

The dyewood industry in Panama is of recent growth. Fustic is the only dyewood exported, and in 1893 the solitary shipper of this wood sent 560,000 lb. to Liverpool and Havre. From Colon, about 4,000 to 5,000 tons are annually exported.

##### *Jamaica.*

*Logwood* grows in the low-lying parts of the island. Considerable care is bestowed on its cultivation, and the demand for it is steady. The amount exported in 1893 (an average year) was 92,621 tons, of which 37,444 tons went to the United Kingdom.

*Fustic* is obtained in the mountainous districts. The total export during 1893 was 6,461 tons, of which 3,016 tons went to the United Kingdom. The demand at present is very slight. There is no probability of the supply being exhausted.

*Green-Heart Ebony.*—There is very little demand for this wood, only 146 tons being exported in 1893.

There is in process or erection at Spanish Town, in this island, a plant for the extraction of dye from these woods; but it is not believed that, even if successfully operated, it will diminish the exportation of the dyewoods.—R. B. B.

#### GENERAL TRADE NOTES.

##### THE MINERAL INDUSTRY OF GREECE.

The accompanying table of production shows clearly the bad effects which the fall in prices of the chief metals has had upon the mineral industry of Greece. The English coal strike also contributed to this by increasing the freight charges. The chief sufferers from low prices were the zinc producers and the lead works of Laurium, while the increase in freight affected the iron ore mines of Laurium and of Seriphos. The high exchange of gold, which stood at 160 per cent. (one franc, gold = 1.60 drachme), somewhat lessened the bad effect of low prices.

One of the chief smelting works, Laurium, treated exclusively the old slags and dump-heaps. The slags are nearly all worked out, and of the dump-heaps only the poorer portions remain, viz., those containing 3—4 per cent. lead, and 70—100 grms. silver per ton. Concentrating works capable of treating 1,000 tons of raw material per day were completed during 1893. There were about 6,700 tons produced from these old dumps during the year, the rest of the lead or a little more than half being obtained from new workings, from which only the purest galena was extracted. The average silver contents of the lead was about 2,000 grms. per ton. Since their erection to the end of 1893, the Laurium works have smelted 283,800 metric tons of lead, to the value of 31,860,000 dol.

The Laurium mines have also produced considerable quantities of zinc ore. Since 1875 to the end of 1893 there have been exported from these mines 555,000 metric tons of calcined calamine, of the value of 12,265,000 dol. There has been a gradual decrease in the production of zinc ores in the last few years, due to the exhaustion of old workings, but there exist several deposits which, only require to be properly developed to yield large quantities.

Since 1881, manganiferous iron ore, containing 33—40 per cent. iron, and 12—18 manganese, which (if it contained 1 to 6 per cent. lead) had only been used as flux, has been exported. It is estimated that altogether about 200,000 tons have been consumed as flux, while to the end of 1893, 1,065,880 tons of the value of 2,679,500 dol. have been exported.

*Iron Ores of Seriphos.*—The iron mines of Seriphos are very ancient, the island being used by the Romans as a colony for convicts, who were made to work the mines. Exploitation was commenced in 1870, but there were only 42,000 tons produced to the year 1880. Since then a French company has operated the mines, and has exported the following quantities in tons of 2,000 lb. :—

Year.	Total Exports.	Exports to United States.
1881	5,159	.. ..
1882	6,250	.. ..
1883	28,109	4,800
1884	21,645	13,185
1885	7,354	3,900
1886	32,640	24,300
1887	51,215	19,380
1888	18,170	6,950
1889	37,759	18,775
1890	89,470	36,120
1891	75,750	19,580
1892	112,445	41,440
1893	67,070	.. ..
Total .....	554,135	217,170

The hematite ores contain: 46—48 per cent. iron, 2·5—4 per cent. silica, and 0·02 per cent. phosphorus; limonite ores: 50—53 per cent. iron, 1—8 per cent. silica, and 0·035—0·01 per cent. phosphorus. Good, natural ports allow large steamers to load rapidly.

*Silver Ore of Milo.*—Silver was discovered in Milo and in the neighbouring isles of Kinolo and Polino about 10 years ago. In 1890 the Government prevented a company, having a concession to work lead-silver mines, from further exploiting these silver deposits, and ordered an examination by Government engineers, which is briefly resumed in the following:—

In these islands there exist in several places ventilar masses of heavy spar, intermixed clay, surrounded by clay and decomposed trachyte. These masses are embedded in fresh trachyte. The heavy spar, clay, and trachyte carry silver. It is estimated that there is no less than 10,000,000 tons of this ore. The silver is unevenly distributed, and the ore is not rich, averaging about 164 grains per ton (5½ oz.). Some assays have given as much as 4,000 grms. per ton.

The engineers being of the opinion that large quantities of ore averaging 250 grms. per ton could be profitably worked, the Government in February 1893 offered to grant a lease to the highest bidder to mine 150,000 tons within 15 years. It was easy to foretell that under these conditions and with the low price of silver no one would undertake to mine 8-oz. ore.

*Magnesite.*—This mineral holds an important position. Its occurrence in nature as magnesium carbonate appears to be rather limited, and nowhere else can it be found in such workable quantities as in Greece. The composition of the ore is as follows:—Magnesium carbonate, 96—98 per cent.; silica, 1—1·5 per cent.; alumina, 0·5—2 per cent.; calcium carbonate, 0·5—1·5 per cent.; iron carbonate, 0·3—0·5 per cent. The manufacture of bricks from burnt

magnesite was first carried out in Styria, Austria, but for some years past there has been an establishment at Eubœa. After many experiments and difficulties on account of the high temperature necessary (above the melting point of platinum) to burn the magnesite to a terrefactive substance, they succeeded about the middle of 1893 in carrying on the operations continuously. The maximum compressibility of magnesite is at 1,800° C., with a diminution of 80 per cent. in volume. The "Chamotteskine" and "Dinas bricks" used in the construction of the furnace (Hoffmann's circular) were unable to withstand the heat; now the magnesium bricks are used.

The specific gravity of these compact bricks is 2·64, and their composition is: Magnesium carbonate, 93·25—96·25 per cent.; calcium carbonate, 1·5—3·00 per cent.; silica, 1·5—2·5 per cent.; iron oxide and alumina, 0·75—1·25 per cent.

Emery is found in several of the Greek islands, but chiefly in Naxos, where it has been exploited for a long while, and occurs in boulders in crystalline limestone of the Archæan age. These mines could very easily compete against those of Asia Minor, situated between Smyrna and Ephesus, as they are purer, containing, before undergoing any cleaning, 68—70 per cent. corundum against 65 per cent. of the Smyrna deposits. The old method of fire-setting is still used on account of the difficulty of boring, even with the hardest steel. The price of emery in blocks at the coast of Naxos is 65 francs per metric ton; loads of at least 300 tons can be obtained, but on account of the bad ports it is difficult and expensive to ship such large blocks. Between the years 1869—1886 the quantity of Naxos emery produced was 41,344 metric tons, valued at 1,809,220 dols. From 1869 to 1889, the Government supplied emery at the fixed price of 218·80 francs at Naxos. Since then the competition of the Smyrna mines has brought the price down, as stated above, to 65 francs.—*Engineering and Mining Journal.*

#### MINERAL PRODUCTION OF THE UNITED STATES IN 1893.

Product.	Customary Measures.	1892.		1893.	
		Customary Measures.	Metric Tons.	Customary Measures.	Metric Tons.
NON-METALLIC.					
Asbestos .....	Short Tons	100	91	120	109
Antimony ore .....	"	850	771	850	771
Asphaltum and asph. rock .....	"	47,040	42,673	34,944	31,701
Barytes (crude) .....	"	28,476	25,833	26,632	24,161
Bauxite .....	"	9,800	8,891	11,041	10,106
Borax .....	Pounds	12,538,196	5,687	8,609,090	3,946
Bromine .....	"	379,180	172	348,399	158
Cement, hydraulic .....	Bbbs., 300—100 lbs.	8,211,181	..	7,503,385	..
Cement, Portland .....	"	517,140	..	596,631	..
Coal, anthracite .....	Long Tons	46,859,495	47,352,696	48,044,834	48,818,356
Coal, bituminous .....	"	114,220,101	116,059,045	113,847,568	115,680,514
Coke .....	Short Tons	12,010,829	12,204,203	9,792,330	9,949,986
Cobalt, oxide .....	Pounds	8,600	Kilos. 3,900	3,893	Kilos. 1,766
Copperas .....	Short Tons	13,250	12,021	16,000	14,515
Corundum .....	"	1,594	1,364	1,747	1,583
Chrome ore .....	Long Tons	1,350	1,677	1,620	1,646
Feldspar .....	"	16,000	16,258	17,000	17,274
Flint .....	"	37,000	37,596	38,000	38,612
Fluorspar .....	Short Tons	9,000	8,165	9,700	8,800
Grind-stones .....	"	..	..	45,550	41,350



## MINERAL PRODUCTION OF THE UNITED STATES IN 1893—continued.

Product.	Customary Measures.	1892.		1893.	
		Customary Measures.	Metric Tons.	Customary Measures.	Metric Tons.
Infusorial earth and tripoli .....	Short Tons	1,323	1,200	1,769	1,550
Limestone, for iron flux .....	Long Tons	4,500,600	4,333,416	4,551,900	4,424,000
Magnesite .....	Short Tons	1,402	1,272	1,143	1,047
Manganese ore .....	Long Tons	19,117	19,425	9,150	9,297
Maris .....	Short Tons	125,000	113,400	110,000	99,792
Mica .....	Lbs.	75,000	34	75,000	34
Onyx .....	Cubic Feet	3,500	..	2,475	..
Ozokerite (refined) .....	Lbs.	130,000	59	None	None
Petroleum .....	Bbls., 42 galls.	50,512,136	7,006,982	50,319,228	6,978,400
Phosphate rock .....	Long Tons	902,723	917,257	983,340	999,174
Plumbago (refined) .....	Lbs.	1,228,363	634	896,603	405
Plumbago (crude) .....	Short Tons	900	816	1,500	1,391
Pyrites .....	Long Tons	106,250	109,957	95,000	96,526
Salt .....	Bbls., 280 lbs.	11,784,954	1,542,133	11,435,487	1,452,388
Slate (for roofing) .....	In Squares	..	..	871,500	..
Soapstone .....	Short Tons.	23,268	21,054	20,100	18,235
Soda, natural .....	"	3,300	2,904	2,500	2,230
Sulphur .....	"	1,825	1,656	1,344	1,219
Talc (fibrous) .....	"	41,925	38,034	36,500	33,117
Venetian red .....	"	4,205	3,815	3,820	3,475
METALLIC.					
Antimony .....	"	260	181	350	318
Copper .....	Pounds.	325,500,000	117,047	322,585,500	116,324
Gold .....	Troy Ounces.	1,596,375	Kilos. 49,652	1,739,700	Kilos. 54,110
Pig iron .....	Long Tons.	8,977,869	9,122,413	7,043,384	7,156,782
Lead .....	Short Tons.	297,430	186,618	193,928	175,931
Nickel .....	Pounds.	96,160	Kilos. 13,614	25,808	Kilos. 11,745
Quicksilver .....	Flasks, 76½ lbs.	27,963	974	39,164	1,046
Silver .....	Troy Oz.	65,000,000	Kilos. 2,022,195	60,500,000	Kilos. 1,881,732
Spiegeleisen and ferro-manganese .....	Long Tons.	17,5131	182,015	81,118	82,424
Tin .....	Pounds.	113,400	65	None	None
Zinc .....	Short Tons.	84,982	76,279	76,255	69,178

## RUBBER PRODUCTION IN ASSAM.

According to the *Calcutta Englishman* for the 7th February, rubber is the most important of the minor forest products of Assam. It has yielded a considerable annual revenue to the Forest Department, obtained through the licensing of contractors, or *mahaldars*, who purchase the sole right of tapping trees, and practically within certain limits a monopoly in the foreign imported rubber trade. The revenue, however, has not been regarded as satisfactory during recent years, and owing to the illicit tapping operations carried on by the hill tribes, and the great difficulty of adequately protecting the Government rubber forests, it was decided in 1892 to discontinue the mahal system. In its place, a duty of 12 rupees per maund was imposed on all rubber, whether of home or foreign origin, obtained from Government forests or imported across the border, and this change promises, in the opinion of the conservator, to work very satisfactorily. There was some difficulty at first in explaining the new order of things to

the traders interested in rubber, and the exports during the first few months consequently showed a decrease as compared with those under the old system. There is every indication, however, that the trade is rapidly recovering itself.

## CHANGES IN U.S. PATENT OFFICE RULES.

In some recent changes in the "Rules of Practice" for the United States Patent Office, Rule 64 has been repealed. It read as follows: "The first step in the examination of an application will be to determine whether it is, in all respects, in proper form. If, however, the objections as to form are not vital, the examiner shall proceed to the consideration of the application on its merits, and in such case he must, if possible, in his first letter to the applicant, state all his objections, whether formal or otherwise, and until the formal objections are disposed of, further action will not be taken upon the merits without the order of the Commissioner." Instead of this the following rule has

been adopted: "Where the specifications and claims are such that the invention may be understood, the examination of a complete application and the action thereon will be directed throughout to the merits, but in each letter the examiner shall state or refer to all his objections. Only in cases presenting patentable substance will requirements in matters of form be insisted upon." The meaning of this seems to be that while an examiner may still specify in incorrectness in form as one of the reasons for rejecting an application, specifications or drawings will not be sent back for correction unless the examiner believes, after inspection, that there is some patentable point in the application.—*Engineering and Mining Journal*.

#### JOHANNESBURG SULPHURIC ACID WORKS.

The sulphuric acid manufactory recently erected near Johannesburg by a party of English capitalists, at a cost for plant of 55,000*l.*, is capable of producing 2,000 tons of acid per annum. The sulphur is obtained from pyritic

concentrates purchased from the mining companies. All the sulphur but 1 per cent. is extracted, and the oxidised ore is then sent on to extracting works for the recovery of its gold contents. The demand for sulphuric acid is increasing daily, and its price per lb., when taken in large quantities, as is done by the chlorination works, is less than that of the imported acid without the duty, which is 1*d.* per lb.—*Chemist and Druggist*.

#### CHEMICAL MANURES IN THE UNITED STATES.

In the United States the manufacture and consumption of chemical fertilisers has increased rapidly ever since the opening of the South Carolina deposits; fully one-half of the product is consumed in the cotton States, but as the rich soils of the north and west are gradually being exhausted, the use of fertilisers in those sections is increasing. The following tables show the production and consumption of fertilisers in the United States since 1888:—

	1888.	1889.	1890.	1891.	1892.	1893.
	Tons.	Tons.	Tons.	Tons.	Tons.	Tons.
Alabama.....	45,000	43,000	40,000	55,000	40,000	45,000
Florida.....	15,000	18,000	20,000	25,000	20,000	35,000
Georgia.....	170,000	185,000	200,000	230,000	175,000	280,000
South Carolina.....	115,000	120,000	125,000	130,000	110,000	115,000
North Carolina.....	90,000	95,500	95,000	105,000	90,000	90,000
Tennessee.....	28,000	20,000	30,000	35,000	30,000	35,000
Kentucky.....	15,000	18,000	15,000	20,000	15,000	20,000
Mississippi.....	12,000	8,500	12,500	15,000	10,000	15,000
Arkansas.....	7,500	8,600	10,000	10,000	10,000	15,000
Louisiana.....	12,000	9,000	10,000	15,000	10,000	25,000
Texas.....	10,000	10,000	12,500	10,000	5,000	10,000
Virginia.....	60,000	65,000	70,000	80,000	55,000	50,000
West Virginia.....	30,000	25,000	25,000	30,000	25,000	25,000
New England States.....	83,000	110,000	105,000	120,000	100,000	105,000
Western States.....	70,000	80,000	75,000	80,000	75,000	80,000
Middle States.....	210,000	370,000	350,000	300,000	305,000	285,000
Total.....	975,500	1,190,000	1,195,000	1,340,000	1,070,000	1,225,000
Production.....	980,000	1,230,000	1,250,000	1,320,000	1,365,000	1,350,000

Prior to 1888 the estimated production was 1,020,000 tons in 1885; 1,099,500 tons in 1886; and 975,000 tons in 1887. In 1862 Great Britain manufactured 200,000 tons of chemical manures, and in 1890 800,000 tons. The production for 1893 is estimated at 850,000 tons, about one-half being consumed in the United Kingdom and the remainder exported.

The world's supply of crude phosphate and guano as given by the best official sources is as follows:—South Carolina, 560,000 tons; Florida, 425,000 tons; Belgium, 400,000 tons; France, 400,000 tons; West Indies, guano, 30,000 tons; South America, guano, 45,000 tons; Russia, 70,000 tons; Germany, 50,000 tons; Spain, 40,000 tons; England, 30,000 tons; Canada, 15,000 tons; Norway, 20,000 tons; and North Carolina, 2,000 tons; total, 2,087,000.

The countries of Europe are estimated to have produced during 1893 the following amounts of acid phosphates:—France, 440,000 tons; Belgium, 275,000 tons; Germany, 550,000 tons; Holland, Sweden, and Norway, 150,000; Italy and Switzerland, 100,000; other countries, 100,000.

#### THE VALUATION OF MANURES.

A very useful guide for purchasers of manures and feeding stuffs has just been issued by the Highland and

Agricultural Society, in the shape of a scale of unit values to be used in determining a fair price, full instructions and illustrations of how to make the necessary valuations being appended. The manures are classified as follows:—

**Fish Guano.**—Finely ground, and containing not more than 3 per cent. oil. **Frey-Bentos Guano.**—(a.) Meat meal, free from horn, yielding over 11 per cent. nitrogen. (b.) Mixed scrap, yielding 6 to 7 per cent. nitrogen and 30 to 40 per cent. phosphates. **Bone Meal.**—(a.) 90 per cent. passing  $\frac{1}{8}$ -in. sieve. (b.) Coarser. Genuine bone meal contains 45 per cent. to 55 per cent. phosphates, and from  $4\frac{1}{2}$  per cent. to  $5\frac{1}{2}$  per cent. ammonia. The better qualities contain little or no fat. **Steamed Bone Flour.**—Ground to flour and containing about 60 per cent. phosphates and about 2 per cent. nitrogen. **Dissolved Bones.**—Must be pure, i.e., containing nothing but natural bones and sulphuric acid. **Dissolved Compounds.**—Including 'dissolved bone manures' and all special manures consisting of ingredients mixed together and dissolved as a whole. The 'average' units should be used in valuing ordinary well-made dissolved compounds. If the manure is of superior manufacture and of high concentration, the units used should be above the average; if of inferior manufacture, or of low concentration, the units used should be below average. In valuing such manures for associations the

units employed must be specified. Mixtures.—To be valued according to the unit values (as given below) of the ingredients of which they are guaranteed and also found to be composed, with an addition of from 5 to 10 per cent., according to the fineness of their manufacture. Thomas-Slag and Ground Phosphates.—Fineness of grinding is of paramount importance. The coarsest kind used should be so finely ground that 80 per cent. passes through a sieve of 10 000 holes per square inch.

#### Unit Values for Season, 1894.

Items to be Valued.	Guanos.		Scrap Manures.			
	Ichaboe.	Peruvian (Riddled).	Fish Guano.		Fry-Porter's Guano.	
	Genuine.	Genuine.			a.	b.
Classes.....	s. d.	s. d.	s. d.		s. d.	s. d.
Phosphates dissolved.....	..	..	..		..	..
„ undissolved.....	2 0	2 0	1 4		1 6	1 4
Nitrogen.....	20 4	24 0	13 6		14 0	12 0
Potash.....	..	3 6	..		..	..
Prices per ton, { From.....	250 0	225 0	130 0		150 0	120 0
March 1893. { To.....	270 0	290 0	150 0		180 0	140 0

Items to be Valued.	Bone Meal.		Steamed Bone Flour.	Dissolved or Vitriolated Bones.	Super-phosphates.	Dissolved Compounds.			
	a.	b.				Average.			
	s. d.	s. d.	s. d.	s. d.	s. d.	From	s. d.	s. d.	s. d.
Phosphates dissolved.....	..	..	..	3 0	1 11	2 0 to 3 0	2 8	..	..
„ undissolved.....	1 4	1 3	1 6	1 6	..	1 3 „ 1 9	1 6	..	..
Nitrogen.....	12 6	11 6	13 0	14 0	..	12 0 „ 17 0	13 6	..	..
Potash.....	..	..	..	..	..	3 4 „ 3 8	3 6	..	..
Prices per ton, { From.....	117 6	112 6	110 0	105 0	29 „ = 59 0	..	..	..	..
March 1893. { To.....	130 0	115 0	120 0	120 0	36 „ = 75 0	..	..	..	..

—Chemical Trade Journal.

#### THE OUTPUT OF PLATINUM.

The total platinum output in the Ural mines in 1893 was 11,196 lb., against 10,038 lb. in 1892. The average annual output for the last 10 years has been 7,848 lb., that of 1893 being the greatest quantity ever obtained.—*Chemist and Druggist*.

#### THE COPPER ORES OF PERSIA.

A. F. Stahl. *Chem. Zeit.* 18, 3—4.

Up to the last 200 years the copper used in Persia was all produced in the country. Only the richer oxidised ores were smelted, ores of 3 to 5 per cent. being non-visible, were left by the miners. The old slags contain 1 to 3 per cent. of copper. The present production is uncertain, but the royalties paid to the Government show it to be considerable. There is little prospect of any extensive works being established, as, excepting near the Caspian, where, on the northern slopes of the Elburs mountains, wood and coal are available for fuel, or on the Persian Gulf (in neither of which regions is there sufficient evidence of important deposits), the cost of carriage would be prohibitive.

Particulars are given of many deposits visited by the author during the last three years.

The district of Anarek is rich in minerals. It is composed of recent limestones resting on ancient eruptive rocks and crystalline schists which have been broken

through by trachytic masses of tertiary age. Water and fuel are scarce, but copper and lead are smelted with charcoal made from the bushes found in the desert. Mining, together with camel-breeding, has supported a population of some 2,500 people, probably for centuries.

To the east of Senman, about 18 kilom. from the petroleum wells, carbonate and oxide of copper occur as an impregnation in clay slate, the mineralised stone assaying 5 per cent. Should the borings now in progress (for petroleum) succeed, the deposits would certainly be worth working, as sheet copper could be used for the transport of the oil.

About 15 kilom. to the east of Berta Agere, at the foot of the Sefid Kuk mountains, are two veins of copper pyrites from 3 to 4 in. in width, which the writer looks upon as payable deposits. The veins occur at the contact between porphyritic rock and jurassic limestone, and chalcopryite is associated with fluor- and calcspar. Coal is found in the vicinity, and communications both with the coast and the interior are comparatively good. In conclusion he says that though Persia appears to be rich in copper ores, the workable deposits are few and far between.—J. H. C.

## BOARD OF TRADE RETURNS.

## SUMMARY OF IMPORTS.

Articles.	Month ending 31st March.	
	1893.	1894.
	£	£
Metals.....	1,866,413	1,788,300
Chemicals and dyestuffs.....	694,670	813,996
Oils.....	696,935	618,050
Raw materials for non-textile industries.	2,450,455	2,450,049
Total value of all imports ....	34,059,485	35,341,037

## SUMMARY OF EXPORTS.

Articles.	Month ending 31st March.	
	1893.	1894.
	£	£
Metals (other than machinery) ....	2,810,024	2,241,893
Chemicals and medicines .....	917,165	827,841
Miscellaneous articles.....	2,809,766	2,468,560
Total value of all exports.....	19,432,904	18,698,903

## IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH ENDING 31ST MARCH.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Alkali..... Cwt.	5,182	7,126	4,904	6,285
Bark (tanners', &c.) ..	17,126	19,589	5,604	6,654
Brimstone..... "	39,327	28,652	9,384	6,916
Chemicals..... Value £	..	..	119,249	122,978
Cochineal ..... Cwt.	283	329	1,697	2,065
Cutch and gambier Tons	3,509	1,862	78,500	42,962
Dyes:—				
Aniline..... Value £	..	..	11,120	14,875
Alizarin ..... "	..	..	22,072	18,644
Other ..... "	..	..	1,821	2,555
Indigo ..... Cwt.	9,812	16,540	209,330	324,157
Nitrate of potash . "	15,578	35,741	14,107	33,001
Valonia ..... Tons	4,295	2,432	55,611	29,290
Other articles... Value £	..	..	161,961	203,678
Total value of chemicals ..	..	..	694,670	813,996

## IMPORTS OF OILS FOR MONTH ENDING 31ST MARCH.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Cocoa-nut..... Cwt.	31,968	26,015	41,545	32,020
Olive ..... Tuns	2,973	2,041	190,112	72,007
Palm ..... Cwt.	90,325	96,324	134,908	113,022
Petroleum ..... Gall.	11,159,708	14,008,835	195,916	216,877
Seed ..... Tons	3,713	2,202	91,248	55,828
Train, &c..... Tuns	1,397	1,877	31,958	35,245
Turpentine ..... Cwt.	9,931	6,799	11,366	6,463
Other articles .. Value £	..	..	79,882	86,588
Total value of oils...	..	..	696,935	618,050

## IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDING 31ST MARCH.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Bark, Peruvian .. Cwt.	8,124	6,027	16,354	10,517
Bristles..... Lb.	183,422	145,689	29,832	23,869
Caoutchouc..... Cwt.	22,111	20,503	268,877	206,732
Gum:—				
Arabic..... "	4,162	5,107	12,017	12,657
Lac, &c..... "	7,242	12,367	33,079	66,041
Gutta-percha .... "	4,170	7,295	24,801	84,890
Hides, raw:—				
Dry..... "	30,039	48,504	71,746	111,916
Wet..... "	43,472	37,487	92,531	75,896
Ivory..... "	849	936	39,516	41,338
Marrure:—				
Guano..... Tons	1,860	528	7,385	2,150
Bones..... "	6,221	7,182	27,171	34,173
Nitrate of soda... "	19,601	11,089	192,825	102,170
Phosphate of lime "	29,754	30,906	57,415	56,933
Paraffin..... Cwt.	58,344	69,820	69,357	69,288
Linon rags..... Tons	6,840	1,692	67,443	17,874
Esparto..... "	15,188	21,240	72,699	94,104
Pulp of wood .... "	15,800	23,142	89,633	114,547
Rosin..... Cwt.	89,752	77,234	22,923	14,459
Tallow and stearin "	53,703	72,779	81,654	94,273
Tar ..... Barrels	2,231	869	1,023	372
Wood:—				
Hewn ..... Loads	90,187	131,852	131,705	208,323
Sawn ..... "	50,589	94,613	123,034	236,572
Staves ..... "	3,536	5,722	15,969	31,364
Mahogany ..... Tons	4,451	7,090	38,671	54,967
Other articles.... Value £	..	..	862,765	684,651
Total value .....	..	..	2,450,455	2,450,049

Besides the above, drugs to the value of 76,297*l.* were imported, as against 82,744*l.* in March 1893.

## IMPORTS OF METALS FOR MONTH ENDING 31ST MARCH.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Copper:—			£	£
Ore..... Tons	8,841	7,430	87,399	71,029
Regulus..... "	9,263	6,558	224,952	149,747
Unwrought .... "	1,644	3,612	75,263	156,015
Iron:—				
Ore..... "	382,089	418,969	252,928	285,280
Bolt, bar, &c.... "	3,707	3,209	32,206	23,946
Steel, unwrought.. "	686	621	7,804	6,125
Lead, pig and sheet "	16,446	15,935	164,359	148,352
Pyrites..... "	60,125	60,784	106,368	102,422
Quicksilver..... Lb.	1,005,243	1,629,450	91,304	124,712
Silver ore..... Value £	..	..	262,891	204,270
Tin..... Cwt.	68,790	73,371	317,885	255,292
Zinc..... Tons	5,388	3,777	96,662	69,895
Other articles ... Value £	..	..	116,392	229,509
Total value of metals	..	..	1,866,413	1,788,300

## EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDING 31ST MARCH.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Gunpowder..... Lb.	676,000	1,128,100	15,267	19,724
Military stores.. Value £	..	..	57,141	136,491
Candles..... Lb.	2,050,300	1,552,900	37,235	27,296
Caoutchouc..... Value £	..	..	111,644	95,328
Cement..... Tons	39,277	36,411	67,830	60,633
Products of coal Value £	..	..	143,601	116,870
Earthenware ... "	..	..	192,535	115,950
Stoneware..... "	..	..	15,892	12,796
Glass:—				
Plate..... Sq. Ft.	97,024	81,158	5,285	4,961
Flint..... Cwt.	7,940	6,950	19,577	15,304
Bottles..... "	50,029	58,834	23,897	27,377
Other kinds.... "	17,832	10,958	15,101	8,236
Leather:—				
Unwrought .... "	14,449	12,023	137,483	113,712
Wrought ..... Value £	..	..	27,161	20,549
Seed oil..... Tons	6,684	5,901	152,912	123,904
Floorcloth ..... Sq. Yds.	1,877,000	1,762,400	71,965	65,896
Painters' materials Val. £	..	..	141,821	119,567
Paper..... Cwt.	75,644	89,433	126,892	136,318
Rags..... Tons	6,704	5,042	50,637	27,918
Soap..... Cwt.	50,841	50,552	57,901	55,537
Total value .....	..	..	2,899,766	2,496,560

## EXPORTS OF METALS (OTHER THAN MACHINERY) FOR MONTH ENDING 31ST MARCH.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Brass..... Cwt.	10,574	10,024	42,472	35,146
Copper:—			£	£
Unwrought..... "	68,996	26,605	169,423	58,955
Wrought..... "	35,564	26,482	102,104	73,642
Mixed metal .... "	30,990	34,637	74,586	76,413
Hardware..... Value £	..	..	199,937	154,416
Implements..... "	..	..	112,440	102,731
Iron and steel..... Tons	220,837	196,509	1,873,592	1,453,516
Lead..... "	3,288	2,714	41,160	33,005
Plated wares... Value £	..	..	26,457	20,006
Telegraph wires .. "	..	..	22,961	113,423
Tin..... Cwt.	8,929	11,262	43,653	41,513
Zinc..... "	25,571	13,970	21,332	9,794
Other articles .. Value £	..	..	81,407	69,354
Total value .....	..	..	2,810,624	2,241,893

## EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING 31ST MARCH.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Alkali..... Cwt.	688,709	458,281	218,319	128,096
Bleaching materials .. "	146,511	101,486	60,775	49,427
Chemical manures. Tons	39,523	35,758	268,521	266,071
Medicines..... Value £	..	..	86,040	84,871
Other articles ... "	..	..	283,519	307,476
Total value .....	..	..	917,165	827,841

## Monthly Patent List.

\* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### APPLICATIONS.

6551A. T. G. Hall. An appliance for breaking down crust in gas producers. Complete Specification. Filed March 19. Date claimed March 28, 1893.

5686. T. Lohnstein. An improved float for determining the absolute weight and specific gravity of solid and liquid bodies. Complete Specification. March 19.

5877. F. M. Robertson, W. S. Miller, and H. S. Fearon. Improvements in the process of and apparatus for evaporating or drying. March 21.

5925. G. Seagrave, J. H. Pickup, and L. Moritz. Improvements in apparatus for heating air or other gases. March 21.

6013. J. Alty. See Class XVII.

6176. J. E. Tolson and G. Tolson. A new or improved automatic hydro-extractor. March 27.

6462. K. C. Edmunds. An improved compound or mixture for freezing or chilling. March 31.

6495. E. Hermite, E. J. Paterson, and C. F. Cooper. A process for protecting iron pipes from corrosion by passage of electrolysed chloride solutions employed as disinfectants. March 31.

6953. A. G. Ogilvie. An improvement in kilns for burning lime or calcining other minerals. April 7.

7019. T. Oxley. Improvements connected with furnaces and relating to means for dealing with the products of combustion. April 9.

7027. L. Wagner and J. Marr. New or improved process and apparatus for purifying liquids. Complete Specification. April 9.

7033. F. E. Whitham. Improvements in depositing tanks for separating solids from liquids. April 9.

7235. D. McGill. An improved method of and means for the compression of air or other gases. April 11.

7305. J. G. Hall. An improved brewing-pan or copper for the boiling of worts used in breweries and distilleries, applicable to steam-boilers and other similar uses. April 12.

7318. T. Wheeler. An improvement in metallic thermometers and barometers. April 12.

7326. E. Hesketh and A. Marcet. Improvements in flasks or cylinders for containing gases under pressure or liquefaction or the like. April 12.

7388. E. G. Scott. Improvements in evaporating apparatus. April 13.

#### COMPLETE SPECIFICATIONS ACCEPTED.\*

1893.

8646. C. Huelser.—From A. Bloch. Process and appliance for mixing gases with liquids, especially carbonic acid, with water for baths and the like. April 4.

9147. J. Bouvier. Improvements in filters. April 18.

10,216. T. Ledward. An improved cooling apparatus for fluids. April 11.

1894.

2588. J. Nagel. Apparatus for distilling and sterilising water. April 11.

4094. S. C. Hauberg. Improvements in centrifugal liquid separators. April 11.

### II.—FUEL, GAS, AND LIGHT.

#### APPLICATIONS.

6152. H. Fourness. Improvements in the process of and apparatus for manufacturing water-oil gas. March 27.

6693. I. I. Gugenheimer. Improvements in heating gases for metallurgical furnaces. April 3.

6706. J. Parkes and J. Parkes. Improvements in gas-carburetting apparatus. April 4.

6737. J. Braunschield. Process for changing asbestos-fibre into an incandescent body for incandescent gas-light. April 4.

7019. T. Oxley. See Class I.

7179. J. Hodgkinson. A new and improved method of heating brine or similar substances. April 11.

7237. W. O. Felt. Improvements in the manufacture of gas. April 11.

7243. L. Parent. Improvements in the continuous manufacture of anhydrous metallurgic coke. April 11.

7372. M. Bernstein and A. Silbermann. Improvements in and relating to incandescent gas lighting. Complete Specification. April 13.

7482. S. A. Johnson. Improvements in the use or employment of hydrocarbon or other suitable oils, and in the method of burning same in firegrates, stoves, ranges, furnaces, or the like, for heating, cooking, and such like purposes. April 14.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1892.

23,497. E. Wardell and J. H. Evers. Improvements in apparatus for washing the gaseous products escaping from boiler and other furnaces, and for increasing the draught in such furnaces. March 28.

1893.

4037. H. Aitken. Improvements in and apparatus for obtaining cyanogen, cyanides, and other products from carbonaceous matter, or substances containing ammonia or nitrogen, or compounds thereof. April 4.

6515. E. Smith. Improvements for the consumption of smoke and waste gases in furnaces. April 4.

6551. T. G. Hall. A process and appliance for electrically treating gaseous and other bodies, a new compound gas made by such process, and apparatus for such producing and consuming said gaseous product. April 4.

7605. J. Smith. A retort and apparatus connected therewith for the purpose of converting coal-tar into gas for illumination and other purposes. April 11.

1894.

81. F. Kempster. An apparatus for the utilisation of mineral oils for heating purposes. April 11.

4719. E. Fleischhauer and M. Bernstein. Improvements in and relating to the manufacture of gas. April 18.

## IV.—COLOURING MATTERS AND DYES.

## APPLICATIONS.

5709. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of new colouring matters. March 19.

5711. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of colouring matters and materials therefor. March 19.

5712. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of new azo-colouring matters. March 19.

5722. J. V. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in the manufacture of colouring matters of the alkylated induline series. March 19.

5853. Brooke, Simpson, and Spiller, Limited, and A. G. Green. A new direct-dyeing yellow colour for cotton, and the process for its manufacture. March 21.

5870. J. Grime. A new or improved method of oxidising indigo blues and the like, and apparatus employed therefor. Complete Specification. March 21.

6004. F. Muhlert. Manufacture of yellow and orange colouring matters from  $\beta$ -resoreyllic acid. Complete Specification. March 22.

6197. B. Willeox.—From The Badische Anilin und Soda Fabrik, Germany. The manufacture and production of new azo-colouring matters. March 27.

6198. B. Willeox.—From F. von Heyden, Nachfolger, Germany. Improvements in the treatment or purification of commercial toluol-sulpho-amide. March 27.

6894. J. C. Mewburn.—From La Société Anonyme pour l'Exploitation des Procédés de Zebrowski, France. The manufacture of a new colouring matter particularly adapted for use in dyeing and printing. April 6.

7044. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of colouring matters of the induline series. April 9.

7046. C. D. Abel.—From A. Fischesser and Co., Germany. Manufacture of 1 naphthylamine 3, 6, 8 trisulpho acid from 1 nitronaphthalin 3, 8 disulpho acid. April 9.

7422. J. Baruch and A. Goldsobel. Improvements in and connected with the manufacture of colouring matters. April 14.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

9895. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of new sulpho acids and derivatives thereof. March 28.

9972. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of colouring matters and processes for dyeing and printing therewith. March 28.

11,556. A. Ashworth and J. Burger. Improvements relating to the production of colouring matters. April 18.

11,902. T. R. Shillito.—From J. R. Geigy. Production of azo colours. April 11.

1894.

3886. W. E. Heys.—From Sandoz and Co. Improvements in the production of colouring matters dyeing with mordants. April 11.

4629. H. Inray.—From The Basle Chemical Works. Manufacture of a new dioxynaphthalene monosulpho acid and of colouring matters therefrom. April 11.

4630. H. Inray. From The Basle Chemical Works. Manufacture of a new dioxynaphthalene monosulpho acid and of colouring matters therefrom. April 11.

## V.—TEXTILES, COTTON, WOOL, SILK, Etc.

## APPLICATIONS.

5718. H. Rous and G. N. West. A new or improved process for rendering textile materials waterproof and damproof. Complete Specification. March 19.

5952. J. Mackenzie. Improved means for the preparation of ramie fibre. March 22.

6032. F. W. Oliver. Improvement in treating cotton, linen, and silk fibres and threads with celluloid. March 22.

7093. J. Hargreaves. Improvements in the manufacture of ornamental woven fabrics. April 10.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

6007. A. E. Newman, R. E. Newman, and J. Newman. Improvements in the treatment of fibrous materials and in the means or apparatus employed therein. March 28.

7104. A. E. Newman, R. E. Newman, and J. Newman. Improvements in the treatment of flax and like fibrous material and in the means or apparatus employed therein. April 4.

22,808. J. C. Pennington and W. O. Allison. Improvements in or relating to the "retting" of fibres. April 18.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

## APPLICATIONS.

5951. S. Fisher, A. Mellor, and G. Wolfenden. Improvements in machinery or apparatus for cleaning and lustreing dyed or undyed yarns of silk, cotton, or other fibrous substances. March 22.

6054. H. Lange and R. Homberg. Improvements in or relating to bleaching, mordanting, dyeing, steaming, and analogous processes, and apparatus therefor. March 22.

6484. B. Thies. Dyeing apparatus for textile fibres. March 31.

6521. B. Cawthorn and J. P. Cornett. Improvements relating to apparatus used in the process of breaking and bleaching fibrous materials. April 2.

6673. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the process of obtaining coloured designs or patterns on indigo-dyed materials. April 3.

6803. A. Baummeister.—From P. Francois, France. An improved steaming or bleaching process for hemp or other textile materials and articles made therefrom. April 5.

6887. C. F. Cross and E. J. Bevan.—From C. Bealle, United States. A process for marking indelibly on paper, fabrics, and like receptive material. April 6.

7011. W. H. Hughes and S. F. Hughes. Improved process for scouring, cleansing, bleaching, and disinfecting wool and other fibres or fibrous materials. Complete Specification. April 9.



7133. W. McNaught. Improvements in or applicable to machinery for scouring and washing wool and other fibrous materials. April 10.

7135. A. Blanchon and A. Allegret. An improved process for printing indigo on fabric and yarn. April 10.

7197. E. Potter and Co., Limited, and A. Hunzinger. Improvements in printing upon calicoes or other woven fabrics and in apparatus employed therefor. April 11.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

10,327. J. Smith. Improvements in or connected with dyeing, tinting, sizing, and bleaching, or similarly operating upon textile fabrics or fibres and other materials. April 4.

15,531. G. A. J. Schott.—From E. Lauhe. Improvements in the method of bleaching cotton cloths and other materials, and in apparatus employed therein. April 18.

### VII.—ACIDS, ALKALIS, AND SALTS.

#### APPLICATIONS.

5808. J. D. Darling and H. C. Forrest. Improvements in producing nitric acid and metals from nitrates. Complete Specification. March 20.

5830. A. E. Hetherington and E. K. Muspratt. Improvements in the manufacture of ferrocyanide of potassium or of sodium from sulphocyanide. March 20.

5832. A. E. Hetherington, F. Hurter, and E. K. Muspratt. Improvements in the manufacture of cyanide of sodium or of potassium from their ferrocyanides. March 20.

5895. G. Webb, jun., and G. Webb, sen. Improvements in the manufacture of chlorine gas and caustic soda. March 21.

6426. T. Craney. Improved method and apparatus for manufacturing caustic soda. Complete Specification. March 30.

6445. C. R. Illingworth and D. Arthur. See Class XVIII. B.

6621. J. B. Readman. Improvements in obtaining cyanides and ferrocyanides. April 3.

6925. W. McDonnell Mackey and J. F. Hutcheson. Improvements in making potassic cyanide. April 7.

7179. J. Hodgkinson. See Class II.

7300. C. P. Shrewsbury, F. L. Marshall, and J. Cooper. Improvements in the method of and apparatus for obtaining sulphate of lead from galena. April 12.

7413. W. Garroway. Improvements in the manufacture of caustic soda and potash, and of nitric and sulphuric acids. Complete Specification. April 14.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

6044. L. O. Helmers. Improvements in obtaining sulphonic acids and sulphonic-like compounds free from inorganic salts from the neutralised product of the action of sulphuric acid upon mineral and similar oils, and particularly hydro-carbons artificially or naturally sulphurated. March 28.

7264. F. M. Lyte. Improvements in the production of chlorine and refined lead, and in the recovery of the silver the lead contains. April 11.

11,040. A. Brand. Improvements in the manufacture of chlorine. April 11.

1894.

4791. H. Wachter. A bleaching preparation or mixture applicable also for other purposes. April 11.

### VIII.—GLASS, POTTERY, AND EARTHENWARE.

#### APPLICATION.

6261. Chance Brothers and Co., Limited, and J. E. Scott. Improvements in tank furnaces used in the manufacture of glass. March 28.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

8758. W. W. Pilkington. Improvements in apparatus for forming molten glass into sheets. April 11.

15,411. M. Rosenthal and R. Walter. Improvements in the production of variously coloured designs, drawings, or pictures on porcelain, glass, or ceramic objects.

22,362. J. H. Bale. Wafer china mosaic. April 4.

### IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

#### APPLICATIONS.

6168. W. Horne. Improvements in the construction of fireproof buildings, and rendering existing buildings fireproof. March 27.

6552. J. H. Blakesley. Improvements in fireproof floors, roofs, girders, joists, and the like. April 2.

6662. W. F. F. Bevis. Improvements in and relating to building blocks. April 3.

6832. J. Kirby. Improvements in road pavings. April 5.

6890. G. S. Cory and J. R. Casbay. Improvements in paving for roads and streets. April 6.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

7211. G. Gin. Improvements in the manufacture of cement. April 11.

10,044. T. L. Banks. Improvements in fireproof floors, ceilings, and the like, partly applicable to other forms of floors, ceilings, and partitions. April 4.

10,045. T. L. Banks. Improvements in fireproof framing for partition and other walls. March 28.

12,227. T. L. Banks. Improvements in fireproof structures for ceilings, floors, and roofs. April 11.

1894.

3803. T. D. Harries. An improved artificial stone. April 4.

## X.—METALLURGY, MINING, ETC.

## APPLICATIONS.

3790. J. A. Mayo. Apparatus for separating metals from and purifying lead. March 20.

3808. J. D. Darling and H. C. Forrest. See Class VII.

3831. A. E. Hetherington, F. Hurter, and E. K. Muspratt. Improvements in and apparatus for the production of alloys of sodium or potassium with lead. March 20.

5934. W. Perkins. Improved means for extracting metals from their ores. March 21.

6693. I. I. Gugenheimer. See Class II.

6743. E. Bonchill. Improvements in an apparatus for, or connected with, puddling raw, cast, or pig iron. April 4.

6783. H. G. Richardson. Certain improvements in the manufacture of iron and steel and other metals, and in apparatus to be employed in the said manufacture. April 5.

6791. C. A. Allison.—From C. Bell and W. S. Bell, United States. Improvements in methods and apparatus for smelting or reducing aluminium ores. April 5.

6914. P. Manhes. Improvements relating to the manufacture of nickel and cobalt. Filed April 6. Date applied for December 9, 1893, being date of application in France.

6916. R. F. Hall. Improvements relating to the casting of metals. April 6.

6974. C. de E. Llatas. Improvements in the manufacture of steel. April 7.

6984. P. Manhes. Improvements relating to the treatment of nickel and cobalt ores. Filed April 7. Date applied for January 16, 1894, being date of application in France.

7380. S. Taylor. A new or improved solder for soldering aluminium and alloys of aluminium. April 13.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

6137. A. J. Smith. Improvements relating to the pickling of metals and alloys to remove oxide and other impurities from the surface thereof. April 4.

7264. F. M. Lyte. See Class VII.

7791. C. T. J. Oppermann. An improved process for the preparation of iron or steel plates for nickel or other plating. April 11.

10,367. O. G. Goodman. A process of leaving malleable or soft portions of steel articles in hardening and tempering. April 4.

10,463. H. J. Kirkman. Improvements in and relating to pickling iron and steel plates and other articles previous to tinning or galvanising. April 11.

10,699. E. Hammesfahr. Apparatus for cooling or hardening objects of metal, alloys, and the like between plates, moulds, dies, or rollers. March 28.

12,946. D. P. G. Matthews. Improvements in and connected with metallic moulds, and in the method of using such moulds for casting brass or bronze alloys. April 18.

21,352. L. W. Mallasee. An improved method of casting steel and apparatus therefor. April 11.

1894.

2527. E. Norton. Improvements in the art or process of manufacturing tin orterne plate. March 28.

2629. W. P. Thompson.—From J. J. C. Smith and E. C. Smith. Improvements in or appertaining to casting metals. April 11.

4980. W. P. Thompson.—From O. Nicolai. A new or improved solder for soldering aluminium, aluminium alloys, and other metals. April 18.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

## APPLICATIONS.

5721. C. Smith.—From G. Jorgensen, Norway. New or improved process and apparatus for the electrolytic decomposition of common salt. March 19.

6016. L. A. P. Liénard and H. E. A. Liénard. Improvements in the methods of effecting the electrolysis of saline solutions. Complete Specification. Filed March 22. Date applied for September 22, 1893, being date of application in France.

6241. F. A. Rhead and H. G. Coleman. A method of depositing and affixing metals by electricity upon a medium which is fastened upon ceramic objects by burning in a kiln. March 28.

6402. G. E. B. Pritchett, T. W. Pritchett, and A. G. Gold. Improvements in electric accumulators. March 30.

6497. E. Hermite, E. J. Paterson, and C. F. Cooper. Improvements in apparatus for electrolysing chloride solutions employed for disinfecting. March 31.

6917. H. Guthrie. Improvements in apparatus for electrolytical purposes. April 6.

6975. A. J. Smith and H. J. Wright. Improvements relating to the manufacture of plates or electrodes for electric accumulators or secondary batteries, and to machinery or apparatus therefor. April 7.

7094. W. J. S. Barber-Starkey. Improvements in and connected with secondary electric batteries. April 10.

7145. S. O. Cowper-Coles and Sir B. W. Walker, Bart. Improvements in or connected with the electro-deposition of metals or metallic compounds and the treatment of deposited metals or compounds. April 10.

7165. E. J. Clabbe and A. W. Sonthey. Improvements in secondary voltaic batteries. April 10.

7205. A. F. B. Gomess. A new or improved process for the electro-deposition of aluminium and aluminium alloys. April 11.

7206. A. F. B. Gomess. A new or improved process for the electro-deposition of aluminium and aluminium alloys. April 11.

7311. B. A. N. Dorez. Improvements relating to secondary batteries. April 12.

7467. A. J. Marquand and E. Hancock. Improvements in primary electric or secondary batteries. April 14.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

6000. B. H. Thwaite. Improved methods for effecting the electric fusion of steel and its alloys. March 28.

6751. E. Placet and J. Bonnet. Improvements in the extraction of chromium by electrolysis, applicable also to the production of chromium alloys. April 11.

6780. V. C. Doubleday.—From M. Sussmann. Improvements in secondary batteries. March 28.

7711. P. Schoop. Improvements in electrical accumulators or secondary batteries. April 18.

10,042. F. King. Improvements in and apparatus for the manufacture of plates, grids, or supports for secondary batteries. April 18.

11,122. A. E. Peyrusson. Improvements in secondary batteries or accumulators. April 18.

11,973. Siemens Brothers and Co., Lim., and E. F. A. Obach. Improvements in apparatus for electrolysis of water. April 4.

1894.

3710. A. J. Boulton.—From C. Cudell, I. Cudell, and M. Cudell. Improvements in galvanic batteries. April 18.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

### APPLICATIONS.

5821. F. J. Machalski. Improvements in apparatus for extracting fat and glue. Complete Specification. March 20.

7126. F. Walton. A process and apparatus for oxidising frying oils. April 10.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893

9315. W. N. Hartley and W. E. B. Blenkinsop. Improvements in or relating to the treatment of oils for refining, deacidifying, and boiling the same. April 18.

9882. G. Pommerhantz. Improved process of producing consistent and semi-consistent fats and oils without applying heat. March 28.

10,664. J. Weir and G. Weir. Improvements in separating oleaginous or fatty matters from boiler feed-water or other water. April 4.

12,817. E. Watel. A process of improving and preserving oils and fats. March 28.

## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

### APPLICATIONS.

5779. S. Banner. Improvements in or relating to treating copal for making gum copal varnish. March 20.

5856. H. P. Scott. Improvements in compositions or paints for coating iron, steel, or metal structures to preserve them from fouling and corrosion. March 21.

6002. J. E. Williams. A new or improved anti-salt paint. March 22.

7129. R. Matthews. Improvements in the manufacture of white lead. April 10.

7465. A. Angell. Improvements in the manufacture of oil paints and compositions from carbonised or calcined iron-stone or iron ore. April 14.

### COMPLETE SPECIFICATIONS ACCEPTED.

1892.

23,572. S. Z. de Ferranti and J. H. Noad. Improvements in the manufacture of white lead. March 28.

1893.

8368. R. Irvine. Improvements in the manufacture of paint. March 28.

11,727. W. C. Nangle. An improved paint or paint-like composition. April 18.

12,266. R. Hutchison. Improvements in treating or preparing gutta-percha and rubber for various purposes. April 18.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

### APPLICATIONS.

5821. F. J. Machalski. See Class XII.

7324. H. A. Leverett and G. J. B. Buckley. An improved process of rapidly making leather. April 12.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

10,445. J. Pullman and A. Pullman. Improvements in machinery or apparatus for treating skins in the manufacture of leather. April 4.

1894.

3615. E. H. Lewis. Improvement in the art or process of waterproofing leather and in the compounds employed in same. March 28.

3626. E. H. Lewis. Improvement in the art or process of waterproofing leather. March 28.

## XV.—AGRICULTURE AND MANURES.

### APPLICATION.

5692. W. Newton. Improvements in the manufacture of phosphatic and nitrogenous fertilisers. March 19.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

### APPLICATION.

6223. L. E. Asser. Improvements in the treatment of feculent matter used in the manufacture of glucose. March 27.

### COMPLETE SPECIFICATION ACCEPTED.

1894.

3782. E. Langen. Improvements in refining sugar. March 28.

## XVII.—BREWING, WINES, SPIRITS, Etc.

### APPLICATIONS.

6013. J. Ahy. Improvements in refrigerators for cooling wort or other liquids and apparatus connected therewith. March 22.

6293. A. D. Watson. Improvements in cooling yeast and in apparatus therefor. March 29.

7456. L. I. N. Norman. Improvements in the manufacture of brewing syrup. April 14.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

10,132. J. Pikhart. An improvement in the manufacture of beer. April 4.

10,263. K. Michel and M. Frank. Improved method of and apparatus for collecting the carbonic acid generated during the fermentation of beer or wine. April 11.

12,391. O. Oldham. An improved mixture for the manufacture of non-intoxicating beer and stout. April 11.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

## APPLICATIONS.

*A.—Chemistry of Foods.*

6010. H. H. Lake.—From F. Delangle, France. Improvements relating to the preservation of alimentary and other organic substances. March 22.

6106. W. F. Maclaren and T. B. Rawlins. Improvements in the manufacture of condensed milk. March 24.

6257. W. Flowers. Improvements relating to the admixture and treatment of certain ingredients for the production of a new article of food. March 28.

7009. A. Girling. Wafer table jelly. April 9.

7055. E. Markham. An improved process of preserving eggs. April 9.

*B.—Sanitary Chemistry.*

5792. R. C. Sayer. Improvements in sanitation. Complete Specification. March 20.

5819. J. Schmeiser and A. Billig. An improved process and apparatus for impregnating air with the essence of fir wood. March 20.

6049. J. B. Alliot. Improvements in apparatus for the treatment of offensive and infected matters. March 22.

6159. M. F. Purcell. Utilisation of sewage. March 27.

6318. H. S. Chapman. An improvement in the treatment of sewage discharging into the sea. March 29.

6445. C. R. Illingworth and D. Arthur. A new method of treating sewage and other liquid organic refuse for the extraction and fixation of ammonia. March 31.

7053. F. E. Whitham. Improvements in depositing tanks for separating solids from liquids. April 9.

7056. J. F. Foveaux. Treatment of sewage by evaporation, heating, and ventilating. April 9.

*C.—Disinfectants.*

6243. G. C. Kinloch. The use of picric acid as an insecticide, antiseptic, and disinfectant. March 28.

## COMPLETE SPECIFICATIONS ACCEPTED.

*A.—Chemistry of Foods.*

1893.

8726. H. Trillich. Improvements in or relating to the preparation of substitutes for coffee. April 11.

*B.—Sanitary Chemistry.*

1894.

4195. H. H. Lake.—From Seaburg and Johnson. Improvements relating to the burning of sulphur for disinfecting purposes. April 18.

*C.—Disinfectants.*

1893.

11,679. I. S. McDougall and J. T. McDougall. Improvements in disinfectants. April 18.

11,807. C. T. Kinzett. Improvements in sulphur candles and their holders. April 18.

11,944. I. S. McDougall and J. T. McDougall. Improvements in insecticides and anti-fungoids. April 18.

12,400. I. S. McDougall and J. T. McDougall. Improvements in fumigants. April 18.

## XIX.—PAPER, PASTEBOARD, Etc.

## APPLICATIONS.

6221. G. Fehrenbach and L. Prud'hou. An improved process for producing artificial whalebone and method of forming same into strips. Complete Specification. March 27.

6301. J. Craig. Improvement in the manufacture of paper. March 29.

6468. F. Duffett. An improvement in the manufacture of blotting and other absorbent and porous papers. March 31.

6522. J. P. Cornett. Improvements in or connected with the manufacture of pulp for paper-making. April 2.

7486. L. Munk. Improvements in the manufacture of paper and the like. April 14.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

## APPLICATIONS.

6033. A. Zimmermann.—From The Chemische Fabrik auf Actien vormals E. Schering, Germany. The manufacture of certain bodies suitable as remedies. March 22.

6190. F. Krafft and A. Roos. Manufacture of acid esters, phenol and other ethers by means of aromatic sulphonic acids. Complete Specification. March 27.

6800. L. Lederer. A new process of producing oxybenzylic alcohols and their homologues. April 5.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

10,701. J. Meikle, jun. Apparatus for extracting substances by volatile solvents. April 18.

12,942. H. Baum. Improvements in the manufacture of ortho-halogen-phenol and the production of pyro-catechine therefrom.

## XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.

## APPLICATIONS.

6256. J. R. Hueto. Improvements in photography called "photo-sculpture." March 28.

6690. E. J. Tobin and F. G. Benet. Improved actinic print and improved method of producing same. Complete Specification. April 3.

6740. F. W. T. Krohn. Improvements in emulsions for photographic printing purposes. April 4.

## XXII.—EXPLOSIVES, MATCHES, Etc.

11,502. E. Kubin. Improvements in or relating to explosives. April 11.

## APPLICATIONS.

5939. H. Priester. An improved compound for use in the manufacture of matches. Complete Specification. March 21.

6700. A. Price. An improved explosive and fuze-warmer. April 4.

7051. A. V. Newton.—From A. Nobel, France. Improvements in the preparation and utilisation of tempered explosives. April 9.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

8392. G. Kynoch and Co., Lim., and E. Jones. Improvements in cordite-loading machines. April 18.

## XXIII.—ANALYTICAL CHEMISTRY.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

7547. C. R. C. Tiehborne and C. J. Thompson. Improved apparatus for the determination of butter fat in milk. April 11.

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## NOTICES.

In accordance with the provisions of Rule 18 of the By-Laws, notice is hereby given that those Members of Council whose names are placed in italics in the annexed list, will retire from their respective offices at the forthcoming Annual General Meeting.

Dr. T. E. Thorpe, F.R.S., has been nominated to the office of President; and Mr. E. C. C. Stanford has been nominated Vice-President under Rule 11.

Mr. John Calderwood, Dr. C. Dreyfus, Dr. A. Dupré, F.R.S., and Prof. W. A. Allen, F.R.S., have been nominated Vice-Presidents under Rule 8; and Mr. Win. Thorpe has been nominated an Ordinary Member of Council under Rule 17, in the place of Dr. C. Dreyfus, nominated a Vice-President.

Mr. C. A. Fawsitt has been nominated an Ordinary Member of Council under Rule 24, and the Treasurer and Foreign Secretary have been nominated for re-election to their respective offices.

Members are hereby invited to nominate fit and proper persons to fill four vacancies among the Ordinary Members of Council under Rule 18. Special nomination forms for this purpose can be obtained from the General Secretary upon application.

*Extract from Rule 18:*—"No such nomination shall be valid unless it be signed by at least ten Members of the Society who are not in arrear with their subscriptions, nor unless it be received by the General Secretary, at the Society's Office, at least one month before the date of the Annual General Meeting, to the election to take place at which it refers. Nor shall any such nomination be valid if the person nominated be ineligible for election under Rules 12 or 15. No member shall sign more than one nomination form."

#### ANNUAL GENERAL MEETING.

The Annual General Meeting will be held in Edinburgh on the 18th, 19th, and 20th July next. An influential Reception Committee has been formed, and an outline programme (subject to modifications) appears below. Tickets of membership will be issued in time for the meeting, and will form, as heretofore, vouchers for visits to works and excursions.

#### *Draft Programme.*

Wednesday, July 18th.

10.30 a.m.—Council Meeting, Queen Street Hall.

11 a.m.—General Meeting, Queen Street Hall, Presidential Address, &c.

For the afternoon the following visits have been arranged:—

1. Galleries and Museums.
2. Dalmeny Palace.
3. Roslyn.
4. Granton. — Garden Party, by invitation of the Chairman.

8 p.m.—Reception and Conversazione in the Industrial Museum, by invitation of the City of Edinburgh.

Thursday, July 19th.

The following works and places of interest will be open for inspection:—

1. Dalkeith Palace and Ground.—His Grace the Duke of Buccleugh.
2. Floor Cloth and Linoleum Manufacture.—Messrs. J. Barry, Ostlere, and Co., Lim., Kirkcaldy.
3. Printing, Colour Printing, and Bookbinding.—Messrs. Thos. Nelson and Sons, Parkside Works.
4. Paper-Making Machinery.—Messrs. Jas. Bertram and Son, Leith Walk Foundry.
5. Paper Works (Rag and Esparto).—Messrs. A. Cowan and Sons, Lim., Valleyfield Mills, Penicuik.
6. Paper Works (Esparto, &c.).—Messrs. Jas. Brown and Co., Lim., Esk Mills, Penicuik.
7. Soap Manufacture.—Messrs. Wm. Taylor and Co., Broughton Soap Works.
8. Printing Ink, Oils, &c.—Messrs. A. B. Fleming, Caroline Park.
9. Flint Glass Manufacture.—Messrs. Jno. Ford and Co., Holyrood Glass Works.
10. Ornamental Glass Works.—Messrs. A. D. Jenkinson, Abbey Hill.
11. Brewing.—Messrs. W. McEwan and Co., Lim., Fountain Brewery.

12. Brewing.—Messrs. Jas. and Thos. Usher, Park Brewery.
  13. Distilling.—The North British Distillery Co., Lim.
  14. Oils and Paraffin Shale.—The Broxburn Oil Co., Lim., Broxburn.
  15. Gas-Making and Refuse Destructor.—Edinburgh Gas Works, Powderhall.
  16. Coal Mines, Brick and Fireclay Works.—The Lothian Coal Co., Lim., Rosewell.
  17. Shipbuilding.—Messrs. Ramage and Ferguson, Lim., Leith.
  18. Leith Docks.—Peter Whyte, Esq., Superintendent.
- 7.30 p.m.—Annual Dinner, Waterloo Hotel, Edinburgh.

(The above list is subject to modification.)

Friday, July 20th.

Excursions from Edinburgh to—

1. Aberdeen.
2. St. Andrews and Perth.
3. Melrose, Abbotsford, and Peebles.

9 p.m.—Smoking Concert.

#### BANKERS' ORDERS.

For the convenience of Members, the Treasurer has arranged with the Bankers of the Society that they shall collect subscriptions from Bankers in town and country; and Members, who have not already done so, are invited to fill up and sign the Banker's Order enclosed with the December number of the Journal, which should then be sent to the Honorary Treasurer, Mr. E. Rider Cook, East London Soap Works, Bow, E.

#### INTERNATIONAL CONGRESS OF APPLIED CHEMISTRY.

We are requested by the Foreign Office to state that an International Congress, under the patronage of the Belgian Government, will open in Brussels on the 4th August next, in connection with the Antwerp Exhibition, on the subject of Applied Chemistry.

The work of the Congress will be divided into four sections corresponding to those of the Belgian Chemists' Association, viz.:—Sugar, Agricultural Chemistry, Food-stuffs, and Biological Chemistry.

Those who desire to attend the Congress should apply to M. Sachs, 68, Rue d'Allemagne, Brussels (Sugar and Agricultural Chemistry), or to M. H. van Laer, 15, Rue de Hollande, Brussels (Adulteration of Foodstuffs and Biological Chemistry), who will forward full particulars on application.

Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 41 of the bye-laws, the Society has the right of priority of publication for three months of all such papers. Infringement of this bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. EYRE and SPOTTISWOODE, the Society's printers and publishers, to whom all communications respecting them should be addressed.



## LIST OF MEMBERS ELECTED 23rd MAY 1894.

Bristed, John, c/o Messrs. Kemp & Co., Lim., Bombay, India, manager.

Brown, Wm. A., Tudor Oil Works, Port Elizabeth, Cape Colony, oil works manager.

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Holliday, Thos., Lunelough Hall, Huddersfield, aniline dye manufacturer.

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Moneriff, John, Elmside, Balhousie, Perth, N.B., glass and ink manufacturer.

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Simomura, T. K., c/o C. and J. Favre Brant, Kawaguchi, Osaka, Japan, technical chemist.

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Baker, Theodore, 10 Emporium; Oakland, Bergen Co., N.J., U.S.A.

Bentz, E., 10 Manchester; The Elms, Handforth, Cheshire.

Bray, D., 10 Mumbles; Broadmoor Chemical Works, Cloderford, Gloucestershire.

Burghardt, Dr. C. A.; Journals to Fern Cottage, Alderley Edge, Cheshire.

Carter, W. Chas., 10 Abercorn Road; 19, Short Strand, Belfast, Ireland.

Chapman, Spencer; all communications to 36, Mark Lane, E.C.

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Colman, Dr. H. G., 10 Noel Road; 23, Stirling Road, Edgbaston, Birmingham.

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Shalleross, A., 10 City Road; Corbett Street, Bradford, near Manchester.

Shalleross, T. H., 10 Fairfield; Corbett Street, Bradford, near Manchester.

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Thackray, R. B., Journals to Leather Works, Newlay, Leeds.

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Wills, J. Lanson, 10 Ottawa; c/o Dr. F. Wyatt, 12, Old Slip, New York, U.S.A.

Wingham, A., 10 Torquay; 112, Warwick Street, Pimlico, S.W.

Withaus, Dr. R. A., 10 55th Street; 303, West 77th Street, New York, U.S.A.

Wyatt, Dr. F., 10 Park Place; 12, Old Slip, New York, U.S.A.

Young, W. C., 10 Forest Gate; Normanhurst, High Road, Romford, Essex.

## CHANGES OF ADDRESS REQUIRED.

Clark, Jno., 10 Great Brook Street, Birmingham.

Isaac, T. W. P., 10 Cavendish Place, Brighton.

Whitehouse, E., 10 Smethwick, Birmingham.

## Deaths.

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*Hon. Local Secretary:* John Heron,  
74, North Side, Clapham Common, S.W.

The names in italics are those of Members of Committee who retire at the end of the current session.

The following have been elected to fill the vacancies, and will take office in July next:—*Chairman:* B. E. R. Newlands. *Committee:* E. Grant Hooper, H. de Brossenthal, E. O'Neill, S. Rideal, A. Shearer, and Wm. Thorp.

SESSION 1893-94.

Meeting held Monday, May 7th, 1894.

MR. WM. THORP IN THE CHAIR.

## THE ELECTROLYSIS OF FUSED SALTS.

BY CLAUDE VAUTIN.

I wish to couple the name of Mr. H. K. Picard with mine in this matter as a slight recognition of the manner in which he, assisted by Mr. Hopkins, has helped me in the investigation I have been engaged upon in the above subject.

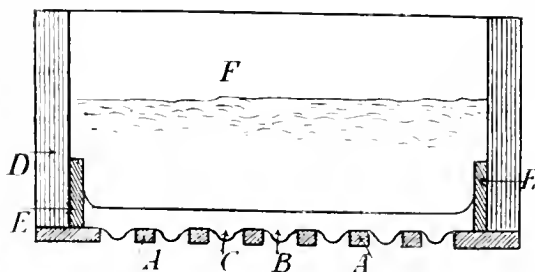
It is my intention to bring under the notice of the Society notes of some of the work that has been done in my laboratory during the past year. I regret that I did not at the outset devise a less comprehensive title for the paper; the commercial electrolysis of fused salts is indeed a subject exceedingly wide, and I purpose to limit my remarks to the decomposition of the fused chlorides of sodium and potassium, trusting at some future date to submit some further notes.

It may be interesting if I explain why I directed my attention to the electrolysis of the fused salts of the alkaline metals rather than their solutions in water. After having endeavoured for a considerable time to devise a method of economically decomposing a solution of chloride of sodium in water, the use of a mercury cathode of somewhat novel form was worked out. You will understand that the surface tension of mercury is so great that it will permit a horizontal layer of from  $\frac{1}{16}$ th to  $\frac{1}{2}$  an inch in thickness to be sustained on a perforated plate or open mesh of certain sized perforations or openings without falling through. In Fig. 1 I have illustrated in somewhat exaggerated form the effect on the under surface of mercury when supported by a perforated plate.

With this application of mercury as a cathode, a vat was constructed in which was an inner tray, the bottom of which consisted of a finely perforated non-conducting material, or, if the bottom was made of metal, it was coated with an insulating material so as to ensure complete insulation, otherwise the retaining plate would become part of the cathode, which would be undesirable. Upon the

bottom of this inner trough a layer of mercury was supported and made the cathode, and carbon in the outer vessel the anode. On passing the current, sodium was

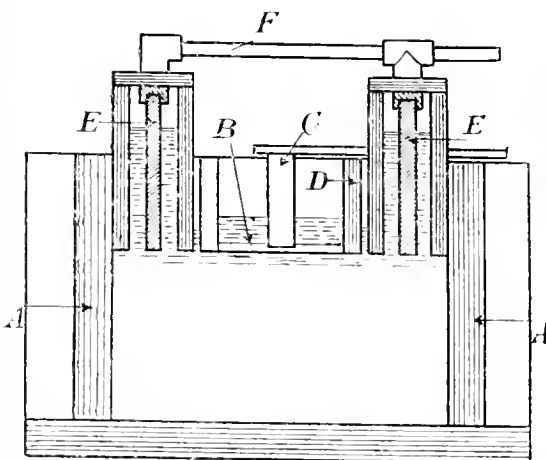
Fig. 1.



deposited upon the under side of the mercury, forming an amalgam which, in consequence of its lightness, rose rapidly to the surface of the mercury, where, coming in contact with water, the latter was decomposed with the formation of sodium hydrate.

Fig. 2 represents diagrammatically the form of apparatus. A represents a vat constructed of suitable material; D the

Fig. 2.



inner or mercury cathode containing tray; B finely perforated plate for retaining the mercury; C conductor dipping into the mercury; E carbon anodes; F chlorine exit.

This method worked fairly well, but, nevertheless, I could not bring myself to believe that any of the methods for decomposing solutions of sodium chloride known to me would, in any great measure, take the place of the existing chemical means of producing chlorine and soda, for all or any of the following reasons:—

1, the vastness of the plant required; 2, the comparatively low conductivity of solutions; 3, the disintegration of the carbons, and the several other well-known difficulties and objections in connection with the electrolysis of solution of NaCl. The foregoing remarks do not refer to the electrolysis of a solution of KCl.

I then turned my attention to the use of fused alkaline chlorides as electrolytes, but the literature on the subject and the results obtained by many investigators, together with our own work, exposed so many difficulties, whenever it was attempted to recover the alkaline metal either by distillation or by collecting under bells, that it was decided, if possible, to obtain sodium or potassium in the form of an alloy.

When endeavouring to obtain the alkaline metals by distillation or any method where a solid cathode was used, the yield fell far below theoretical quantity; and it appears that either the statements that have been frequently made

as to the existence of the subchloride of the alkali metals must be true, or else these metals must be dissolved in an electrolyte of their fused chlorides.

In 1873 Werdermann obtained letters patent in England for a process wherein he claimed the formation of sodic subchloride by electrolysis fused salt with carbon electrodes. By this process, when it was estimated that a considerable percentage of sodium subchloride had been formed, the contents of the vessel were permitted to cool, treated with water, when it is stated that sodium hydrate was formed according to the formula :—



It was also proposed that the subchloride should be used as a reducing agent, and, as an illustration, the reduction of cryolite was mentioned according to the following formula :—



I mention these facts as showing that previous investigators had, in all probability, been confronted with a low yield of sodium when attempting to electrolyse its fused chloride.

Referring to the recovery of the alkaline metals by distillation during electrolysis, I am of opinion, even supposing that anything like the theoretical quantity of metal could be obtained, that a very considerable amount of the electrolyte would distil over with the metal; thus it would be difficult to obtain the metal in a state of purity and free from the electrolyte.

In attempting to collect the metal under bells, as has been lately suggested, considerable difficulty has been experienced in preventing the bells, if composed of non-conducting refractory material, from becoming destroyed, both by the action of the electrolyte and the freed alkaline metal. When iron bells have been used, insulated on the inside, trouble has been experienced in preventing the insulation from breaking down and the outside of the bell becoming part of the cathode, thus causing a portion of the alkaline metal to be liberated at the anode, where recombination with the chlorine would cause a great waste of energy.

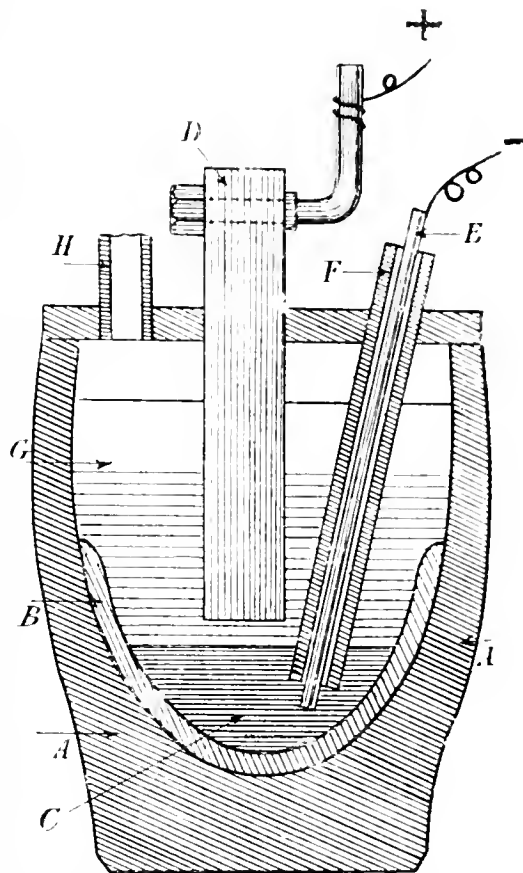
With a view to overcoming these difficulties, it was decided to obtain the sodium in the form of an alloy: lead naturally suggested itself as a most convenient metal with which to form this alloy because of its low fusing point, its indifference to a solution of sodium hydrate, its stability under either fused  $\text{Na}_2\text{O}$  or its hydrate, and its cheapness.

I requested Mr. Picard to make an alloy by adding sodium to lead; and this alloy containing upwards of 20 per cent. of sodium was heated in a crucible under fused  $\text{NaCl}$ . I was quite prepared to find, upon examining the contents of the crucible after it had been removed from the furnace and allowed to cool, that the sodium had distilled out, or had been acted upon by the fused chloride. Such was not the case, for it was found upon careful examination that little, if any, sodium had been removed from the lead at the temperature at which the operation had been conducted.

This experiment, followed up by one in which molten lead was made the cathode, using melted sodium chloride as the electrolyte, gave results confirmed by subsequent investigations, that warrant me in stating that the question of economically producing chlorine and soda or potash by the electrolysis of their fused chlorides has, in a measure, been solved. The experiment was conducted in the apparatus represented by Fig. 3. A represents a crucible, B a lining of magnesite, C molten lead, D carbon anode, E iron conductor dipping into the lead at C, F non-conducting tube to insulate the iron E from the electrolyte G, G molten salt, H exit for chlorine. Upon passing the current of about 100 amperes, the lead alloyed with the liberated sodium, the surface of the lead being the true cathode. The experiment was run until about 8 per cent. of sodium to the weight of lead had been deposited. After allowing the crucible to cool, it was broken, and the alloy removed; it was brittle, and acted with energy on water. The salt was not alkaline, intimating that no sodium had been removed from the lead with the formation of any basic chloride. The operation,

from start to finish, was uniform; no variation of resistance was noted. Indeed why should there be any disturbing element, for the conditions of the electrolyte are constant

Fig. 3.



and, provided the anodes stand up and the sodium is alloyed as liberated, how are the conditions to vary?

A number of experiments were made in the apparatus represented by Fig. 4, the lettering being the same as in Fig. 3, with the addition of A', which is an inner crucible containing the lead cathode. By this arrangement the removing of the alloy was facilitated, for, after having run for a stated period, the inner crucible could be removed by a pair of tongs and the contents either cast into a mould or allowed to cool down with the crucible, and removed by breaking. This method was economical so far as large crucibles were concerned. Subsequently, lined iron pots were used, as will be explained farther on. A fact of the utmost importance is the possibility of obtaining nearly the theoretical quantity of ions when working in this manner.

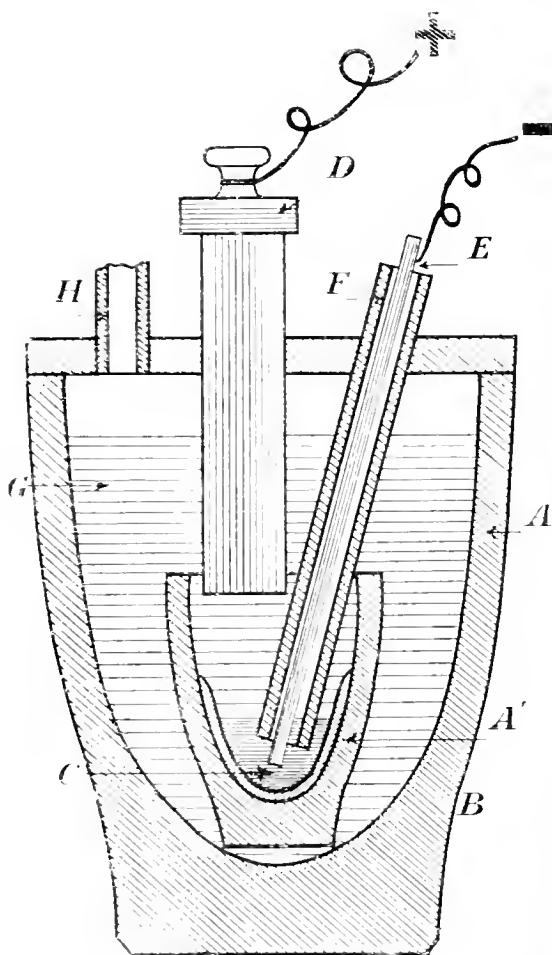
With the view of ascertaining in a practical manner the exact difference in the resistance of a solution of sodium chloride and the same chloride in a fused condition, the following experiment was conducted.

Two crucibles were selected, practically corresponding in shape and size; in the bottom of each an equal quantity of lead was carefully melted and the insulating tubes containing the cathode conductor were inserted.

In one, a saturated solution of salt was placed; in the other salt was melted; in both cases the depth of the electrolytes was the same. The anodes were of the same size and character and placed in exactly corresponding positions in both crucibles, that is to say, the distance from the lead cathode and depth of immersion were the same, and the experiment demonstrated that, at least five times the quantity of current could be passed through the fused salt as is possible

through the solution at the same pressure. At 3.5 volts, 1.4 amperes passes through the solution, and at 2 volts, 4 amperes passes through the fused salts. Although the foregoing experiment gave results considerably in favour of fused salt, so far as unit of plant is concerned, in practice a much larger ratio in favour of the fused salt is possible; for in any wet installation it would not be possible to so advantageously arrange the relative position of anode and cathode as in this experimental run, to say nothing of the

Fig. 4.



extra resistance, in systems where diaphragms are used. In our own work when using the mercury cathode, as previously explained, and under the most favourable conditions as to the position of the anodes, it was not possible to pass more than  $\frac{1}{2}$ th of the current at the same voltage, area for area, as when using the fused electrolyte.

Naturally, to a great extent, the ultimate purpose to which the alloy is to be put will determine the form of the apparatus; if the alloy is to be used as a source of metallic sodium or potassium, sodic or potassic oxide or hydrate, then, at present, I am inclined to the use of steel pots lined with magnesite, except at the bottom, for a reason to be explained further on. Fig. 5 represents what I mean.

The lettering to E represents the same parts as in Fig. 3. Here, the pot being of iron or steel and being insulated to a point below the surface of the lead, any convenient part of it can be made the cathode connection; it is shown here at E. L represents a pot in which lead can be melted and run into A when required by pipe J. K pipe from bottom of A to caustic pot L. Starting with a given

quantity of lead in A, the alloy is formed, and, when deemed rich enough, the valve in K is opened and the alloy run into L, where it is submitted to the action of steam, by which means the sodium of the alloy is converted into its corresponding hydrate.

A fresh supply of lead is run into A from I when the alloy is tapped into L from time to time. The lead in L, after the alkaline metal has been removed, is taken back to I for further use.

You will note that the pot itself is made the cathode connection, but, in consequence of the lining above referred to, coming down below the surface of the molten lead, the surface of the lead is thus caused to be the true cathode; the metal of the pot forming a most convenient means of conducting the current from the lead. If the sides of the pot were not protected by a neutral insulating material, not only the surface of the lead but also the pot itself, or rather that portion of the pot in contact with the molten salt, would become a part of the cathode. The covers of the pots, instead of being constructed of fragile refractory material, can be constructed of iron protected from the action of the chlorine by the following means:—

The iron cover is dipped into molten salt, when a film or layer is frozen, so to say, on it, provided the iron is removed from the molten salt soon enough; and, the temperature to which the cover is exposed not being high enough to melt the coating of salt thereon, a most effective means of protecting the iron from the action of the liberated chlorine is obtained.

Iron is attacked by heated dry chlorine, in spite of the often repeated statement to the contrary.

Objection has been made to the use of cast-iron pots in which to melt salt, for, if heated from the outside and the salt permitted to come in contact with the inside, the salt, to a considerable extent, sweats through; but, if cast-iron pots lined with a neutral material down to a point below the level of the lead are used, this objection falls to the ground; however, in my opinion, steel is much to be preferred as material for the construction of the pot. The question of melting the salt either by external means by the combustion of gas or coal, or, internally, by means of electricity, is not quite a settled matter; there are a number of pros and cons, that can be settled only by actual work; personally, I lean towards maintaining the salt in a molten condition by means of the current, for, by so doing, the life of the apparatus will be considerably prolonged, and greater simplicity in the construction of the plant ensured.

Melting in basic lined muffle furnaces is advocated by those well versed in the salt-cake furnace operations, but there are, at present, many objections and difficulties that I foresee, which, however, may be overcome by chemical engineers. I refer particularly to the difficulty of collecting the chlorine and of so arranging the furnace that the principle of having several decomposers in series can be advantageously applied.

A convenient form of apparatus for producing the alloy of potassium and sodium with lead, tin, &c., in small quantities is represented by Fig. 7. The lettering down to and including H represents the same as in Fig. 3.

The section of the furnace T is rectangular, with an exit for the products of combustion at U, and provided with an injector for gas or petroleum at V, the steel crucible A rests on and in the support W, at the bottom of furnace T. A cover X made in four sections containing a circular opening and resting on the sides of the furnace and extending in to the furnace far enough to cover the top of the crucible. Between the true cover Y and X is a space from which an opening H is provided as a means of exit for the chlorine. The true cover Y is provided with an aperture through which to insert the anode. To the bottom of the pot is connected an iron pipe marked Z. The pipe Z passing through the bottom of the furnace and connected with the bottom of the pot affords a ready means of removing the alloy from time to time, and, as can be easily understood, from reference to the sketch, the alloy in Z could be caused to set by circulating water through the pipe M, which is tightly coiled round Z, to remove the alloy; or a valve in Z can be used instead of the setting method. When it is desired to remove the alloy, the water can be shut off from

M, when the alloy will melt and run into vertical ingot mould N. The potassium or sodium can be protected from the oxidising influence of the air by burning some hydrocarbon in the vicinity of the exit pipe H and the mouth of the mould, say at O. You will note that the pot, or any portion of it, can be made the cathode connection.

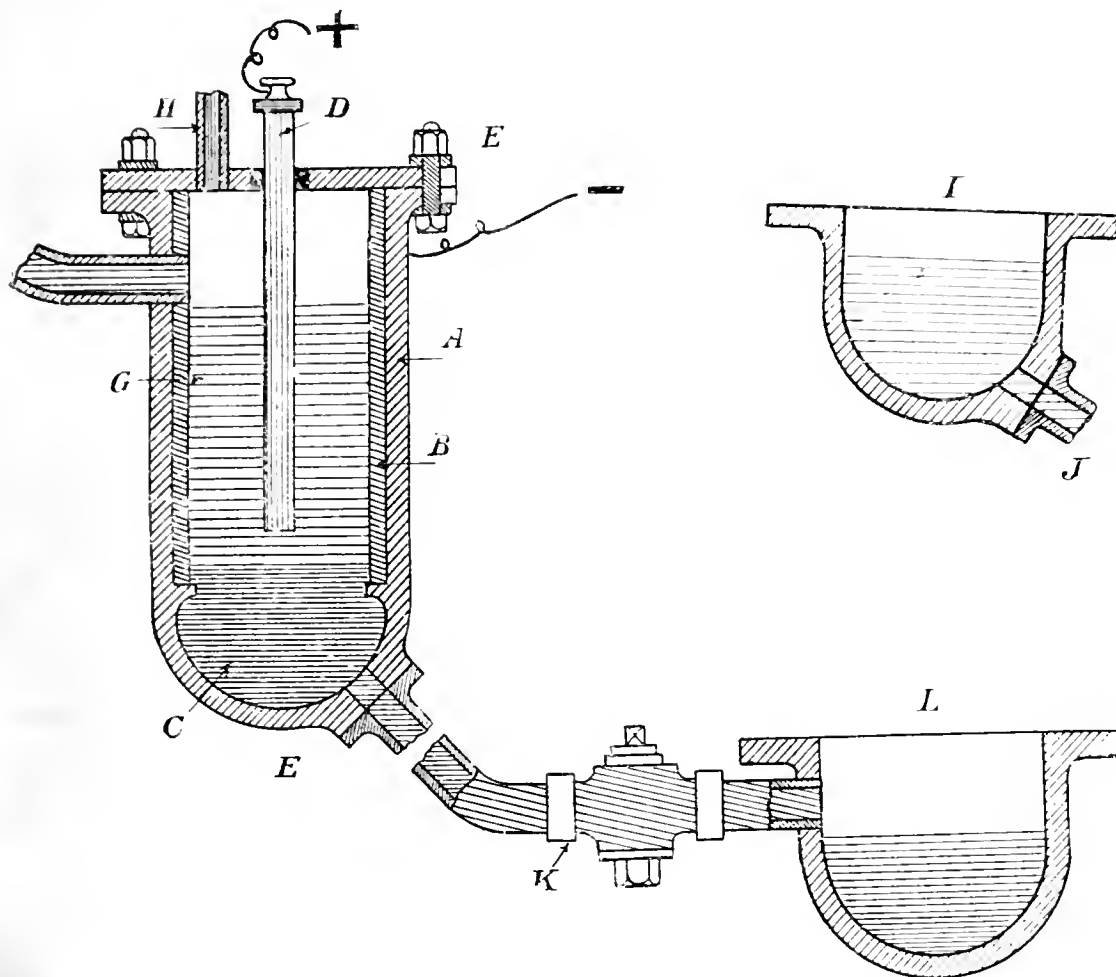
This method of removing the alloy can be used with advantage in larger installations. In working the process the temperature at the bottom of the pot, or rather that portion of the pot surrounding the alloy, should be kept as low as is possible consistent with the fluidity of the metal; therefore it is well that the support or rest for the pot L should continue for several inches above the bottom of the pot.

In actual work it is found expedient to limit the percentage of the alkali metal in the alloy to from 10 to 20 per cent., the use to which the alloy is to be put regulating the percentage.

The sodium or potassium can be removed from the lead in the form of a hydrate by any of the following methods:—

A, by tapping the alloy from the decomposing vessel into moulds as previously described, allowing to cool, removing from mould, breaking in pieces (which can be done with facility in consequence of its brittleness), placing in water, when, as can be easily understood, the hydrate of the alkaline metal is produced, fortunately the lead remaining in a condition in which it can be easily re-melted, without any serious loss; or the alloy is re-melted in an iron pot and

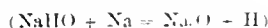
Fig. 5.



submitted to the action of steam, when fused caustic is produced, which can be ladled from the surface of the lead; or by arranging the apparatus as represented in Fig. 6, so that the lead forming the cathode in the decomposing vessel is connected by means of an iron pipe N to molten lead in an outer or separate pot M, where it is submitted to the action of steam, introduced by pipe P and, in consequence of the rapid diffusion of the sodium or potassium through molten lead, contained in the connecting pipe N, the lead in the outer pot becomes impregnated with the alkaline metal, which, coming in contact with steam, alkaline hydrates are formed in a fused condition and can be ladled or tapped out, and it is thus possible to have a continuous process of producing caustic alkali in a fused condition; the lead

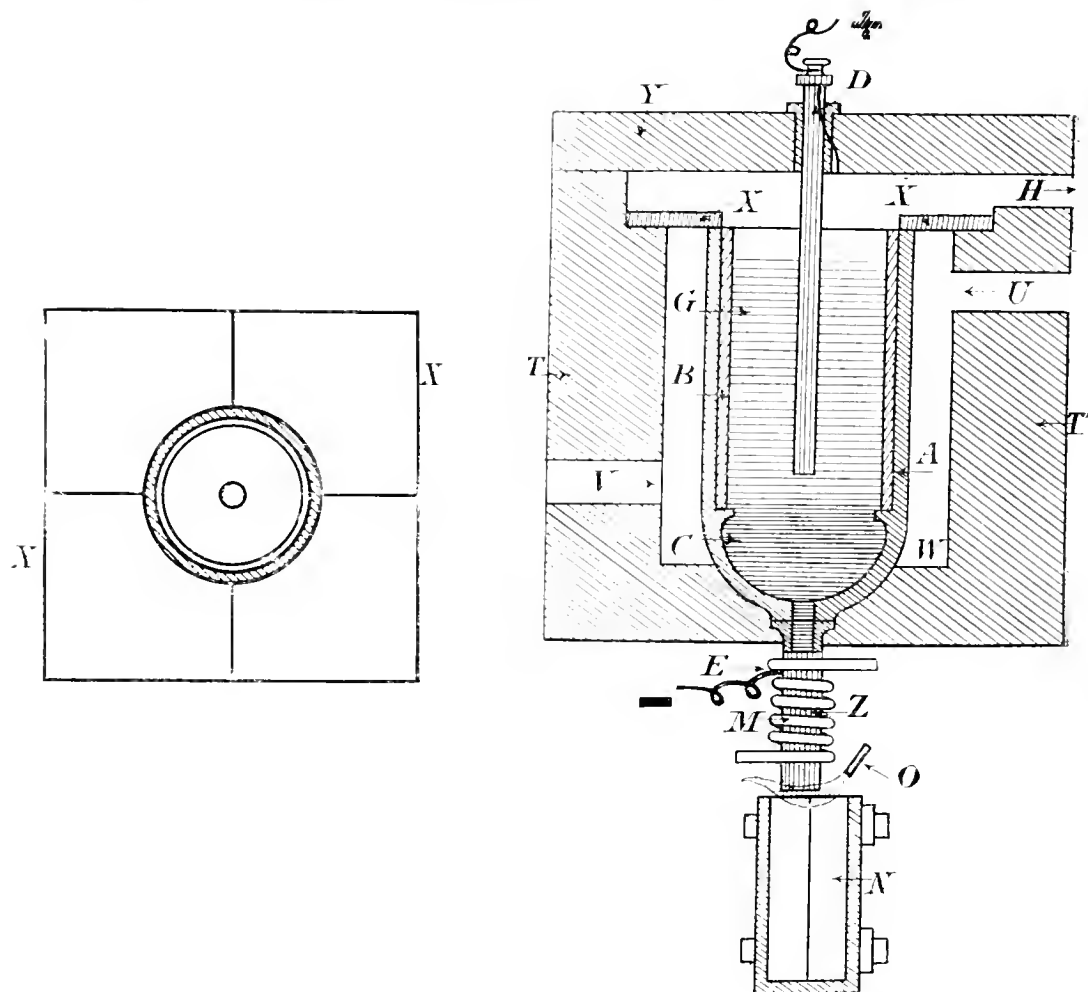
simply acting as carrier of the alkaline metal from the decomposing pot to the caustic pot. O represents exit for hydrogen and excess of steam, R fused caustic, S outlet for caustic.

When it is desirable to remove the sodium from the lead in the form of sodic oxide, all that is necessary is to melt the lead sodium alloy under a layer of fused caustic soda according to the well-known reaction.



I refer to this reaction as being of considerable importance, for it is exceedingly probable from the result of some experiments lately made, that sodium peroxide can be manufactured by the oxidation of  $\text{Na}_2\text{O}$  so formed. The alloy can un-

Fig. 6.



doubtedly be put to other uses, for instance, the production of potassium cyanide or a mixture of sodium and potassium cyanide by decomposing the ferro salts. The lead alloy, in consequence of its brittleness, can be easily crushed, and, provided a small quantity of mineral oil is used during crushing, little if any loss of the alkaline metal by oxidation is experienced. The crushed alloy can be mixed with dehydrated ferrocyanide and the mixture submitted to heat, when the alkaline metal will take the place of the iron, the lead and iron settling out well from the fused cyanide. Being able to finely divide the alloy and thus intimately mix the sodium with the ferrocyanide, it appears that a more perfect reduction can be obtained than when unalloyed metallic sodium is used, in consequence of the difficulty of finely dividing and mixing that metal through the ferrocyanide.

Metallic sodium can, to some extent, be distilled in properly constructed retorts from its alloy with lead; this is an important matter, since it enhances the value of the process as a ready means for obtaining metallic sodium or potassium from chloride by means of electrolysis, and experiments are now being conducted with a view of working this out to a practical issue.

As to cost of decomposing a given quantity of salt, I think I cannot do better than refer you to the able paper of Messrs. Cross and Bevan, published in the Journal of this Society in December 1892. In small installations I feel inclined to suggest that the use of large gas engines

should receive attention. I had an interview with a representative of Messrs. Tangye on Monday last and, from the information given me by that gentleman, it appears that with producer gas large gas engines can be run with great economy, and I am informed that Mr. Parker is putting down a 200 h.p. gas-motor plant to drive dynamos in some electrolytic installation.

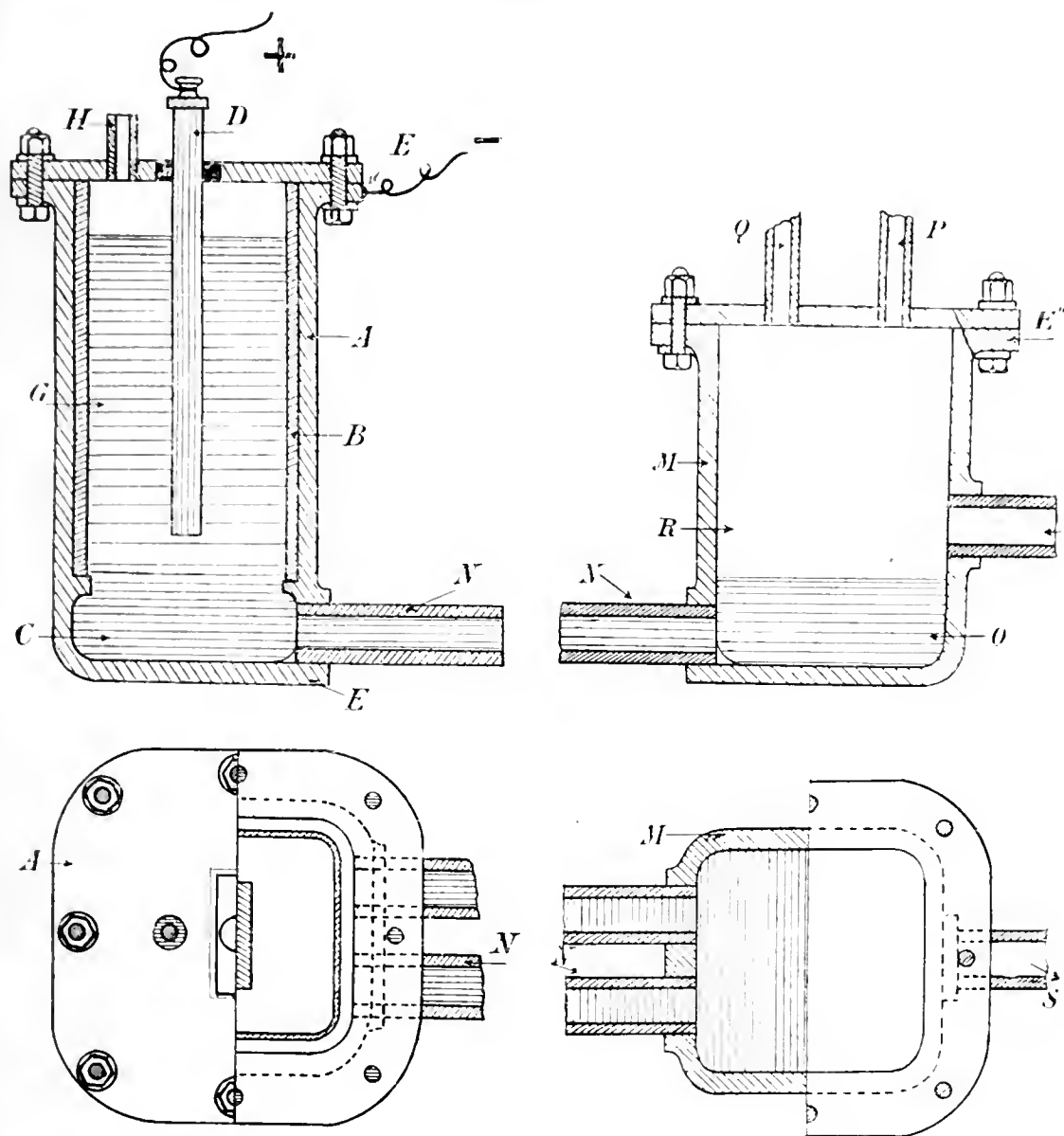
The voltage necessary to decompose the fused chlorides of the alkaline metals is just under 2.

By using pure NaCl and lead and decomposing the resulting alloy of Na and Pb by means of distilled water in silver dishes, it is possible to obtain sodium hydrate in as great a state of purity as can now be obtained in the market. I mean pure NaHO for laboratory purposes.

The anodes used are of gas carbon, but in order to ensure continuous working with the minimum of disintegration, it is necessary that the carbon should be submitted to the following treatment:—

The anodes boiled in a concentrated solution of sugar carefully re-carbonised and, when thus treated, appear to stand sufficiently well for all practical purposes. I have here an anode that has been in work for several months, and you can see by comparing the thickness of that portion of the carbon that has been in the electrolyte with that portion that has not, that little, if any, abrasion or loss has taken place. If the carbons are used without the above-named method or other pore-filling process, after running for a few hours, a considerable increase of resistance in the

Fig. 7.



circuit is noted, probably in consequence of chlorines being generated in, and remaining in, the pores of the carbon, thus causing the noticed increase of resistance, in fact an ordinary porous carbon after two hours' run becomes perfectly insulated, even under 10 volts pressure. That the life of gas-retort carbon in melted chloride is greater than in the corresponding solutions I am sure of. I most willingly admit that there are many points to get over, but when the great chemical engineering skill available in England is taken into consideration, I have no doubt as to the ultimate results.

#### DISCUSSION.

Mr. C. F. CROSS said he had listened to what he considered an extremely modest account of a departure in electrolytic practice which appeared of the greatest technical importance. He was not able to follow the author through his criticism of previous literature with regard to the electrolysis of fused salts. He had followed somewhat closely

the developments of electrolysis as applied to solutions, more especially solutions of common salt. Mr. Vantin evidently regarded that matter rather as ancient history, and could not have followed very appreciatively some of the developments which had taken place, more especially during the last eighteen months or two years. If he had not been so enamoured of his new discovery, he might have ascertained that the Le Snecr method, which he knew of as a more or less "laboratory process," had been running on strictly commercial lines now for nearly two years in the United States. Without going into details of production, he might state that 85 per cent. of efficiency was steadily realised on the commercial scale, and that all difficulties with regard to diaphragms and carbon anodes had been reduced to a highly satisfactory minimum, so that he thought they might congratulate those who were still working on the wet methods on having achieved a great deal in what appeared ten years ago to be a very far off problem. Mr. Vantin had mentioned the method of electrolysing salt solution with a



mercury cathode in which Kellner and himself appeared to have run a close race for priority. This was a philosophically interesting development, but there did not appear to be much prospect of realising anything like commercial results with this cathode. The continuous separation of the cathode from the electrolyte was also the subject of Hargreaves and Bird's patent of more recent date, whereby with mere purely mechanical appliances the same absolute separation of the sodium hydrate from the chloride was accomplished. Here, again, considering the low voltage at which this process was run, and the steady realisation of an efficiency certainly over 80 per cent., he would have formidable rivals to contend with. In reading his paper he had not given many details in point of measurements; possibly he had not gone far enough to ascertain these with precision. He gave a comparative estimation of the amount of current which could be passed—all other conditions being maintained equal—through a saturated solution of salt and through his fused salt, and arrived at a multiple of 5 in favour of the latter. He (Mr. Cross) was unable to traverse that statement, and accepted it with the reservation that all statements of this general kind were liable to error when all the factors were not distinctly stated and separated one from the other. He would like to know, as the next element of cost of production, the cost of the fusion of the sodium chloride and the cost of keeping it in a state of fusion. Of course, theoretically the amount of heat necessary for the fusion of sodium chloride was small, but when dealing with practical quantities those matters had to be taken into serious consideration. Those who were dealing with brine had the obvious advantage that they could operate directly on the product, whereas in dealing with a solid which had first to be mined in a more costly way and then fused, the initial cost in the cell would be greater, giving an advantage in favour of brine. Then there was the question of the chlorine, and he should like to know how he dealt practically with the chlorine, which was evolved at a very high temperature. Generally the technical public would require full comparative statements both of current measurements and chemical statistics in order to form a satisfactory judgment as to the relative cost of the dry and wet processes.

Mr. W. F. REID said he should also like to make a remark on what was said about the aqueous electrolysis. It seemed to him that keeping up this high temperature during the process must to some extent balance its economy. If he understood the author rightly, he claimed economy on the ground of the lesser amount of electricity required to effect decomposition. If that was so, of course it would be a question of the difference between the amount saved in that way and the amount of fuel expended in keeping up the high temperature. He was of opinion that in electrolytic processes for the production of caustic soda, there were at present two difficulties. One was the plant which was used, and the deterioration of that plant, and the cost of keeping it in order. That was only partially overcome, and was still a difficulty, in spite of the sanguine views expressed by some patentees with regard to their anodes. The other difficulty was the cost of the electricity itself. Even when the process worked smoothly one found oneself running very close indeed, as regards cost of production, with the old methods of producing caustic soda. With electricity at its present price, the margin in favour of electrolytic soda was not great, and it depended on economy of management, and having very good apparatus. When he referred to that point once before, a gentleman present, an engineer, said they could not hope to get cheaper electricity, because they had almost attained a maximum of efficiency. That referred really to the efficiency of the steam-engine, but perhaps at some future time electricity might be obtained without the steam-engine, and then, no doubt, caustic soda produced by the wet process or, perhaps, by this one, would be cheaper than caustic soda produced by chemical means. It also occurred to him that there might be a difficulty from the volatilisation of the lead as the process was conducted, and if so, special means would have to be devised, not only to keep that from entering the atmosphere

of the work-shop where the operations were carried on, but also to prevent the escape of free chlorine, which must be a great source of nuisance. Of course it might be made a source of profit, but he should like to hear whether that had been accomplished. Certainly the process as described would be classed amongst the dangerous industries. He should like to know the fusing point of the alloy of lead and sodium, and also whether the apparatus, Fig. 5, had been carried out in actual practice, for it appeared that there would be some difficulty in running the alloy between the two vessels, though perhaps this could be overcome if the fusing point were very low. With regard to the lining of crucibles with magnesia, he would suggest that it might be possible to employ crucibles made almost entirely of magnesia. He had, himself, in research experiments, made crucibles, which stood very well, of Portland cement. He mixed it, and burnt it very carefully after it had set, and found he got a fairly strong crucible to which a basic lining adhered very well. It would stand a temperature far higher than that used in these experiments. It seemed to him that on a large scale there was a great source of energy which might be further utilised in the decomposition of the raw material. With regard to the statement that no trace of lead could be found in the solution, that was entirely against his own experience. He should have thought that in the caustic soda solution produced by decomposition of lead and sodium even more than traces of lead would be found, and he should be glad to hear if the author had made a careful examination on that point.

Mr. J. SWINBURNE took exception to the author's statement that wet processes of electrolytic caustic and bleach manufacture would not pay. The assertion was too sweeping. There were too many processes before the public; some good, most bad; but the difference between the worst and the best was very small in comparison with the difference between any reasonable electrolytic method and the present chemical processes. The difference in expense was infinitely less than that between a badly managed limited liability company and a worse managed limited liability company.

Mr. Vautin said dynamo makers could not make low pressure dynamos. As a dynamo maker he would correct this statement. Dynamos could be made for any pressures that might be wanted. The reason why the vats were generally put in series is that if in parallel the copper leads would be too expensive. There was thus a disadvantage in using very low pressures.

Before discussing the merits of fixed salt and solution in making electrolytic caustic and bleach, he would point out that Mr. Vautin's excellent process had other openings. There is a large market for sodium, which could, no doubt, be largely filled by a sodium lead alloy. The alloy could, no doubt, be used for making cyanide of potassium and sodium from yellow prussiate of potash. This method had the advantage that, as potassium cyanide was estimated by its cyanogen, and the combining weight of sodium was low, one could make impure cyanide which would pass as 100 per cent. cyanide of potassium, or even higher. There was an increasing demand for pure cyanide of potassium, and also for sodium peroxide. Sodium oxide could of course be made by treating lead sodium alloy with a limited weight of water or steam; and the oxide may be peroxidised. Then there was a demand for sodium in the reduction of aluminium; and if the price were brought down the demand would probably increase. Then though chlorine attacks red-hot iron, if dry it does not attack cold iron. The chlorine from fused salt can therefore be cooled and stored and transported in iron or steel tube flasks in the liquid form. The saving due to this method of packing would be very considerable.

Coming down to the question of making caustic, he thought Mr. Vautin had laid too much stress on the purity of his caustic. It was a matter of pounds, shillings, and pence. If the removal of salt in the wet process added so many shillings a ton to the cost of manufacture, that must be taken into account, but that was all that was to be said. The cost of fusing the salt would, he thought, be nil, if done electrically from the inside. The surplus of the electrical

energy above the heat energy of combination must in any commercial process be amply above that necessary to heat the salt. The question of keeping in the heat was only a question of size of vat and of lagging. The contents of a vat altered to scale in all its dimensions varied as the cube of its dimensions, and its output varied between the square and cube, while the loss of heat varied as the dimensions only, so a size was easily reached above which the difficulty was to keep the vat cool. This method of heating overcame a very serious difficulty not mentioned by Mr. Vautin, namely, the wear and tear of the iron pots when heated externally. On the other hand, the cost of evaporating and finishing was very serious in the ordinary process. On the wet electrolytic process it might be less because there was more salt. In the wet process the waste power could not be utilised in the same way as in the electrolysis fused salt.

While on the subject of waste power he would make a digression on the calculation of what is called the "theoretical" electro-motive force from the heats of combination. There was seldom a paper or a book on electrolysis published in which the authors did not calculate the electro-motive forces necessary for decomposition by dividing the heats of combinations by constants. In the first place, writers generally took wrong figures to begin with. For instance: to find the electro-motive force necessary to decompose solution of sodium sulphate, they would take the figures due to burning metallic sodium in gaseous oxygen, which have nothing to do with the matter. This was a mere detail, however. The whole principle was wrong. It was shown by Helmholtz in 1882 that the heat of combination was only one term, and another must be taken; so that the ordinary method was quite wrong. Yet people still go on calculating what they call the theoretical volts. To publish such a calculation in 1894 brands a man as an electrolytic crank of the deepest dye.

A great deal was said about the price of electrical power. He had given the figure as a farthing per kilowatt hour, including depreciation and interest, at the Institution of Electrical Engineers a few years ago, and he thought this figure has been generally accepted by electrical engineers. The cost of energy must not be taken from central station figures, as the engines and boilers are there running under the worst possible conditions.

The CHAIRMAN said this was one of those processes in which it seemed to him the error came in the passage from the small scale to the large. He did not know whether Mr. Vautin could give any more definite figures, but if so they would be very important.

Mr. PARKER said the difficulty was much greater in dealing with sodium salt in solution than with potassium salt.

Mr. VAUTIN said in reply that he would first refer to the question of temperature and in his notes he referred to the question of maintaining a mixture of salts in a molten condition by means of an internally heated crucible by means of the current.

By a judicious mixture of the chlorides of the alkali metals it was possible to have a mixture of a very low melting point. He was sorry to have created an impression in Mr. Cross's mind that he assumed that all wet processes were ancient history; what he did say was referring to the wet process known to him; and he happened to know the Hargreaves process or principle, or one of exactly similar principle before he left Melbourne some seven years ago.

In addition to the reasons he had given he considered that the amount of doubt and uncertainty that attached to the wet processes, rightly or wrongly, rendered it very unlikely that there would be an immediate change from the chemical method of manufacture.

With regard to the criticism that he did not give the exact figures, he had said the proportion was five to one; he had the figures in the paper, but omitted them in reading. As a matter of fact in the wet process at  $3\frac{1}{2}$  volts, 1.4 amperes passed through the solution. On the other hand, just under 2 were needed in the dry, and 4 amperes passed,

which just made a difference of 1 to 5; and there was this difference in his favour, that with 2 volts he could not work the wet experiment at all, but required  $3\frac{1}{2}$ . As far as the cost was concerned, it was to a great extent the fact that they were able to work at very much lower voltage, and that compensated to a great degree the cost of keeping the salt molten. They could afford to use a portion of the electricity over and above that necessary to decompose a solution of salt for the purpose of keeping the salt molten. In fact, in a large operation the trouble would be to keep the temperature down, and in all probability it would be great economy to burn a little extra coal under the boiler and keep the whole of the electrolyte molten by means of the electric current. In that case the life of the plant would be considerably enhanced. With regard to the volatilisation of the lead, he could only say that such did not exist. Lead protected from the air was rather a fixed metal at a temperature much higher than that required for the operation. As for chlorine the one object was to lead it into a chamber in which there was finely-divided hydrated lime. The chlorine coming away from an apparatus on a large scale was not nearly so high in temperature as the hydrochloric acid from a salt-cake furnace. He had shown this small apparatus simply for the sake of giving a practical demonstration that the alloy could be formed. It was very different on a larger scale. Again, with regard to the cost, they had not to evaporate the solution of caustic nor a mixture of caustic soda and chlorine of sodium to separate them. His criticisms of the wet processes in no way referred to solutions of potassium chloride, but he thought he was right in saying that 90 per cent. of the wet electrolytic processes in the States or elsewhere were in connection with potassium salts. He knew that in potassium salts, advantage could be taken of the ease with which chlorate crystallised out from the electrolyte.

## THE TESTING OF HYDRAULIC CEMENTS.

BY W. HARRY STANGER, M.I.C.E., F.C.S., AND BERTRAM BLOUNT, F.I.C., F.C.S.

FOR a good many years past there has been a large and increasing amount of attention devoted to the testing and control of the quality of hydraulic materials of construction and especially of Portland cement. This can be traced to the failures that have occasionally occurred from not very obvious causes, in large and costly engineering undertakings, and proximately to the natural anxiety, on the part of engineers, thus engendered. Persistent technical research and the constant comparison of results obtained in the laboratory and testing room with those observed in actual work have improved the methods of testing, and increased the accuracy of the deductions that can be made from a properly executed series of tests, until at the present time no engineer using hydraulic cement who desires to be assured of the permanence of his work omits to obtain proof of the quality of the material by systematic testing and analysis.

The general methods of testing cement are too well known to warrant a lengthy description in this paper, but it may be permissible to indicate the methods which, among the many that appear in text books and technical journals, are of sufficient importance and general applicability to stand as representatives of modern practice. The following order will be found convenient.

**Fineness.**—The only method of ascertaining the fineness of cement that is in common use is that of sifting. It has been recognised comparatively recently that the coarser particles of an hydraulic cement, certainly all that remain on a sieve having 76 meshes per linear inch, and probably many that pass this sieve, are either wholly inert or so slow in setting as to be useless or even objectionable. The inertness does not arise from any chemical difference between the coarse "core" and the rest of the cement, but

from the fact that the surface presented by a coarse particle is relatively smaller than that by a fine particle, and the rate of attack by water in the process of setting is correspondingly diminished. It has resulted from the recognition of this fact that cement specifications now demand greater fineness than was formerly usual, and manufacturers have striven to comply with this demand by the use of improved grinding machinery, and by sifting the finished cement before putting it on the market. Not very long ago a residue of "10 per cent. on a 50" was regarded as indicating quite a fine cement, whereas "10 per cent. on a 76" is now in no way an uncommon requirement, and much cement is of still finer grade. The plan of specifying that not more than a certain residue of coarse core shall be left on a standard sieve is convenient, but possesses certain disadvantages. In the first place it offers no guarantee that a large portion of the cement may not consist of particles just small enough to squeeze through the meshes of the sieve, but still too coarse to possess much value as cement. This can obviously be remedied by adding to the specification a clause requiring that not only shall the cement leave no more than a certain amount of coarse core, but that it shall also contain not less than a certain amount of finely-ground matter, estimated by passage through the finest sieve in ordinary use, *e.g.*, that having 160 meshes per linear inch. When this requirement becomes general it will be time enough to consider whether the limit of the determination of fineness can be pushed with advantage beyond that set by a 160 sieve. In some special instances when it was desirable to have a means of separating the fine fraction of a cement isolated by sifting, into further fractions, we have successfully employed the plan of elutriating the material with a liquid incapable of acting chemically upon it, *e.g.*, turpentine, freed from dissolved water by distillation and drying over quicklime, or a mineral oil of low viscosity. Should it be found requisite to adopt any such method in the ordinary testing of cements, to discriminate between samples containing various proportions of "flour," it will become necessary to arrive at a standard method of applying the test to secure comparable results. The contingency is not, however, immediate, though the test is a useful one in certain cases.

Reverting to the customary method of determining fineness, only a few points claim notice. The first is that accidents in wiring or stamping sieves occasionally occur, and it is therefore prudent to verify the number marked upon the sieve by counting the wires, if need be, by the aid of a lens. The next is that a standard wire, as well as a standard number of meshes per linear or square inch, should be adopted, as otherwise sieves identical in title may give diverse results. The third relates to the manner of ascertaining the results of sifting. It is generally preferable to weigh the portion that remains on the sieve rather than that which passes through, as some loss from the latter is almost inevitable, sifting cement being a very dusty operation. It is probable that this canon holds good in the case of the determination of the "flour" of the cement, *i.e.*, the portion which passes through the finest sieve, an estimation by difference being for once in a way more trustworthy than a direct determination. Nevertheless, the direct weighing may be profitably used a check.

**Weight per Unit Measure and Specific Gravity.**—These properties of a cement are intrinsically distinct, but are frequently confused. The practice of making a determination of the weight per cubic foot, or more commonly weight per bushel, doubtless arose from the fact that a convenient method of bringing the weight and apparent volume of cement into commensurate terms is a necessity for engineers computing the requisite quantities of structural materials, as witness the wide use of the weight per cubic foot of the most diverse substances, from copper to asphalt. When it was observed that an apparent unit volume of cement varied in weight according to its quality, a natural attempt was made to deduce quality from weight per apparent unit volume. Hence the now standard requirement that a Portland cement shall weigh a specified number of pounds per bushel. As long as all cement was ground to about the same degree of fineness this system proved moderately effective, when made workable by the use of a standard hopper and measure for

the determination of the weight of a struck bushel, but as a demand for finer cement arose, it was found that the weight of 115 to 120 lbs. per bushel that once was easily reached, could no longer be readily attained. In short, the weight per bushel depends, other things being equal, on the fineness of the cement, and a sliding scale for the weight per bushel regulated by the specified fineness becomes necessary, transforming a test of simplicity and fair accuracy into one cumbersome and inexact. Fortunately, the whole difficulty is removed by the substitution of the determination of the specific gravity for that of the weight per bushel. The latter is properly retained for making a series of rapid tests capable of execution by any fairly intelligent person acting as inspector of cement at a works to ascertain that there is at least tolerable uniformity throughout a heap of many tons. The former can be best applied in the laboratory where the testing and analysis of the cement are carried out.

The average weight of fresh, fairly finely ground Portland cement (*e.g.*, leaving a 10 per cent. residue on a 76 sieve) is 108–110 lbs. per bushel. The specific gravity of the same sample will be about 3.15. The apparent specific gravity deduced from the weight per bushel is 1.37. The difference is so great that there is a large margin for an alteration of the weight per bushel by the closer packing of the cement particles, and it is easy to see how considerable an error may exist. The determination of the true specific gravity of cement is not free from pitfalls on its own account, especially when it is carried out—as is often the case—by comparatively unskilled persons with somewhat crude apparatus. Owing to the circumstance that hydraulic cements are necessarily acted upon by water, that liquid is obviously inadmissible for use in the determination of their specific gravity, and some fairly mobile and not very volatile liquid, such as petroleum or spirits of turpentine, must be substituted. It is of the first importance, whatever liquid be adopted, that it be perfectly dry. Mere freedom from turbidity is no guarantee that all water is absent. Complete dehydration must be secured by allowing the liquid to stand over good "fat" quicklime or similar dehydrating agent which is unacted on by the liquid itself. With regard to the method to be used, it may be said that the ordinary specific gravity bottle affords as accurate a result as can be wished, but necessitates a knowledge of the specific gravity of the liquid employed. The plan of determining this value for a large supply of the liquid, and using it for a series of determinations extending over a long time, is not to be recommended, as slight changes may occur on keeping (particularly in the case of turpentine, the specific gravity of which increases by re-refinement), and give rise to an appreciable error. On this account, and because of the necessity of operating at a standard temperature, the use of the specific gravity bottle is generally given up in favour of a volumometer, in which the displacement of a known weight of cement is read directly. The ordinary Schumann apparatus, consisting of a graduated glass tube ground into a bottle, is a rough apparatus of this kind. The apparatus is filled with turpentine or petroleum to the zero mark and a known weight of the cement, usually 50 or 100 grms., shaken in through a funnel down the graduated tube, the liquid displaced rising in the graduated tube, where its volume can be read. In this case the weight taken is fixed and the volume variable. A second instrument on the same principle, but in some respects preferable, is that shown in Fig. 1. Here the upper bulb is of known capacity (*e.g.*, 1,000 grains) between the fiducial marks A and B. The vessel is filled with liquid to the mark A, weighed, and cement is introduced until the level of the liquid reaches B, when it is weighed again. In this case, therefore, the volume is fixed and the weight variable.

An improved form of the Schumann apparatus which we were at one time in the habit of using, is that illustrated at Fig. 3.

A side tubulus carries a small thermometer, and the main reservoir is fitted to a broad wooden base to increase its stability.

In the two forms of Schumann apparatus the serious fault exists that the cement must be introduced after the liquid down a long tube, the natural result being that there is a great tendency for the cement to stick to the sides of the

tube and form obstructive plugs just at the surface of the liquid, which are sometimes difficult to dislodge. This evil cannot be overcome by an increase in the width of the tube without introducing a large reading error. Conversely, close reading is impracticable without so diminishing the

Fig. 1.

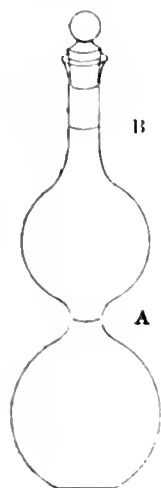


Fig. 2.

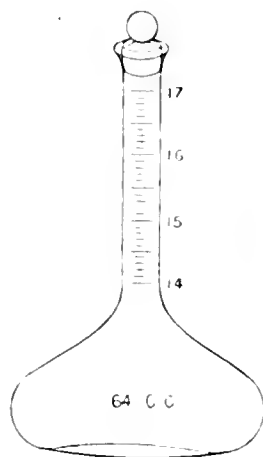
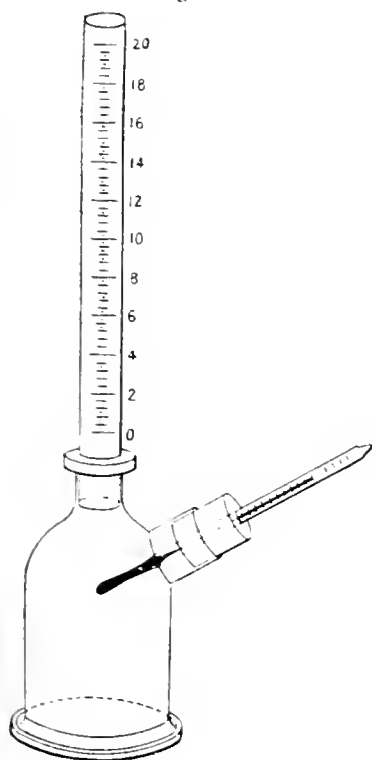


Fig. 3.



diameter of the graduated tube that the introduction of the cement becomes well-nigh impossible. The bulb form of apparatus illustrated in Fig. 1 is better in these respects, but necessitates two weighings in place of one.

For rapid routine work, we have devised an extremely simple form of volumenometer which is free from most of the defects of those generally made. It consists, as will be seen from Fig. 2, of a small flask very much flattened

in shape so as to ensure its stability, and provided with a short slender neck with fine graduations, not ground into the lower part of the vessel, but blown in one piece therewith.

The lowest graduation mark on the neck is not 0 but 14 cc., and the capacity of the flask to the first graduation mark is known and equals 64 cc. The instrument is used by running into it 50 cc. of turpentine from a pipette which has been accurately calibrated against the flask. As the turpentine is introduced by means of a pipette the neck of the apparatus is kept dry, and the known weight of cement (50 grms.) can be readily added through a small funnel without blocking the tube. The shortness of the neck conduces to the easy introduction of the cement. The plan of first placing a known weight of cement in the flask and then running in 50 cc. of liquid appears at first sight preferable to the method described above, but the complete displacement of the air entangled in the cement is less easily accomplished than when the cement is caused to shower down through the liquid. The first 14 cc. represents that portion of the displacement of the cement accommodated in the body of the flask which, as stated above, has a capacity of 64 cc. to the lowest graduation mark; the remainder of the displacement is registered on the stem. The temperature of the turpentine must be known, but need not be fixed at any standard point, and after the operation the volumenometer (which is stoppered to prevent evaporation) is brought to the same temperature by immersion in the same vessel of water as that previously used for the stock-bottle of turpentine. As all ordinary Portland cement has a specific gravity, such that the displacement of 50 grms. falls between 14 and 17 cc., this range of the graduations on the stem is ample. It is obvious that in dealing by means of this instrument with any material differing largely in specific gravity from 3, more or less than 50 grms. can be taken so as to bring its displacement within the range of the graduations on the stem. Seeing that in all cases the temperature before and after the displacement of the liquid by the cement must be identical, because of the high coefficient of expansion of turpentine and petroleum, it is evident that an apparatus of small total capacity, such as that just described, is convenient, as it can be immersed bodily in a beaker of water and brought to the proper temperature with ease and rapidity. In this, as in the forms of volumenometer previously described, the indications of the instrument are in no way dependent on alterations of the specific gravity of the liquid selected that may take place on keeping, so that a large supply of dry turpentine or petroleum can be prepared at one time and used to the last drop without further examination.

The deductions to be drawn from the specific gravity of a cement are various, and are modified by a knowledge of its other properties. It may be taken as a safe rule that the specific gravity of a true Portland cement of good quality, fresh from the stones, will be not less than 3.15. The same cement on exposure to air in a thin layer for a week may have decreased in specific gravity to 3.10, and further decrement is possible on continued exposure. The aeration to which the sample has been subjected must therefore always be taken into consideration in judging a cement from its specific gravity. It follows from this that the specific gravity of a cement will be found to decrease as the quantity of volatile constituents absorbed from the air (water and carbonic anhydride) increases. Indeed, experience enables the analyst to predict the specific gravity of a cement with fair accuracy from its analysis, provided the composition fall within ordinary limits. With cements of less regular composition than Portland cement a greater range of specific gravity naturally occurs. So-called natural cements, *i.e.*, hydraulic cements made by burning argillaceous limestone of approximately the same composition as slurry, come nearest to Portland cement in this respect, and are followed at some distance by Roman cements which have a specific gravity of 2.5–3.0. In cements of the puzzolana class, including slag cement, the specific gravity depends more on accidental circumstances than on the intrinsic quality of the material, and is therefore rarely of much importance.

*Time of Setting.*—The setting of hydraulic cement being a gradual process, methods for defining its period must be more or less arbitrary. At present the Vicat needle and its modifications still hold undisputed sway, though at least one ingenious apparatus has been devised for giving an autographic record of the setting process. Only two points need notice here. The first is that most cements are rendered slower setting by keeping, consequent on the hydration of the calcium aluminate, to the presence of which the initial setting is to be attributed, and therefore the setting time of a cement tested in this country and then abroad, after a lengthy voyage, will differ, being longer in the latter case. Want of recognition of this fact has often led to trade disputes. The second is that the temperature of the room in which the cement is exposed has a serious influence on the speed of setting. A higher temperature means more rapid setting. As no definite relation can be traced between the temperature and the time of setting, specifications in which stress is laid upon the time of setting falling within certain limits should state the temperature at which the test is to be made, and the cement tester should take steps to attain and maintain that temperature, by artificial means if needful.

*Strength in Tension and Compression.*—So much has been written about the testing of the mechanical strength of cement, and the information is in such accessible form for all interested in the subject, that it would be useless and wearisome to recapitulate what is a matter of common knowledge. We will confine ourselves to indicating what are the streams of tendency in this country at the present time. In the first place, the righteous condemnation of the demand—which demand is now on the wane—for an excessive tensile strength at short date, has carried some of its upholders to the opposite extreme, and led them to look with suspicion on cement merely because it is above the average in strength. It is surely unreasonable to condemn a material on this ground, always provided that its reliability be indisputable. (The means in use for deciding upon the safety of an hydraulic cement will be dealt with under a separate head.) Secondly, the substitution of compressive for tensile tests has made but little progress, and is unlikely to make much more. The latter method may be illogical, but is certainly convenient. In the third place, the use of sand in place of neat tests is growing daily, and will continue to grow as the demand for a more finely ground cement increases. In neat tests the full cementing power, as distinct from cohesive value of a cement, never comes into play, and a comparatively coarse sample may give tests as high as those afforded by one unusually fine. With sand, on the other hand, the property of surrounding, coating, and uniting inert particles (*i.e.*, the sand) is exercised, and the finer of two cements equal in other respects will give the better result. In view of the general increase in fineness of cement, it is not improbable that a sand test with a higher proportion of sand (1 of cement to 3 of sand being at present usual in this country) may be adopted with a corresponding advantage for finely-ground cements. Another useful innovation would be the insistence on mechanical tests of concrete made of the same sand and aggregate as that intended to be used for any given undertaking. The best cement may be rendered useless by admixture with bad sand and aggregate.

*Tests for Soundness of Cement.*—Owing to the fact that the completion of all the reactions initiated by contact of cement with water takes place only after the elapse of a considerable time—often extending to many months—the mechanical properties of ordinary test pieces when determined within a few days or weeks after the date of their preparation, are no certain criterion of the reliability of the cement. This circumstance has led to the device of special tests intended to meet the need of arriving at the same results in a short time as would eventually occur after the elapse of a long period.

The natural suggestion that any chemist would make, on being confronted with this requirement, is that the test pieces should be kept under conditions that would hasten the completion of the reactions occurring in them, and that an increase of temperature would be likely to effect this

purpose. This idea underlies all the varieties of "hot test" that have been proposed and practised from time to time. In its simplest form the "hot test" may consist in merely heating a pat of neat cement in an air bath to a moderate temperature (*e.g.*, 160° F.) and observing whether it buckles or develops any cracks, and whether it shows signs of weakness when wetted after heating. A distinct advance on this is achieved by exposing pats of neat cement to a warm damp atmosphere, or actually immersing them in warm water. Although it cannot be said with certainty that results are obtained by such tests identical with those that would be arrived at by efflux of time, yet it may be safely asserted that any thoroughly unsound cement will be detected and nearly all sound cements will be passed. It is not our purpose to discuss here the causes of unsoundness of cement, for such discussion would be foreign to the subject of this paper, but it may be mentioned that in nine cases out of ten a cement fails because it has been made from an imperfectly burnt clinker, and contains a certain amount of unsaturated base, which is conventionally known as "free lime." No one has yet succeeded in determining free caustic lime in cement, the problem being one of extreme analytical difficulty, but there is good reason to believe that unsaturated lime exists in badly burnt clinker, and, therefore, in the cement produced from it. Consequently the phrase is defensible on the score of convenience.

A system of hot testing which gives a quantitative expression of the unsoundness of a cement is that devised by Deval, and fully described in this Journal (1891, 255). From the results of a long series of experiments with various kinds of hydraulic cement, Deval established the fact that in most cases a sand briquette (1 : 3) kept in water at a

1 Cement : 3 of Sand by Weight.		
No. of Sample.	28 Days Cold.	7 Days Hot.
	Average tensile strength, lbs. per sq. in.	
*1	162	19
2	163	Nil
3	153	"
4	165	"
5	247	"
*6	..	137
(made abroad).	217	137
7	217	137
8	186	Nil
9	222	20
10	257	52
11	165	42
12	197	193
13	173	37
14	183	152
*15	217	43
16	198	10
17	185	19
*18	182	Nil
19	187	25
20	218	178
*21	177	Nil
*22	247	150
23	227	140
*24	228	107
(made abroad).	252	221

\* Those marked with an asterisk were analysed.

temperature of 176° F., 80° C., for two days has the same tensile strength as one kept in water at the ordinary temperature for seven days, while a similar relation holds good for briquettes kept in water at 176° F. for seven days and those kept at the ordinary temperature for 28 days. This is found to be generally true of normal hydraulic cements of good quality. In the case of an indifferent

cement the hot test pieces have a lower tensile strength than those exposed to the ordinary temperature, while those of an unsound cement crack and disintegrate.

At the date of the introduction of this method of testing we made a series of experiments on cement of various makes, the results of which are indicated in the table on preceding page.

ANALYSIS OF CERTAIN OF THE SAMPLES IN THE ABOVE TABLE.

Running Number.	1.	6.	15.	18.	21.	22.	24.	25.
Insol. res. ....	0.56	5.44	0.70	1.18	0.78	0.68	0.76	1.52
SiO <sub>2</sub> .....	10.96	24.80	20.82	10.46	21.00	20.30	20.08	21.98
Al <sub>2</sub> O <sub>3</sub> .....	9.50	3.80	7.04	8.14	11.88	8.32	9.34	6.32
Fe <sub>2</sub> O <sub>3</sub> .....	3.22	1.64	3.54	3.68			3.94	3.92
CaO .....	62.98	55.46	64.00	62.14	60.94	62.52	61.16	60.92
MgO .....	6.98	2.20	1.01	0.82	1.16	1.01	1.16	1.44
SO <sub>3</sub> .....	1.42	1.28	1.13	0.36	1.59	1.12	1.08	1.14
CO <sub>2</sub> .....	1.61	3.61	0.96	1.32	0.82	0.80	1.40	2.37
H <sub>2</sub> O .....								
Alkalis and loss. ....	0.17	1.77	0.80	1.30	1.00	1.60	0.88	0.42
Specific gravity .....	3.14	2.90	..	3.16	3.15	3.13	3.14	3.105

The upshot of these experiments was that but few English cements made in the Thames and Medway district could be relied upon to stand the test unless previously aerated. The cause of this result is probably the difficulty of preventing underburnt clinker (of which a certain proportion occurs in most charges as drawn from the kiln) finding its way to the stones, and giving rise to the presence of some quantity of unsaturated base ("free lime") in the cement. When underburnt clinker is eliminated the test is most useful in discriminating between cement of normal composition and that containing unsaturated lime or other base. We have used it extensively in determining the quality of raw materials for cement making, and found it of great value. The following samples of cement were made in our laboratory:—

No. of Sample.	1 Cement; 3 of Sand by Weight.	
	28 Days Cold.	7 Days Hot.
Average tensile strength, lbs. per sq. in.		
*1 (burnt with gaseous fuel at a very high temperature).	347	295
*2 (overlimed) .....	43	Nil
*3 (badly mixed) .....	158	"
*4 (bad raw materials) .....	Nil	"
*5 " " .....	"	"
*6 (bad raw materials improved by washing).	400	365
7 " " " .....	300	243
8 .....	207	170
9 (from cement stone) .....	90	Nil

\* Those marked with an asterisk were analysed.

Analyses of some of the above samples are given in next column.

With regard to this series of tests it will be observed that a percentage of lime that would be considered excessive in cement manufactured on a large scale is no bar to perfect strength and soundness in one that has been burnt with the thoroughness possible in a laboratory trial. Samples 1 and 6 may be especially noticed. This circumstance goes

Running Number of Deval Tests.	1.	2.	3.	4.	5.	6.
Insol. res. ....	0.64	0.66	0.28	0.72	1.04	0.38
SiO <sub>2</sub> .....	20.80	20.58	20.06	28.80	28.60	21.48
Al <sub>2</sub> O <sub>3</sub> .....	7.00	5.26	8.12	8.54	7.40	5.02
Fe <sub>2</sub> O <sub>3</sub> .....	4.46	2.04	3.18			
CaO .....	65.44	65.74	66.38	60.46	61.20	60.60
MgO .....	0.89	0.97	1.13	0.54	0.94	1.79
SO <sub>3</sub> .....	0.03	Trace	Trace	Trace	Trace	Trace
CO <sub>2</sub> .....	0.33	2.51	Trace	Trace	Trace	Trace
H <sub>2</sub> O .....						
Alkalis and loss	0.01	2.84	0.55	0.94	0.82	0.35

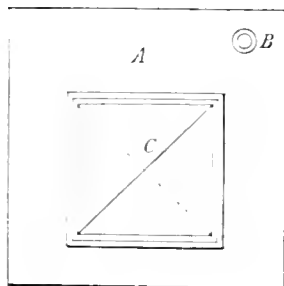
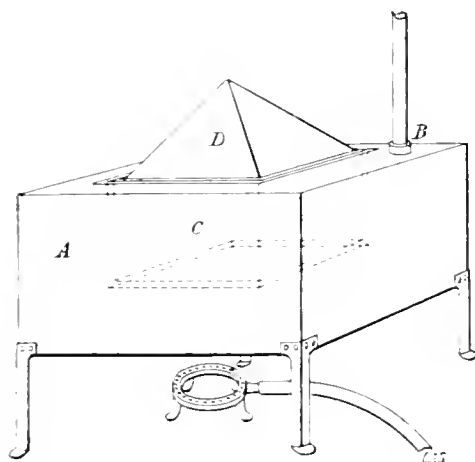
to show that uneven and defective burning is one of the chief causes of imperfection in commercial cement, and indicates in what direction improvement is chiefly to be expected.

For carrying out the Deval test, a bath of water kept at 80° C. = 176° F. is essential, as the operator is not justified in departing from the exact prescription of the originator of the test unless he be prepared to establish relations corresponding with those worked out by Deval between hot and cold tests, for such other temperature as he may select. For this reason all attempts to substitute temperatures more easily maintained, e.g., 100° C. = 212° F., for that adopted by Deval, cannot be usefully discussed while the data to support them are lacking. It being conceded that a temperature of 80° C. is essential to the proper execution of the Deval test, it remains to ascertain the most practicable method of maintaining this temperature with reasonable constancy. The use of an ordinary thermostat is not wholly satisfactory in a case of this kind. The hot bath to contain the briquettes is more a piece of testing-room plant than a laboratory apparatus, and its management is not necessarily in the hands of a chemist. Glass thermostats are therefore rather too delicate for the purpose in hand. For a large tank used in carrying out the Deval test, we first constructed an air thermostat of the usual design for this class of instrument, with a long copper tube

coiled in the bottom of the bath so as to give a good-sized expansion chamber, and a steel tube bent to form a U to contain the mercury column controlling the gas supply. This worked very fairly, but was somewhat troublesome to set up and adjust, owing to the opacity of its parts, a drawback always felt by a chemist when he has to abandon the use of glass as a structural material. It also possessed the disadvantage common to most thermostats, viz., inadequacy of controlling power when dealing with large fluctuations in the pressure of the gas supply (e.g., differences of as much as  $1\frac{1}{2}$  to 2 ins. of water). In fact, a thermostat to be perfectly reliable, needs a governor on the gas supply to remove the effect of large differences of pressure, and in fact to do the rough work of control.

Having regard to these facts and also to the need for a Deval apparatus capable of use on cement works where no gas is to be had, we designed the apparatus shown in Fig. 4.

Fig. 4.



about to be stated. The principle underlying the working of the apparatus is simple enough. The outer bath, which, as it is provided with a good condenser, can be kept boiling vigorously, constitutes a source of heat at a constant temperature,  $100^{\circ}\text{C}$ . The inner bath is separated from the outer by a constant distance, which offers a constant obstruction to the transmission of heat from the outer bath. The temperature of the inner bath is therefore always less than that of the outer bath by a constant number of degrees. The size of the separating space must be determined empirically. It is best made sufficiently great to ensure the temperature of the inner bath being somewhat lower (when the space is empty) than that ultimately to be obtained. The obstruction offered by the separating space is then gradually diminished by introducing into it successive small portions of any convenient substance in grain or powder, e.g. shot or sand. By a few trials a point is reached when the obstruction to the transmission of heat from the outer to the inner bath is so adjusted that when the former is at  $100^{\circ}\text{C}$ , the latter is at  $80^{\circ}\text{C}$ . It is obvious that fluctuations of the gas supply, provided that it be always large enough to keep the outer bath boiling, have no effect on the temperature of the outer bath, and therefore are equally without effect on that of the inner bath. The only change of external condition likely to effect the temperature of the inner bath to an appreciable extent, is that of the temperature of the room in which the apparatus is placed. When the air temperature rises, loss of heat from the pyramidal cover, which is the only portion exposed to the atmosphere, naturally diminishes, while the supply of heat from the steadily boiling bath is maintained at its former rate; consequently the temperature of the inner bath rises slightly. The converse is true on a fall in the temperature of the air taking place. Experience has shown that the fluctuations due to this cause are not large in an ordinary laboratory atmosphere, of which the temperature does not vary greatly; they might, however, become considerable if the bath were used in an unprotected shed or other slight building. The apparatus in the form shown is quite independent of any particular method of heating. It can be used with a paraffin lamp as well as with a gas burner, the sole condition being that the source of heat shall always be sufficient to keep the water boiling briskly in the outer bath.

We have dwelt at some length on the principle employed in this apparatus because it is, as far as we know, one that may be applied for many purposes through a considerable range of temperature, the essential features being (1) a source of heat of constant temperature, such as is obtained by the use of a liquid for the outer bath having a definite boiling point; (2) a space between the outer and inner bath offering a constant resistance to the transmission of heat; and (3) a fairly even air temperature, so that the loss by radiation (which may be made small by minimising the area of the radiating surface) may not fluctuate largely.

Many hot tests, other than that due to Deval, have been devised, but the only example that need be quoted here is that known as Erdmenger's high pressure test. It consists essentially in exposing briquettes to the temperature obtained in a closed boiler carrying a pressure varying from 10 to 40 atmospheres. It is claimed that sound cement rapidly attains the strength at which it ultimately, after a period of weeks or months, arrives under normal conditions, and that an unsound cement—especially one containing an excess of magnesia—acquires a low tensile strength or disintegrates, according to its degree of faultiness, and the temperature to which it has been exposed. The establishment of quantitative relations as definite as those existing in the case of the Deval test does not appear to have been effected with the Erdmenger test. It is not quite apparent what purpose is served by using the high temperatures and consequent high pressures that distinguish the Erdmenger test from others of its kind. Indeed the temperature, which at 20 atmospheres is  $212^{\circ}\text{C}$ . =  $415^{\circ}\text{F}$ ., may be so high as to cause the dissociation of the products of the hydration of the cement to which its character as a cement is due. Such dissociation may occur even in an atmosphere consisting of one of the products of dissociation, i.e., water, an inference borne out by the statement that

A is a rectangular copper water-bath completely closed save for a small hole B, to which a condenser can be attached. This condenser may be a straight metal tube, jacketed with water like a Liebig condenser, or, if space permit, a piece of lead or "compo" pipe, 10 feet long or so, passing out of the room containing the bath and going up the outside wall to a neighbouring chimney. An air condenser of this kind is cheap, convenient, and, if long enough, quite effective even in hot weather. The tube must be vertical, or have at least a sharp rise, and be free from any kink or obstruction, or the return flow of the condensed water will be impeded. The top of the water-bath A is not flat, but has a rectangular depression or well into which the hot bath C, to be maintained at the required temperature, fits, clearing the walls of the well, and supported by a flange on the top of the bath A. A pyramidal cover D is provided for the bath C, and, if fitting well, suffices to condense such water vapour as may be given off at  $80^{\circ}\text{C}$ . The space between the inner and outer bath is either empty or filled partially or entirely with sand or shot, for a reason



long exposure at high temperatures in the Erdmenger apparatus is capable of causing the deterioration of cement of admittedly good quality. With regard to the detection of magnesia, actual analysis must be in every case preferable to any mechanical method of testing, however drastic and ingenious, and as the permissible limit for magnesia is now pretty well defined, no exception to the exclusive use of analysis for this purpose can well be taken. A highly practical objection to the Erdmenger test may also be urged, viz., that it is doubtful whether good Portland cement as made in this country can be relied upon to pass it; it is generally conceded that the Deval test errs, if anything, on the side of severity, and until a stage of manufacture has been reached capable of producing cement which can pass the milder test, it is useless imposing a more exacting criterion.

**Analysis.**—The actual analysis of hydraulic cements is not a difficult matter, and to detail such methods of separation and the precautions in using them adopted by us would not be of sufficient interest to compensate for the space which the description would occupy. It is sufficient to point out a few of the pitfalls that beset the tyro in matters of this kind.

Thus it is futile to attempt to determine "free lime" by any method which does not take into account the fact that the cementitious compounds proper to hydraulic cements are themselves decomposed by water, and therefore yield an alkaline extract with any aqueous liquid. As far as we are aware no reliable method of determining "free lime" in an hydraulic cement has ever been published. There is good reason to believe that an extremely small quantity of "free lime" (1 per cent. or less) is present in Portland cement of good quality, and analyses showing anything in excess of this should not be accepted without close scrutiny. The separation of the silica—as distinct from the insoluble silicates, sand, &c., which amount to not more than 1.5 per cent. in an ordinary Portland cement—into "soluble" and "insoluble" is a purely arbitrary distinction turning merely on the particular conditions of working adopted by the analyst. The whole of the silica separated by the decomposition of the cementitious silicates by means of hydrochloric acid is "soluble," and no distinction in function between different portions of it has yet been successfully drawn. The return of "moisture," which is sometimes set down in cement analyses, is an error too obvious to need comment. It would be nearly as rational to return "moisture" in a sample of quicklime. In the analysis of cement, as in that of most siliceous and earthy materials, especial attention must be bestowed upon reagents, filter-papers, and vessels, lest small quantities of silica, alumina, and lime creep in during the course of analysis and cause an apparent creation of matter when the analysis comes to be added up. A similar surplus sometimes arises from the insufficient ignition of precipitates that are not easily brought to a constant weight, e.g.,  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , and  $\text{CaO}$ . For such substances we invariably use a gas-muffle. Finally, it must be remembered that the best possible analysis of cement is not proximate, the separation of the actual compounds forming the cement, to which its properties are due, being still beyond the range of analysis. A double measure of caution must therefore be used in arriving at a judgment from data that are necessarily not exhaustive.

In conclusion it may be said that neither by the analysis nor mechanical testing of cement singly can its quality be gauged with complete certainty. It is imperative that both chemical and mechanical data should be available before a positive pronouncement can be made. Although experience allows a valid opinion to be passed even when such complete data are not forthcoming, yet the removal of the opinion into the ranks of ascertained facts can only be accomplished by determining both the composition of a cement and its leading physical properties. When both have been ascertained, the suitability and safety of a cement for any given purpose can be accurately adjudged.

#### DISCUSSION.

Mr. W. HARRY STANGER said that, until his association with Mr. Blount, his practice was entirely confined to the testing

of cements by mechanical methods. The results were, never, in his opinion, absolutely reliable; with high unquestionably in trained and experienced hands, they were sufficiently so to ensure a just appreciation of the merits of the sample under test. The main difficulties in the way were the various shapes of "standard" bushed measures, the means used for filling the measures, the grading and mixing of the cement, and the speed of application of the load. Bushed measures (all Imperial stamped standard) varied in shape from shallow tubs 8 in. deep by 19 in. diameter to cylinders 15 in. deep by 13 in. diameter. Each had a bushed by measure, but as the weight also of the measured material was wanted, the shape of the measure itself was an important factor when dealing with so compressible an article as Portland cement. The measure and hopper exhibited were chosen and designed by Mr. William Mathews to ensure in all cases equal treatment, and this apparatus had now come into very general use. The mixing of the cement with water was an operation requiring "knack" and experience which were not always available, consequently the excellent device designed by Mr. Rajja was largely used. The automatic apparatus for applying the load to the briquettes was also the invention of Mr. Mathews, and by its means the accuracy of cement-testing machines had been immensely improved. Sufficient attention had never been given to the construction of the sieves. He had tested many sieves, and he could not remember a single case in which the number of holes stamped upon it by the maker agreed with the number per square inch it actually possessed. The wires used varied considerably, and he knew of cases in which sieves alleged to be "40" "40," *i.e.*, 1,600 holes to the square inch, neither possessed that number, but actually had holes smaller than sieves stamped "50" "50," *i.e.*, 2,500 per square inch. He mentioned the above facts as his instruction for calling in the assistance of the chemist. By the improvements in the mechanical apparatus already mentioned, much had been done, and he was glad to find that engineers were beginning to grasp the fact that the manufacture of hydraulic cements was really a chemical process, that precision and accuracy in the testing of the product were essential, and that as it was necessary to know a great deal about the material which mechanical testing, however careful, could not suffice to determine. The services of the chemist were therefore indispensable. If all users of cement would follow in the lines of those thoughtful engineers who now called in the chemist's aid, we should hear of fewer failures and cease to be agitated by periodical scares.

Mr. W. F. RENN said this subject was by no means a new one, and had been pretty well thrashed out, but still, even this paper did not contain some of that which was newest in this particular branch. With regard to the coarse particles left on the sieve used for testing cement, it had been known for a great many years that those were comparatively useless. In fact, Michaelis, in his book in 1869, said that they were inert and as useless as sand. He had stated practically the same thing about the same time himself. The question whether this fineness could be tested by means of a sieve was very important. Recent experiments in Germany tended to show that everything that went through the finest sieve was not really what you could call a cementing agent; that even through the finest silk gauze used in flour mills cement passed which was extremely slow in attaining its ultimate strength. That had been further separated by means of a current of air, and there was a very ingenious apparatus used in Germany for that purpose. They tested not only with a current of air but also adopted the method of elutriation, using a liquid not acted on by the cement. He would warn anyone who had not great experience against using turpentine for this purpose. He had frequently used it himself and found that it was a liquid of very variable specific gravity, generally containing a good deal of moisture, and it had a considerable action on the lime which was undoubtedly present in the Portland cement, and which, if not absolutely free, would react with anything with which it came in contact. The test by the weight per bushel was originally introduced by Mr. Grant, assistant engineer of the Metro-

politan Board of Works, who, he believed, took it from a French engineer. It was said in the paper that it doubtless arose from the fact that it was a convenient method of comparing the weight with the volume and was therefore a convenience to engineers in prescribing the requisite quantities. But that was not the case. One reason why it was introduced was to distinguish Portland from Roman cement. The first makers of Portland cement were not quite sure of the quality, and he knew on very good authority that they frequently mixed a little Roman cement with it to bring it up to the required strength. Of course that was perfectly ridiculous now, but in those early days it was sometimes a toss up whether a kiln was going to be good or bad. Engineers then thought it was sometimes necessary to distinguish Portland from Roman, and when mixed it was very difficult; but this was a means of doing so. Another reason which prevailed then—though it might not now—was light-burning. There was an incentive to light-burning, and although one could discover whether cement was light by its tensile strength, no doubt a good deal of light cement did come into the market at first. The weight test as a test—and there was an apparatus shown for carrying it out—was absolutely valueless, and if he had been writing on the subject he should have said weight per bushel was an old test which was now useless. A coarser-grained cement weighed heavily, and the same cement finely ground, and hence more valuable, weighed lighter. In the present state of knowledge, if such a test were specified at all, it ought to be that cement should not weigh more than so many pounds per bushel, but no engineer thought of doing that. The difference in the weight of a coarse and fine cement had been stated by a well-known French engineer to amount to as much as 20 per cent. on some samples. That is to say, you could take a cement which weighed so many pounds per bushel and grind it until it weighed 20 per cent. less per bushel. With a margin of error like that, it was perfectly impossible to attach any importance to the figures. As Mr. Blount had said, the specific gravity of all cement was about the same; there was only a comparatively small range of difference. Much importance was attached to this test by Fresenius, who regarded it as one of the best means of distinguishing between cement adulterated with slag and true Portland cement; but this test had since been abandoned, as it was found useless for practical purposes. It was very interesting scientifically, and the little apparatus shown would no doubt give the specific gravity with a minimum of trouble. He never took the specific gravity unless requested, though he had tested and made many hundreds of thousands of tons of cement. He agreed with the authors with regard to the importance of sand testing, and the time of setting was also extremely interesting from a theoretical point of view. From a practical point of view, however, it was absolutely necessary to get a simple test which could be applied to all cements by almost anybody in the habit of conducting such operations. If the ingenious apparatus shown on the table were required for ascertaining the time of setting, he feared it would add considerably to the cost of the installation of a laboratory at a cement works and less cement would be tested where it ought to be—by the makers. Testing with sand was certainly the best method known of ascertaining that which was the chief object of a cement—its cementitious value. The difficulty here arose that you introduced another substance which was in itself variable. No two sands were alike. In Germany they got over that difficulty in the usual way, by standardising it. There was a normal sand, a normal testing station, and a normal price, but we had not arrived at that stage here; there was no sand which had been agreed upon by engineers or any public body which could be taken as a standard sand. It was not enough to take sand of a definite size, or even pure quartz sand of a definite size, because much depended on the shape of the grains, and as there was no uniform sand there was a great difficulty about this test. Mr. Grant, who had done so much for the testing of cement, tried a great number of experiments and found nothing so uniform as quartz sand, and he used sand from a certain place and recommended it to all his friends; but you could not say there was any standard sand. With regard to the

heat test which was ascribed to Deval, it was not his originally. It was first used by Michaelis and then by Edmenger. He had worked at it for the last 15 years. He tried it to ascertain the effect of magnesia on Portland cement, which was a subject which had been very much talked about lately, and he found that where he used a high temperature, superheated steam, he could detect the presence of magnesia. It was not generally adopted in Germany by experts, and in France it had been rejected for the following reason:—Several factories, whose cement was undoubtedly excellent, and which was used largely by the Government and found to be thoroughly excellent, could turn out no cement which would stand this test. The cement could not be wrong, as it had been used for a number of years satisfactorily, and therefore the heat test must contain some latent defect, probably because in a short time the cement had to exert an action which ought to have been distributed over a much longer period.

Mr. BURNAYS said he had lately had a good deal of experience in using cement, and had to thank Mr. Blount for making many special tests for him. He was a little frightened at some statements in the paper, because he found most of the tests hitherto recommended for use more or less run down by the very gentlemen who had either invented or improved upon them. Engineers were working as far as they could in accordance with what they had learned from specialists, but one after another those tests which had frequently been considered good were now deprecated and something new was presented for their acceptance. In their ordinary practice they ascertained the weight per bushel, the tensile strength, and the fineness, with some precautions, and found that the result was very uncertain. Sometimes they got a cement which bore the weight test well; at other times, from the same makers and made, according to their statement, in exactly the same way, they had a cement which weighed very differently, and when they came to try the cement by sifting it and taking the amount of the residue they found it very different from the previous sample. Then they made tests with briquettes, breaking them with the machine and obtaining at times very extraordinary values. Sometimes from one sample of cement they got briquettes which would after seven days break at perhaps 250 lb. per square inch; another briquette from the same set would break at 500 or 550 lb., and they had not the slightest notion how the difference could arise. But the most curious thing of all was that, in spite of all these varying results, all the cements they had used were of excellent quality, whether the test had turned out well or not. They were all cements which came from a good firm, were made to comply with a given specification, and they made first-class foundations, as hard as steel, and as lasting, he believed, as the earth itself.

Mr. MATTHEWS said he had been a user of cement during the last 25 years, and his experience did not in many respects coincide with the remarks of the first speaker. First, with regard to the hopper and bushel measure, he found such an apparatus extremely useful in determining the weight of cement. Many years ago he used a cement which, when sifted through a 40 × 40 mesh sieve with a 10 per cent. core, gave an average weight of 120 lb. per bushel. Now cement was ground to give 10 per cent. core on a 50-mesh sieve, thereby reducing the weight per bushel to 116 lb. If they employed a 76-mesh sieve they had to still further reduce the weight to 110 lb. If you expected to get the same weight per bushel with a fine as with a coarsely ground cement you would be altogether disappointed. He had used the bushel and hopper for many years and found that by careful handling it gave results as to weight, with cement of the same fineness, within 2 lb. per bushel. When an inspector was sent to a works to examine a parcel of cement he would sometimes be shown a heap of 400 or 500 tons. Instead of getting two or three pinches to try the specific gravity he would take a bushel from different parts of the heap, and by that means get a fair idea of the average weight of the cement, thus showing whether the clinker from which the material had been ground was properly burnt. The main object of the weight test was

to show, before proceeding with further tests, whether the underburnt or yellow clinker, which was, he took it, the dangerous element in cement, had been picked out and cast aside, and that only well-burnt clinker, which gave a good sound cement, had been ground. He agreed with the authors of the paper generally as to the desirability of fine grinding. They were all tending in that direction. A very fine sieve was, however, a delicate thing. When the authors referred to a sieve of 160  $\times$  160 meshes to the inch it must be evident that the wire in it would be extremely fine, and the slightest damage to the sieve would upset the results. Therefore he looked in some other direction, possibly in that at which the authors hinted in their paper—towards an improvement in the determination of the amount of the core, or those particles in the cement which were incapable of setting. It was a matter of great importance that the extent to which those particles prevailed should be determined. He did not think they always arrived by the sieve at what that proportion was in extremely fine cements, and some other process would no doubt be devised to determine this point. He had tried blowing air through cement and other means, with the idea of determining the amount of the core, but found difficulties arose. He quite agreed that the sand test was a valuable one, but for engineers as a guide in accepting cement it was not practically of much use. They could not expect makers to keep large quantities of cement under trial and unaccepted for 28 days, whilst sand tests were being made. The sand test, as a check to the used by engineers after the delivery of the cement, was useful, but was not capable of practicable application generally as a guide upon which to accept or reject cement. Aeration of cement before use was of the utmost importance as promoting soundness of work. He had used cement which had been in a dry shed near the sea for 10 months, and the effect of age and aeration was to reduce the weight per bushel to the extent of 12 lb. On instituting tests to ascertain whether or not the strength had deteriorated, it was found that, although the two days' test was slightly less, the seven days' test was fully maintained. As a matter of fact they kept that cement for pointing, and the best work on the job, as they found it was the soundest and most satisfactory material. He had recently made experiments to determine what was the effect of turning over cement on its bulk. He found that six turnings, allowing the cement to stand a short time between each, increased the bulk 6.25 per cent.; 12 turnings, 7.80; 18 turnings, a similar increase; thus showing that after about 12 turnings there was no increase of bulk. He believed that the increase of bulk consequent on three or four turnings was a positive gain to a contractor, because he bought his cement by weight and sold it by measure, so that the increase in the bulk would pay for the cost of turning. He hoped engineers would endeavour to induce contractors to adopt this practice, seeing the value of aeration in securing sound work. With regard to analysis, he held such an investigation of great importance, as determining the percentage of lime and other constituents of the cement. Where the soundness and permanence of structures so largely depended upon the satisfactory character of the cement, as is generally the case in these days of extensive employment of concrete, every precaution should be adopted in carrying out the tests and in placing the same in the hands of experienced men.

Mr. HUGHES asked Mr. Blount to explain more fully the analyses on the diagram, and to state which was the better.

Mr. BLOUNT, in reply to Mr. Hughes, said that the cement marked No. 6 was undoubtedly the better of the two, in spite of its high content of lime, because coarse sand had been eliminated from the raw materials by washing, and the clinker had been thoroughly burnt. With regard to what Mr. Reid had said, he (Mr. Blount) thought that on reading the paper Mr. Reid would see that there was substantial agreement on many points between them. The fact that coarse particles in cement had little cementitious value had long been known by the few, but it was only now becoming generally recognised, and for that reason particular stress had been laid upon it in the paper. He was interested in Mr. Reid's information concerning the origin of the weight per bushel test, and thought the explanation probable

enough. Whatever its origin, however, it had come into general use, and had to be taken into account in any paper dealing with the testing of cement. It was somewhat embarrassing to find that Mr. Reid viewed him as a defender of the weight per bushel test, when as a matter of fact his whole opinion and course of argument were for its replacement (as far as practicable) by the determination of specific gravity. With regard to what had been said as to the abandonment of the determination of the specific gravity, he could only regret that those called upon to pronounce upon the quality of cement, should deprive themselves of what, in his opinion, was a valuable criterion. It neither the weight per bushel nor the specific gravity was worth determining, the onus of devising a better test plainly rested on the critics of these methods of testing. A normal sand would doubtless be a convenience, and such a sand was already used by Mr. Stanger and himself. There was no ground for supposing that Deval claimed to be the originator of the hot test; the contrary was clear from what had been said in the paper. What constituted the cardinal merit of the Deval test was that it was exact and quantitative, its forerunners being merely qualitative, and based, moreover, on less cogent and complete experimental data. Personally he knew that, especially in pronouncing upon the quality of new raw materials for cement making, it was invaluable. The detection of magnesia by any method of mechanical testing was a poor substitute for the precision of its analytical determination.

The continual displacement of old tests by new, and the apparent contradictions involved, mentioned by Mr. Bernays, were a necessary incident of progress.

In reply to Mr. Matthews' criticism, based on the use of a small quantity of cement for the determination of specific gravity in place of the hundredweight needed for the weight per bushel test, he would say that it was an easy matter and one of everyday experience to obtain a pound or so of material that should be absolutely representative of a thousand tons or more. Sampling, properly conducted, would remove the risk of the portion taken for the specific gravity not fairly representing the bulk. Given a representative sample, the error in the determination of specific gravity would be less than 0.2 per cent., whereas that of the weight per bushel was, as Mr. Matthews had mentioned, about 2.0 per cent.—10 times the former value. He was much interested in what had been said anent the sizing of cement by other means than sieves, and was gratified to find that Mr. Matthews agreed with him in believing that a practicable method of elutriation—whether by a liquid or a gas—might be worked out, and a standard process arrived at.

Meeting held Monday, May 21st, 1894.

MR. WILLIAM THORP IN THE CHAIR.

#### A JAPANESE PSEUDO-SPEISE (SHIROMÉ), AND ITS RELATION TO THE PURITY OF JAPANESE COPPER AND THE PRESENCE OF ARSENIC IN JAPANESE BRONZE.

BY W. GOWLAND, A.R.S.M., F.R.S., F.I.C., LATE OF THE IMPERIAL JAPANESE MINT.

IS the separation of silver from copper by lixiviation with lead by the Japanese process, known as the "namban fuki" method, an alloy of copper, arsenic, lead, and antimony, called "shiromé," is obtained as a by-product, in larger or smaller quantities, according to the nature of the copper treated.

As this substance is unique in composition among metallurgical products, as, besides, it forms an important

constituent of some Japanese bronzes and allied alloys and has not hitherto been analysed or described, the following notes on its occurrence, composition, and uses will, I hope, be of interest to the members of our Society.

The process, too, in which it is produced will not be of less interest, as it possesses several important features and exhibits some curious relations or affinities of the metals copper, lead, arsenic, antimony and bismuth, under conditions which are not found in ordinary metallurgical operations. Affinities which have played a prominent part in the separation of the three latter metals from Japanese copper.

I propose now, with your permission, first, to describe the process in which the shiromé is a by-product, and its effects on the copper treated by it, and subsequently to consider the by-product itself.

The process of liquating copper with lead for the separation of silver is called, as I have stated above, "namban fuki," which signifies "melting after the style of the southern barbarians," the method having been introduced into Japan by the Portuguese. Since the date of its introduction, about the latter half of the 16th century, it has been continuously employed in the treatment of all copper containing sufficient silver to pay for its extraction.

As most of the copper ores of the country are more or less argentiferous the process has been and is still in extensive use, and very large quantities of silver have been extracted by means of it. Some gold also has been obtained, but only a part of that present in the metal treated can be liquated out with lead, a considerable proportion being always retained in the copper.

This process was not, however, introduced by the Portuguese in its present form. What they introduced was the old German method, known as "Das Saigern," only the principles of which have been followed by Japanese metallurgists, who, with these principles as a basis, developed the furnace, appliances, and method of liquation which are now in use and which are essentially Japanese.

Great credit is due to these metallurgists, especially to those of the early days, for the ingenuity they have exhibited in their radical alterations of the European method, for by these alterations they have not only made the process suitable for the work of the smallest mine, but have achieved results with it on all scales far surpassing in economy and in completeness of separation of the respective metals anything that had been accomplished by it in its original form.

Thus, for example, in the Japanese process less lead is required, a lead richer in silver is obtained, less lead and silver are left in the copper, and as small a quantity as 80 lbs. of copper can be treated at a time.

Copper suitable for liquation:—

The amount of silver in the copper which is necessary to make the liquation process a profitable one varies in different parts of the country.

In Tokio, 0.077 per cent. silver (25 ozs. 3 dwts. 1 gr. per ton) is said barely to pay the costs of the operation, but in other localities, where charcoal and labour are cheaper, this proportion of silver would be remunerative.

Generally speaking, in all districts when the copper contains 0.092 per cent. silver (30 ozs. 1 dwt. 2 grs. per ton) or more it is subjected to this process.

The following analyses (No. 1) show the proportions of silver contained in typical specimens of copper from different localities.

Two specimens of copper from Korea are included in this table as they represent the most highly argentiferous crude copper I have met with in the East. The occurrence of such large percentages of silver in these specimens leads me to direct the attention of miners and metallurgists who may proceed to that country to the copper ores as sources of silver, as hitherto prospectors have confined their search for the metal to outcrops of galena and silver ores proper, and have not had the success which their energy deserved. The large amount of gold in the copper from Towata is worthy of note. About five tons of the sample containing 25.51 oz. per ton were imported into the imperial mint as crude copper, the importer being ignorant of the presence of gold in it until he received the assay report.

## No. 1.

## SILVER AND GOLD IN CRUDE COPPER.

Locality.	Silver.	Silver.	—
	Per Cent.	Oz. per Ton.	
Province of Banzo....	0.060	19.60	Gold trace; nickel, cobalt, 0.251 per cent.
" Echigo (Kosakura).	0.078	25.18	Gold trace.
" Kozuke (Ashiwa).	0.094	30.70	"
" Tosa.....	0.135	44.19	"
" Uzen.....	0.140	45.73	"
" Bichu.....	0.165	54.40	"
" Hida (Takayama).	0.185	60.43	"
Yezo.....	0.230	75.13	Gold 0.003 per cent. (0.96 oz. per ton).
Province of Bichu.....	0.252	82.32	Gold trace.
" Unknown...	0.327	102.15	"
" of Rikuchu (Kosaka).	0.650	212.33	Gold trace, lead 4.09 per cent.
" Iwami (Omori).	Trace	..	Gold 0.0165 per cent. (5.39 oz. per ton).
" Rikuchu (Towata).	"	..	Gold 0.0285 per cent. (9.31 oz. per ton).
" "	"	..	Gold 0.0436 per cent. (14.22 oz. per ton).
" "	0.011	3.60	Gold 0.0810 per cent. (26.51 oz. per ton).
Korea.....	2.330	816.07	Gold trace.
" .....	3.000	980.00	"

The operations of the Japanese liquation process are divided into two stages, each being conducted in a different furnace, viz.:—

A. Preparation of an alloy or mixture of copper and lead.

B. Separation of the lead (and with it the silver) from the mixed metals.

When, however, the crude copper already contains sufficient lead, stage A is omitted and it is submitted at once to the operations of stage B.

Several mines in the province of Hida, where the metal is obtained by smelting ores rich in galena, yield copper of this description, in which the amount of lead, as will be seen from the following analysis, occasionally reaches 39 per cent.

## No. 2.

## CRUDE COPPER CONTAINING EXCESS OF LEAD (PROVINCE OF HIDA).

	Per Cent.
Copper.....	58.87
Lead.....	39.28
Iron.....	0.08
Arsenic.....	Trace.
Antimony.....	Nil.
Sulphur.....	1.64
Silver.....	0.185
Gold.....	Trace.
	100.055

This is exceptional, and generally in most districts the addition of lead is required.

Stage A.—Preparation of the mixture of lead and copper:—

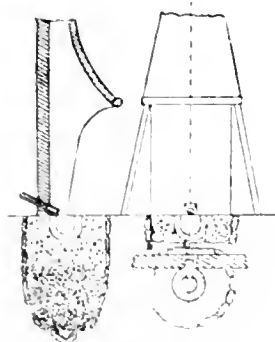
The proportion of lead to be mixed with the copper varies at different mines and smelting works, ranging from 1 part of lead to 55 parts of copper at Ani (prov. of Ugo) to 1 part of lead to 3 parts of copper at Omodani (prov. of Echizen), the general ratio being about 1.4.

Both lead rich and poor in silver are used, the former being preferred when it can be procured, as more silver can be obtained from it than the old Japanese methods of assaying indicate, and the final cupellation is hastened.

The amounts of silver usually present in these two kinds of lead are as follows:—

		No. 3.	
		SILVER IN JAPANESE LEAD.	
Poor lead...	0.003%	silver (20.56 oz. per ton);	traces of gold.
"	0.004%	" (30.71 " "	"
"	0.012%	" (101.92 " "	"
Richer lead	0.366%	" (119.56 " "	"
"	0.124%	" (138.51 " "	"
"	0.448%	" (146.35 " "	"

Fig. A.



MELTING FURNACE.

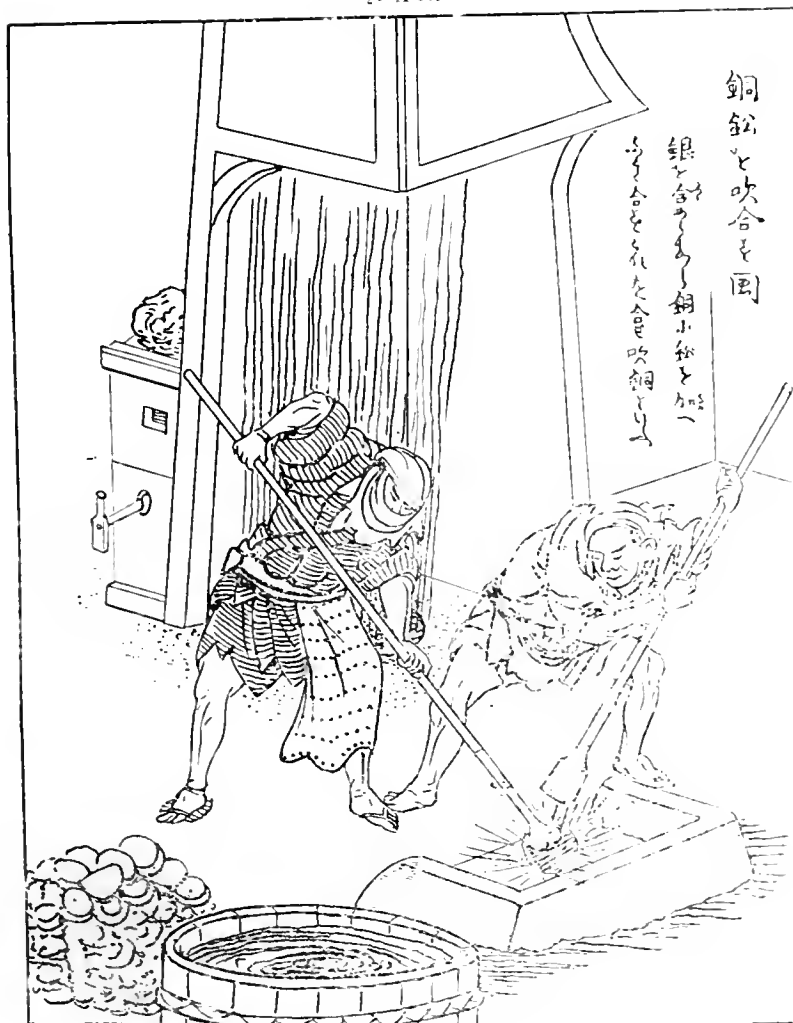
The furnace used is the typical Japanese smelting furnace shown in Fig. A. It consists simply of a rudely hemispherical hole about 16 in. diam. and 15 in. deep in a specially prepared portion of the ground of the smelting room, lined with a brusque of refractory clay and charcoal, and to which a blast is supplied through a clay twyer from the ordinary double-acting hand bellows in general use in China and Japan, which is placed behind the furnace.

Ignited charcoal is placed at the bottom of the cavity, which is then nearly filled up with fresh charcoal. Upon this the copper, which is in irregularly-shaped lumps, is placed, and more charcoal is heaped up over it.

The bellows are then started, more copper and fuel are added from time to time until the whole of the charge has been put in, and the blast is continued until all the copper has completely melted. The fire is then raked off, the metal skimmed, and the lead added and mixed thoroughly with the copper.

The alloy is then removed from the furnace by means of a curious tool consisting of a rudely spherical-shaped iron head about 4 in. or more in diam. attached to a long handle. This is dipped into the molten alloy, lifted out, and the adhering crust of metal knocked off and thrown into water. The tool is then cooled with water and another crust taken out, and this is continued until the furnace is emptied of its contents. This part of the operation is graphically shown in the Japanese drawing, Fig. A A. Six or seven charges, each of about 250—300 pounds of copper, are worked per day with a consumption of 20 per cent. of charcoal.

Fig. A A.



In a large copper refinery in Tokyo, erected after my designs, the copper is melted in charges of three or four tons, and is ladled or run out in quantities of half a ton at a time into a large casting ladle in which the lead is mixed with it. The alloy is then cast in iron moulds into flat plates with intersecting furrows, so that it can be easily broken up when cold.

The former method is, however, that in general use.

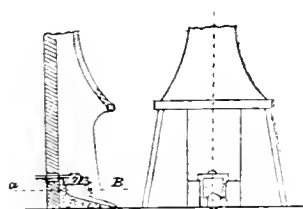
The copper-lead alloy is now taken to the liquation department, where the second part of the process, stage B, is conducted.

Stage B.—Separation of the lead from the copper-lead alloy.

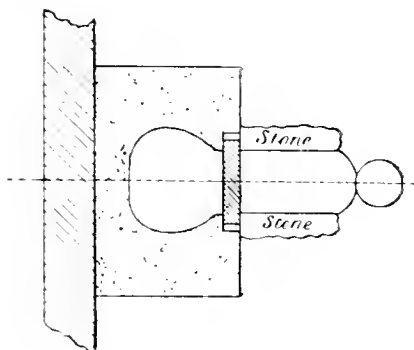
The operations of this stage are carried on in a furnace of simple but ingenious construction.

A vertical section showing its general form, the arrangement of the twyer, and the curious fore-hearth is given in Fig. B.

Fig. B.



LIQUATION FURNACE.



SECTION ON A B (ENLARGED).

Its exterior, as well as that of the fore-hearth, usually consists of flat stones set on edge, but in the mint and some modern refineries these parts are built of brickwork.

(In Dr. Percy's Metallurgy—Silver and Gold—Part I., p. 341, a drawing is given by Mr. Tookey of the brick furnace used in the mint in 1871.)

The interior is a circular cavity about 18 inches in diameter and 13 inches deep, with clay sides slightly converging towards the bottom, which slopes downwards towards the front side.

From the front side the fore-hearth projects in the form of a shallow trough in a line with the bottom and inclined at about the same angle. Below the end of the trough there is a shallow circular hole in the ground for receiving the liquated lead.

The interior of the furnace and of the fore-hearth is lined with the usual brasque of clay and charcoal.

The front side is open, but is partially closed during working with a fire-clay tile.

The top is covered with a clay slab excepting a small opening which is used for the addition of fuel, and is also covered during working with a movable tile.

The blast pipe is usually of clay, sometimes of iron, and terminates in a clay nozzle or twyer which passes through the cover of the furnace. The twyer is bent sharply at an angle, the correct inclination of which is considered to be of great importance, its object being to direct the blast downwards and towards the middle of the back wall.

The furnace is placed under a hood for carrying off the fumes.

The charge of alloy weighing from 100—160 lbs. is placed on the bottom of the furnace, the larger pieces below, then a little charcoal, and then the smaller pieces. The front tile is now fixed in position, and the furnace almost filled with charcoal, the covering slab luted on, and the twyer adjusted.

Glowing embers are now put in, the charging opening is closed, and the bellows started.

The furnaceman then takes up his position, squatting on the ground in front of the fore-hearth.

Great skill is required in regulating the force of the blast, so that too high a temperature may not be produced, as the alloy must not be melted but merely brought to a pasty condition.

When it has reached this state it partly protrudes through the opening in the front on to the upper part of the trough forming the fore-hearth, and is there kept hot by the flame—which issues continually from this opening—passing over it.

The mass—a copper sponge saturated with lead—is now patted and squeezed by the workman with a tool consisting of a small block of wood about 5 or 6 ins. long and 3 ins. in diameter fixed on a hooked iron rod attached to a wooden handle, the lead trickling from it being collected in the hole in the floor (Fig. C.). As it becomes cool and lead ceases to exude, it is pushed back towards the hotter part of the furnace with a small iron rabble, and when sufficiently pasty it is again patted as before.

When the lead ceases altogether to flow from it the temperature is raised and the pasty mass is raked down the spout with further patting and allowed to solidify there.

The average duration of the operation is about 2½ hours, and usually three to four charges are worked per day, with a consumption of from 40 to 50 per cent. of charcoal.

The products of this liquation process are: copper containing little silver, argentiferous lead, and shiromé.

The specimen on the table is a sample of the copper in the form in which it is taken from the fore-hearth. Its composition is represented by the following analysis:—

No. 4.

COPPER AFTER LIQUATION.

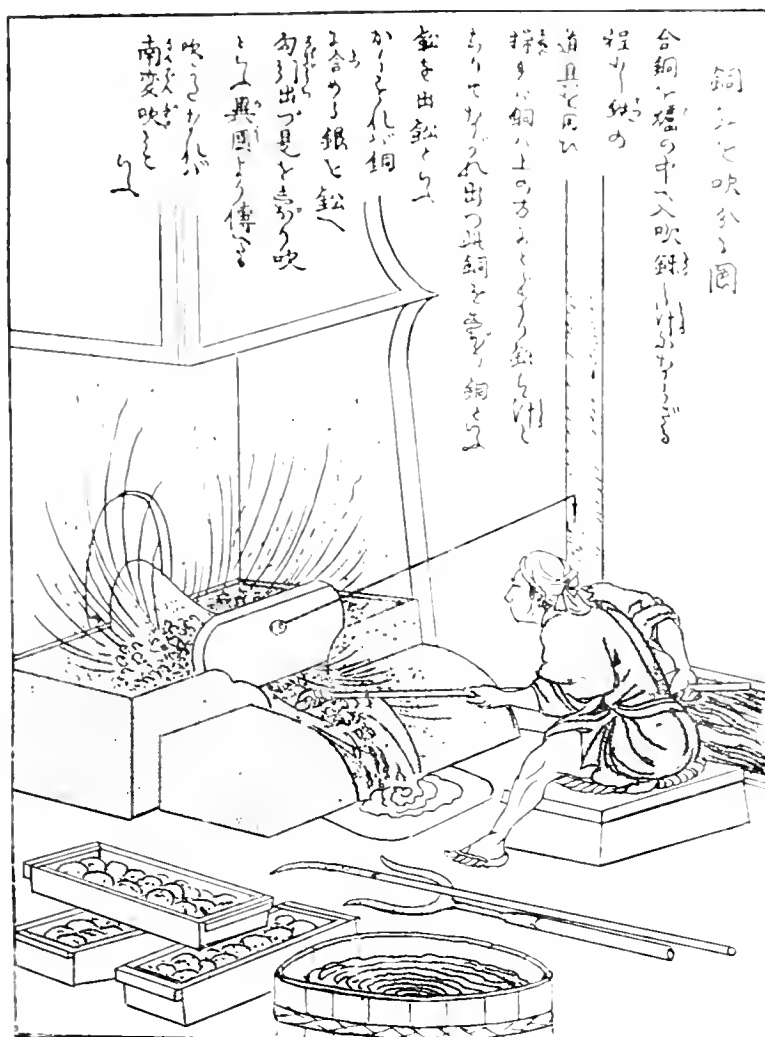
	Per Cent.
Copper.....	99.12
Lead.....	0.52
Arsenic.....	0.04
Antimony.....	Trace.
Iron.....	0.04
Silver.....	0.031
Sulphur.....	0.02
Nickel.....	Trace.
Insoluble sandy residue.....	0.06
	99.831

Sometimes when the process has been hastily or unskillfully conducted the amount of lead retained by the copper exceeds that in this specimen, but the greatest amount I have found has been 0.97 per cent.

The lead which has collected in the cavity in the floor contains varying quantities of silver according to the amount present in the copper and in the original lead used for the liquation.

Rarely less than 0.25 per cent. (81.66 oz. per ton), frequently 1.3 per cent. (424.66 oz. per ton), and occasionally as much as 2.2 per cent. (718.66 oz. per ton), is present

Fig. 1.



It also contains the whole of the bismuth, a little of the arsenic and antimony which may have been present in the copper, and small quantities of copper.

Usually only about 80—90 per cent. of the weight of the lead added to the copper is obtained, the remainder being lost, part being retained in the copper, shiromé, and slag, and part being volatilised.

The lead is sent to the cupellation department, where it is first submitted to an oxidising-melting process on a hearth of wood ashes and clay, and then is cupelled on a similar hearth of smaller dimensions, a cake of silver containing a little gold being obtained (see specimen).

The shiromé accumulates on the outside of the spongy mass of copper in the fore-hearth in irregularly shaped semi-fused lumps, and is removed by the furnaceman from time to time during the liquation.

The amount yielded by any operation varies generally from 1—2 per cent. of the weight of the copper treated, but is entirely dependent on the purity of the copper; if this contains only traces of arsenic and antimony no shiromé is formed; if, on the other hand, much of these are present the proportion may greatly exceed 2 per cent.

When sufficient shiromé has accumulated in the liquation department it is twice treated by liquation with lead for the separation of as much as possible of the silver which it contains, but in spite of this treatment a considerable

amount of silver is always retained in it owing to the arsenic, which is one of its essential constituents.

*Note.*—The word shiromé (meaning "white solder"), the name of this substance, is unfortunately also used to designate antimony, the Japanese not having had any specially distinctive name for this metal excepting Iyo-shiromé or shiromé from Iyo (Iyo being the province in which the chief antimony mines were situated) until recently, when "anchimoni" was adopted from the English word. Frequently "Iyo" was omitted and only shiromé written.

The term is also applied to ordinary tin and lead solder. I think it necessary to mention these three uses of the name, as they have led to much confusion in Japanese metallurgical books, and especially in translations from them. Thus a careful and learned writer has conveyed a completely wrong impression of the composition of some Japanese alloys by translating shiromé as antimony when this copper-arsenic-lead-antimony alloy is meant.

Shiromé as it is obtained after liquation with lead is in rough irregular shaped, somewhat vesicular lumps. When melted and cast into ingots it is of a very dark bluish-grey colour. It is harder than lead, rather brittle, and can be reduced to a coarse powder by careful hammering in a steel mortar. Its fracture is dull, coarse grained, and free from crystalline structure.



The following complete analysis No. I. represents a sample taken directly from a liquation furnace at the smelting works of the Onodani mine (prov. of Echizen), which, as we shall see later, are noted for the purity of the copper they produce.

Nos. II and III are partial analysis of specimens from other districts, that from Iyo, No. II., having been liquated with lead for the separation of as much silver as possible.

No. 5.  
SHIROMÉ.

	From Onodani (Province of Echizen).	From the Province of Iyo.	From the Province of Kaga.
Copper.....	72.76	..	..
Lead.....	8.53 } 8.75 } 8.32	..	..
Arsenic.....	11.37 } 11.50 } 11.25	7.64	9.18
Antimony.....	4.27	0.39	5.75
Tin.....	0.93	..	..
Iron.....	0.13	..	..
Silver.....	1.33	0.426	0.312
Sulphur.....	0.33	..	..
Zinc.....	Nil.	..	..
Gold.....	Trace.	..	..
	39.50		

The two determinations of lead and arsenic respectively were made in different portions of the specimen I., and they show that contrary to what might be expected it is tolerably homogeneous in composition. Its characteristic features, as will be seen from these analyses, are the presence of arsenic in considerable proportions, of antimony, of lead, and a large percentage of copper. In the specimen No. I. the occurrence of the two former metals in such large amounts is specially noteworthy, as it demonstrates that without treatment by the liquation process, by which this shiromé was obtained, the Onodani copper would be seriously contaminated with both, and would be noted rather for its impurities than for its purity.

I desire here to record the deep obligations I am under to my friend Prof. Roberts-Austen for having kindly placed the resources of the Metallurgical Research Laboratory of the Royal School of Mines at my disposal, so that I was able to make there not only the analyses of shiromé, but also to complete others which I had left unfinished in Japan.

After a very careful search I have failed to find any record of a metallurgical product approximating to shiromé in composition. A substance somewhat analogous to it in its mode of production is the Saigerdorner of the old German liquation process, but this, as will be seen from the accompanying analysis, differs entirely from it in composition.

\*Saigerdorner (E. von Szamit):—

Copper.....	11.36
Lead.....	79.68
Antimony.....	7.30
Silver.....	213.045
Gold.....	5000.065
	99.123510

It is impossible to assign any definite chemical formula to this complex substance shiromé, owing to its non-occurrence in a crystalline form, to the total absence of any crystals disseminated in its mass, and to its variability

in composition at different works. It may, however, be regarded as an arseno-antimonide of copper and lead, in which an excess of copper and lead is held in solution, *lev.* as a pseudo-speise. The various copper-lead so called speises, when they are the products of metallurgical operations, although differing from it in the relative proportions of their constituents, appear always to have this constitution, the amounts of arsenic and antimony present being less than sufficient for combination with the copper and lead. In this respect they are distinct from the true arsenical speises containing nickel and cobalt, in which the arsenic is frequently combined with these metals in definite atomic proportions.

Having now described the method of liquating copper with lead, and the products which result from the operation, I will endeavour to point out as briefly as possible the influence which this process has had on the purity of Japanese copper.

The aim of the process is the separation of silver from copper, and to the success with which this is accomplished by it, it chiefly owes its importance and value in Japanese metallurgy. But another and almost equally important result accompanies its use, *viz.*, that by it copper containing arsenic, antimony, and bismuth is either entirely freed from these metals or has the proportions in which they are present so reduced that they no longer exercise any injurious effects on its physical properties or unfit it for use as a constituent of brass or other alloys. The action of the process in removing these metals may be explained as follows:—

Under the conditions which prevail lead does not form a definite alloy with copper. When the two metals are melted together in the first stage A, only a mixture, and not an alloy, results.

During this melting, however, there would seem to be a transference of the arsenic, antimony, silver, and bismuth from the copper to the lead, so that the copper-lead mixture, after being rapidly cooled, may be regarded as a solidified lead solution of the silver and bismuth as metals, of the arsenic and antimony as an arseno-antimonide of copper and lead, and through which comparatively pure copper is disseminated in granular or crystalline particles.

When this mixture is heated in the second stage B, and it is important to remember that the temperature never reaches the fusing point of copper, the lead solution of the silver and bismuth trickles away from the spongy agglomeration of the particles of copper into the receiver prepared for it, but as its temperature is lowered as it leaves the copper at the upper end of the hearth below that required to retain the shiromé in solution, this substance is gradually deposited there.

The greater part of the arsenic and antimony separates out in the form of this pseudo speise. The whole of the bismuth, excepting mere traces, and nearly all the silver, passes into the liquated lead.

I am not acquainted with any other furnace process by which bismuth can be removed from the metal copper. The percentage when much is present may indeed be reduced by prolonging the ordinary process of refining, but more than traces are always retained by the copper.

It is important to note that when the lead is subjected to the subsequent cupellation process much of the bismuth passes into the silver, communicating to it the greatest brittleness. As, however, a simple furnace operation suffices for its removal it is no longer a source of trouble to the metallurgist.

Several kinds of Japanese copper which have, and deservedly so, a high reputation for purity, would, but for this process, be unfit for most industrial purposes, and in demonstration of this I will ask for your attention to the consideration of the following examples. The first, which is specially noteworthy, is the copper from the mine of Onodani (prov. Echizen), previously alluded to, which is obtained by smelting ores containing falherz and native bismuth. The falherz occurs in such quantities that the roasting heaps at the mine are incrustated with arsenious anhydride and realgar. Yet the Onodani copper is free from more than mere traces of arsenic and bismuth and entirely so from antimony, and has been long regarded as the best copper in

Japan for its malleability and ductility. In a recent report of the Director of Mines it is stated that it is the only copper now used in the Imperial arsenal for special copper work.

I have, unfortunately, no analyses of the metal before treatment by the liquation process, but from the composition of the *shiromé* obtained, given above, it must have contained about 0.20 per cent. arsenic and 0.08 per cent. antimony. The proportion of bismuth present would be variable, as the native bismuth is irregularly disseminated through the ore.

Its composition, after liquation and subsequent refining, is as follows:—

No. 6.  
OMODANI COPPER.

	Wire Bar.	Slab for Hammered Work.
Copper.....	99.81	99.67
Arsenic.....	Faint trace	Faint trace
Antimony.....	Nil	Nil
Bismuth.....	Nil	Nil
Lead.....	0.119	0.252
	99.919	99.922

(In these analyses a ft. tr. of arsenic means that when 13 grms. of the copper were taken, no weighable precipitate of ammonium magnesium arsenate was obtained in the final solution (measuring 60 to 80 cc.) but merely streaks on the sides of the beaker indicating its presence).

Both analyses show a metal of great purity for copper which has not been electrolytically deposited.

The higher percentage of lead in the slab is intentional, as it is believed by the Japanese that the presence of lead in copper increases its malleability, and when it is not supposed to be present it is always added. The addition of lead to some kinds of copper have undoubtedly this effect, although the amounts sometimes added by the Japanese with excellent results so far as mere hand hammering is concerned, would be altogether detrimental to copper which had to be subjected to heavy forging.

The second example is a sample of copper which was sent to the Imperial Mint from one of the chief mines. I may say that before accepting any copper, or indeed, anything which could be chemically examined, I made an inflexible rule that it should be first analysed; a rule, the observance of which contributed largely to the success of many of the operations in the Mint.

This copper was partially analysed, and found to contain the following proportions of arsenic, antimony and bismuth:—

No. 7.

ASHIHO COPPER BEFORE LIQUATION.

	Per Cent.
Arsenic.....	0.26
Antimony.....	0.16
Bismuth.....	0.10

The injurious effects of bismuth, the *bite noir* of the copper refiner, on the ductibility and malleability of copper are well known. As small a quantity as 0.06 per cent. sometimes will cause commercial copper to be so red-short as to be quite unfit for any purpose where forging is necessary. The effects of arsenic and antimony on the physical properties of brass and some other alloys are not less familiar to metallurgists.

The above copper was hence promptly rejected as worthless for the manufacture of hammered or rolled articles on account of the bismuth present, and for the manufacture of brass on account of the arsenic and antimony. It was

then subjected to the liquation process at the works of the importer, after which it was again brought to the Mint, when on analysis it was found that these metals had been almost completely removed, the amounts present being as follows.

No. 8.

ASHIHO COPPER AFTER LIQUATION.

	Per Cent.
Arsenic.....	0.04
Antimony.....	Trace.
Bismuth.....	Trace.

These two examples, I think, sufficiently demonstrate the important influence which the liquation process has on the purity of copper.

Unfortunately it is not applicable in England owing to the high price of charcoal and labour, although several brands of copper in the London market which are contaminated with bismuth contain sufficient silver for their economic treatment in Japan.

The application of the by-product *shiromé* to practical uses exhibits well the ingenuity and skill of the Japanese as workers in metal. Alone it is worthless, but as early as the beginning of the last century they discovered that not only could it be used as a constituent of some alloys, but that by its addition certain valuable properties were conferred on them.

The first official record we have of its use is contained in an edict of the Japanese Government in 1761, prescribing its addition to the copper-lead bronze to be used in the Mint for casting "Do-sen," a small coin commonly known to Europeans as "copper cash."

In 1768 we have another edict directing the minting for the first time of brass coins called "shimon-sen" and the addition of *shiromé* to the alloy to be used. These two kinds of coins, as shown by the following analyses, hence contain considerable quantities of arsenic and antimony.

The analyses of another coin, "Bunkio-sen," which was chiefly a re-coinage of "Do-sen" in 1863, is also given.

No. 9.

JAPANESE "COPPER CASH" CONTAINING SHIROMÉ.

	"Do-sen."	"Shimon-sen."	"Bunkio-sen."
Copper.....	77.39	75.62	83.19
Tin.....	4.92	0.73	3.21
Lead.....	15.33	2.85	14.22
Arsenic.....	1.14	1.99	1.59
Antimony.....	0.31	0.14	0.19
Zinc.....	Nil.	16.54	Nil.
Iron.....	1.01	1.76	0.27
Silver.....	0.04	0.016	0.04
Gold.....	Trace.	Trace.	Trace.
Sulphur.....	0.52	0.09	0.58
	99.99	99.734	100.23

The objects of its use in these cases were: to give additional hardness to the alloy without impairing its fusibility at moderate temperatures, and to obtain, when cast, a sharper impression of the mould than was possible with the copper-lead alloy alone. Additional hardness could also have been given by increasing the proportion of tin, but this would have had the disadvantageous effect of raising its melting point and of diminishing its fluidity when melted, both of which the Japanese desired to avoid, for two reasons—the first being that their crucibles, owing to the absence of a good refractory clay, were incapable of

sustaining long exposure to a high temperature, so that with metal of a high melting point they were soon worn out; and the second, that the coins were cast in very delicate moulds, made of a sand always more or less fusible, in which if metal with a high melting point was cast, the castings would be generally coated with a crust of semi-fused material not easily removable.

The specimens on the table demonstrate clearly the success with which the Japanese foundries contended with the difficulties inseparable from the want of a refractory clay and non-fusible sand by thus modifying the composition of their alloys to suit these unfavourable conditions. I do not think any other alloy would give equally good results so far as sharpness of impression, clean surface, and hardness are concerned.

During the latter half of the last century—probably from an earlier date—and up to the present time, shiromé has been largely used by the bronze founders of Osaka and Kyoto, the chief centres of the metal industries as a frequent addition to “Karakane,” a copper-lead bronze which is the principal alloy used in the manufacture of the numerous metal articles for ornamental and useful purposes in the production of which the Japanese are unrivalled. It is added to this bronze for the reasons given above for its addition to the old coinage alloys, but there is also another reason for its use, *i.e.*, it facilitates the production of the deep grey patina which is preferred for those vases or other objects which have to be decorated with inlaid line designs in silver (*zogan* work).

On the other hand, when a specially deep black patina was required in high-class bronzes, it was the practice of some of the best art founders to omit it from their mixtures for “Karakane.”

These last remarks apply solely to the older bronzes and not to the modern so-called “copies” of them.

In the old bronzes the colour and character of the patina is largely dependent on their composition.

On the other hand, in recent imitations—which however are not comparable with them in any way, excepting that they bear a slight but imperfect resemblance to them in superficial colouring—a false patina of almost any colour may be given to any copper alloy, irrespective of its composition, and even to unalloyed copper.

The following analysis of an old bronze (Karakane) incense burner (date 18th century) affords an example of the use of shiromé.

No. 10.

INCENSE BURNER OF BRONZE CONTAINING SHIROMÉ.

	Per Cent.
Copper.....	87.5
Tin.....	1.75
Lead.....	9.13
Arsenic.....	1.15
Antimony.....	0.40
Zinc.....	Nil.
Iron.....	0.33
Silver.....	0.675
Gold.....	Trace.
	<hr/> 99.66

From the proportion of arsenic present I should think that about 10 per cent. of shiromé had been used in making this alloy.

And here I wish to correct an error into which several writers have fallen who have accounted for the presence of arsenic and antimony in Japanese bronzes by assuming that it is due to the use of impure copper. Now no Japanese copper—and I have analysed some hundreds of specimens—ever contained, even when imperfectly refined, sufficient quantities of these metals to account for the percentages found in many bronzes.

The highest percentages I have ever found until the recent introduction of high blast-furnaces and modifications of European methods of smelting have been 0.085 per cent. arsenic and 0.090 per cent. antimony, and these amounts were quite exceptional. In fact, of all the samples received

at the Imperial Mint during 16 years 96 per cent. contained less than 0.015 per cent. arsenic, generally only a trace being present, and 97 per cent. had less than 0.02 per cent. antimony, this metal being usually absent altogether.

So that when arsenic and antimony are found in large proportions in the older bronzes their presence is due to their addition to the alloy in the form of shiromé, and not to the use of impure copper.

In some cases the arsenic has been added in the form of “Hakudo,” a Chinese alloy of copper and arsenic, but these are rare. An interesting example of this is afforded by a sword guard, the composition of which is taken from a valuable series of analyses\* of modern Japanese metal-work by my distinguished friend Prof. Roberts-Austen:—

SWORD-GUARD. COPPER ARSENIC ALLOY. (ROBERTS-AUSTEN AND WINGHAM).

Copper.....	98.060
Arsenic.....	1.066
Antimony.....	0.910
Lead.....	Nil
Bismuth.....	Nil
Iron.....	0.018
Nickel and cobalt.....	0.023
Zinc.....	Nil
Tin.....	Nil
	<hr/> 99.182

Another use for shiromé is as a cheap substitute for tin in the manufacture of metal mirrors, especially of the commoner kinds. This would seem to be a modern innovation, as in the older kinds I have not found arsenic and antimony in those proportions in which they would have been present had it been so used.

The following mixtures used in Tokyo are given in Ayrton and Perry's paper on “Japanese Magic Mirrors,” read before the Royal Society 2nd October 1878:—

Copper.....	81.3	71.5
Iyo shiromé (impure antimony) ..	2.4	..
Shiromé.....	16.3	28.5
	<hr/> 100.0	<hr/> 100.0

I have not, however, found such mixtures in use in the chief foundries of Osaka, tin always forming a constituent of their alloys, for the following reason: that when shiromé is used without any tin the mercury amalgam employed in silvering the face of such mirrors produces a surface which is deficient in brilliancy, is easily tarnished, and has but little endurance.

I have endeavoured this evening to describe a process developed by the old metallurgists of Japan, an interesting by-product obtained in it, and the uses to which this by-product has been put; and I am sure you will all agree with me that the skill and ingenuity the smelters and founders of Japan have shown in accomplishing the results I have somewhat imperfectly placed before you are worthy of our warmest admiration.

DISCUSSION.

Professor ROBERTS-AUSTEN said he had listened with the greatest possible interest to this account of the working of those consummate metallurgists, the Japanese. No one could possibly deal better with the subject than Mr. Gowland, who, he was sure, could give more than one paper on this subject if he would. No one knew better than metallurgists who had examined Japanese alloys how marvellous was the skill with which they added a minute quantity of what we should call an impurity for the sake of producing a definite result in the way of patina. The very smallest

\* Report on the analyses of various samples of Oriental metal work. Made under the direction of Prof. Roberts-Austen by Arthur Wingham, 1892.

quantity of gold would produce the most beautiful purple patina. He feared that in writing on this subject he might have said that the antimony and arsenic in Japanese alloys was not deliberately introduced, as they now knew to be the case, but found their way in from impurities in the copper. With regard to the process itself, Mr. Gowland said it was introduced into Japan in the middle of the 16th century, and the best account was that of George Agricola, who wrote in the middle of the 16th century, but that was not by any means the earliest period at which the process was known. There were earlier accounts of it, and quite recently, in some excavations made at Silchester, specimens of lead used by the Romans had been discovered, some of which were submitted to him and analysed by his assistant, and he thought there could be no doubt from their examination that in late Roman times this process of liquation was employed, and probably with success. There was one curious point about the bismuth, a very small quantity of which exercised a very deleterious effect on copper; some curious work which he did recently for the mechanical engineers showed that however small the proportion of bismuth in copper might be, it always remained free, it did not unite with the copper. It was possible by studying the cooling curves by means of an ordinary thermal couple to find veins of free bismuth, even although its amount did not exceed 0.1 per cent. or less. Mr. Gowland had thanked him for placing the laboratory of the School of Mines at his disposal, but seeing that Mr. Gowland had placed all the specimens there, the least one could do was to beg him to examine them *in situ*. He confessed he should have thought the introduction of the shiromé would have had a tendency to make the copper cash brittle, but that did not seem to be the case, and there was no doubt an extreme sharpness given by its addition. He should like to ask if the Chinese used similar alloys, and whether such Indian tribes as used coins of a similar appearance used the same kind of alloys.

Mr. CLAUDE VAUTIN said this very interesting paper had cleared away what was always a mystery to him, viz., the removal of the bismuth from copper, which, as was well known to everybody connected with copper, was a very serious impurity. If he understood the process aright, the explanation was something of this kind: in the melting furnace the copper and lead were both brought to a state of fusion, the mixture was then removed by plunging in a cold piece of iron on which a thin layer of the metal was, as it were, frozen; that was knocked off, the iron cooled, and again dipped into the mixture. In the melting furnace, while the two metals were in a fluid condition, no doubt there was a transference of the arsenic, bismuth, silver, and gold from the copper to the lead. The sudden cooling on the surface of the iron prevented the copper re-uniting with the bismuth and arsenic. Then in the liquation furnace the copper was never allowed to become in a fluid condition, and the operation of the workmen in front of the hearth consisted in pushing back, working up, or puddling the material. In so doing they gave the lead an opportunity of running away in exactly the same condition as it was in the melting furnace without the copper ever having been in a sufficiently fluid condition to re-absorb the bismuth, antimony, and silver. He should like to know if this was the correct rationale of the process; if so, it might have very important consequences. Personally, he was engaged a few years ago with a copper company in trying to remove bismuth from the copper, the presence of which reduced the value of that metal in the London market by 8*l.* a ton. On one occasion a remarkable result was obtained which unfortunately was never repeated, but of which he thought they had an explanation. They granulated some copper containing the bismuth and melted it with lead; he did not think it was quite fused, but the crucible was put on a slab and suddenly cooled, so that the copper set more rapidly than usual, and the lead when examined was found to contain most of the bismuth. They never got the same result when the copper was brought to a state of fusion. If this were the explanation it would be possible to introduce the operation at certain copper mines where there was difficulty in removing the bismuth, and in

this way produce good copper and make the mines pay. If this proved to be the case they would be very deeply indebted to Mr. Gowland for having placed these important facts before the Society. On looking over the analyses, he noticed that traces of gold were remarked in all the by-products, and in the first analysis one went as high as 26 oz. per ton. Would the gold follow the silver right through the whole operation?

The CHAIRMAN said that people in the West were rather apt to think that Easterns did not understand their own business, but this paper showed the importance of an observant eye, and proved that they did understand their business perhaps better than we did.

Mr. GOWLAND, in reply, said that in the sixth and seventh centuries the Chinese used a similar copper-lead alloy, and the first Chinese coins were made in imitation of that. The latter Chinese coins were nearly all of brass. He did not know that Professor Roberts Austen had stated that the arsenic in bronzes was due to impurities in the copper, and did not think he had—he was referring to some other writers. With regard to the reasons for the removal of the arsenic, bismuth, &c., he thought it was entirely due, as Professor Austen had pointed out, to the bismuth not alloying with the copper. It remained fused throughout the mass of copper, and then in this process of liquation the copper was never melted. It was just like a sponge, and the bismuth and arsenic were squeezed out along with the lead. With regard to the gold, he always put down gold as present in traces when on taking 20 grms. there was no weighable quantity although its presence was indicated. With regard to copper containing 40 oz. to the ton, that was peculiar to one of the mines in Japan. They produced it in large quantities from 10 to 20 tons or more per annum. This copper containing 40 oz. per ton was brought to the mint as common copper, they being quite unaware that it contained gold until he sent the assay report; in fact, however, the gold was much more valuable than the copper. In treating copper containing gold by the liquation process, only a part of the gold was removed; it did not all go with the silver; a portion went, but a variable quantity remained behind in the copper.

## Liverpool Section.

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Hon. Local Secretary:

Dr. Chas. A. Kohn, University College, Liverpool.

The names in italics are those of Members of Committee who retire at the end of the current session. The following have been elected to fill the vacancies, and will take office in July next:—Committee: C. L. Higgins, C. A. Kohn, and E. K. Muspratt. Hon. Secretary: T. Lewis Bailey.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

Meeting held Wednesday, April 4th, 1894.

MR. LUSACE CAREY IN THE CHAIR.

# TREATMENT OF CUPREOUS IRON PYRITES AS CARRIED ON AT THE PORTUGUESE MINES.

BY J. HENRY BROWN.

THE most important of the Portuguese mines is that of San Domingo, worked by Messrs. Mason and Barry. The next in point of importance are, in the south, the Aljustrel, Grandola, and Brancannes mines, and in the north those of Palla and Telhada. More recently the mines actually engaged in work are the San Domingos, Brancannes, Palla, and Tinoca mines.

The first named is the most important mine, and has for a number of years supplied by far the greatest amount of sulphur ore put into the market.

Since the year 1884 the export of crude ore has ceased, the whole production being treated locally.

There is and always has been a notable difference in the preliminary processes carried on at the Spanish and Portuguese mines. This of itself is a very noteworthy feature.

In Portugal, unlike the neighbouring country, there are certain restrictions in the way of dealing with the mineral, and stringent regulations relating to the disposal of the waste product in the way of liquors.

Owing to the importance of the sea and river fisheries, it is not allowable to send waste iron (from copper precipitation) liquors into the streams at will. Wherever the configuration of the country allows, liquors have to be held back in enormous reservoirs until such time as the river shall be in heavy flood, which is generally in winter or early spring.

The burning of the mineral has been out of the question owing to local opposition and the impossibility of getting over the compensation question. Even where such difficulties might have been overcome, the question of disposal of waste liquors has been a difficulty. In some places the natural surface of the country does not allow of the formation of "holding-back" reservoirs, and for this reason the treatment of cupreous iron pyrites by the humid process has not been possible.

There are two distinct methods of treatment in use at the various mines:—

(1.) "*Burning the Mineral in the open.*"—This method has been practised to some extent at the mines of Aljustrel, but has only been tried on an experimental scale at those of San Domingo.

The process is rapid and has the advantage of yielding liquors on lixiviation containing comparatively a small amount of arsenic. The disadvantages of the process are that it is wasteful and involves a considerable loss of sulphur; it also leaves on lixiviation a residue of no commercial value, thus causing the accumulation of vast deposits of spent material. The spread of this process has been prevented in many instances by the opposition of the agricultural interest in the surrounding country.

(2.) "*The Weathering Process.*"—This process was first initiated at the San Domingo works of Messrs. Mason and Barry, and in spite of the fact that the process is slow and the liquors on lixiviation are rich in arsenic, which is thrown down with copper on treatment with metallic iron, it is still largely and successfully employed.

It is carried out somewhat as follows:—The ore from the mine undergoes a careful scrutiny for the purpose of selecting the most suitable for "weathering." That of a hard nature and a close texture is rejected; it generally has a low assay, and is further unsuitable for process because of not being able to absorb water.

Again, mineral containing copper pyrites is likewise rejected, although the assay is probably very high. The reason for this is that this class of mineral is also very hard and impenetrable to the action of water, and would yield no result at process, *i.e.*, not give up its copper.

Having selected the best ore for treatment, it is loaded into waggons and trammed away to the sites where the heaps of ore may undergo the "weathering" process.

The heaps are formed of from 100 to 250,000 tons of mineral, varying in assay from  $1\frac{1}{2}$  to 2 per cent. copper. These figures on the larger quantity named would give 3,750 tons, and 5,000 tons total fine copper. As a matter of fact about 88 per cent. of the total copper present is exhausted in the course of six years, therefore 3,300 and 4,400 tons is the yield; the balance is not recoverable except by long and unprofitable continuance of the treatment.

This seems a long time to bring about this result, but it must be remembered that enormous quantities of ore are at some places under treatment. At San Domingos this amounts to 3,000,000 tons, and if only  $\frac{1}{100}$  of a per cent. be extracted per annum, there would result an annual production of 3,000 tons fine copper; a more likely figure would be  $\frac{1}{2}$  per cent., corresponding to a produce of 6,000 tons per annum.

Plenty of stone flues are distributed on the surface of the natural ground, and the mineral is tipped on the top. These flues are connected one with another longitudinally and transversely, and at certain intervals with the outer air by vertical pipes or stone shafts. The object of all this is to provide a plentiful supply of air for the promotion of oxidation. This action goes on with wonderful rapidity and at times with such violence that water must be applied to prevent the heaps taking fire.

Such a state of things is very detrimental, as the ultimate denudation of the copper contents is prevented by reason of the formation of masses of agglomerated mineral.

For lixiviation of smalls it is best to use water by a sort of irrigation scheme, *viz.*, condensing it about in channels into small receptacles or shallow tanks, from which the water gradually drains away through the mineral.

Under these circumstances it is clear that great altitude of the heaps is desirable; the same volume of water serves for washing a 10-metre-high heap as a four-metre one. The use of water only becomes very much increased when the area covered is very great in proportion to the altitude attained. Thus it is economical to have deep heaps, for there is less water used, better liquors produced, and less space to be sought for the construction of these heaps.

By judicious treatment the already-mentioned untoward symptoms may be avoided, and by a careful alternation of application of water and competent rest for the re-establishment of the lost heat of the heap, 88 per cent. of the total copper may be extracted in the reasonable time of six years.

There is a further source of copper manufacture, that of pumping the natural drainage, rich in soluble copper, from the mine. These liquors being very free from ferric salt make remarkably good precipitate. At the mines of Messrs. Mason and Barry this feature has been very much developed by systematic flooding of the opencast as the workings have progressed. This has resulted in an enormous yield of cheaply-made copper.

For the treatment of a few million tons of process ore in a large metallurgical establishment, there must of a necessity be large supplies of fresh water stored in dams. These are filled by draining the adjoining country. By preference it is better to have the heaps of mineral under treatment at lower levels than the water supply, which can then be run to them by gravitation.

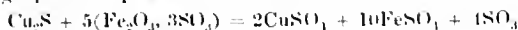
After undergoing the above-described process for a space of time, the copper contents are reduced to so low a figure that any further continuation of the treatment would be unproductive. The ore by this time has become very much disintegrated, and at such mines as are favourably situated for economical carriage and facilities for shipment, the residual small ore is exported in large quantities, being used extensively in the production of vitriol. The percentage of sulphur in the remaining smalls is but little inferior to that in the crude or untreated ore.

Previous to precipitation, or cementation, as it is called, of the copper on to pig iron, it is necessary that the liquors should be purified. There are three constituents which consume iron, *viz.*, the copper, free acid, and ferric salts; the two latter, especially the last, if present in excess, lead to

an immense consumption of iron, because of its subsequent reduction to ferrous salt by the agency of free acid and iron.

It is requisite for economical work that before precipitating the copper, the ferric salt should be as completely reduced as possible. This has in time past given much trouble.

It is now very effectually removed by filtering liquors through rich assaying cupreous sulphur smalls; the ferric salt is thereby completely reduced to the ferrous state. The operation is carried on in large dams, where the liquor may be kept some time in contact with the smalls. The following equation represents the reduction effected:—



The liquors to be treated are run through a series of settlers for the purpose of retaining any fine suspended matter, and are then brought into the precipitating plant.

This, in modern establishments, consists of a series of wide but shallow canals, constructed of either timber or brickwork, and of great length, being arranged that the liquors run through at great speed.

A fall of 1/100 amply suffices for this, but in some places they give the liquors when well stripped of copper, a fall of 1/50.

The canals may conveniently be 4 ft. wide and 8 to 10 ins. deep; pig iron is then placed in them in a methodically stacked manner, leaving plenty of interstices for the passage of liquor.

Probably most of the copper precipitate produced in Portugal is after proper preparation exported to England. There are in the former country no extensive smelting works for the treatment of precipitates or ores of copper; the high price of fuel evidently precludes any operations of this kind.

At the present time, unremunerative prices of copper have brought about a stagnation in the production of cupreous iron pyrites in Portugal.

## METHODS FOR THE RECTIFICATION OF VITRIOL. A NOTE.

BY FRANK TATE.

SINCE the publication of my paper on "Methods for the Rectification of Vitriol" (this Journal, 1894, pages 206—211), Mr. William Garroway, of Glasgow, has called my attention to a patent taken out by him for "Apparatus for the Manufacture and Concentration of Sulphuric Acid," Specification No. 1673, 1883. The specification states that the improved apparatus comprises a series of open beakers, basins, or tubes of hard Bohemian glass or glazed ware of cylindrical form, which are fitted into a flue or flues and so connected together that acid or liquor may be caused to flow continuously through the series. The beakers may be placed in an inclined flue or in a long compartment, and may be connected together by glass siphon tubes; or, in a modified arrangement, the beakers may be formed with a projecting lip or spout on one or both sides and arranged on steps or on a slope in the flue so that the liquid entering the highest beaker of the series overflows into the second, and each in turn flows over into the next in order continuously.

Mr. Garroway says "this system of concentrating sulphuric acid has been working here (*i.e.* Netherfield Works, Glasgow) for years. The wear and tear is very slight, with hardly any breakages of the beakers."

## ERRATUM.

### THE MANUFACTURE OF CALOMEL IN JAPAN.

BY EDW. DIVERS, M.D., F.R.S. (this Journal, 1894, p. 108).

On page 110, col. 1, line 9, for "roots" read "rods."

## Manchester Section.

CHEMICAL THEATRE, OWENS COLLEGE.

Chairman: Ivan Levinstein.

Vice-Chairman: Edw. Schunck.

Committee:

G. H. Bailey.	P. Hart.
F. H. Bowman.	J. M. Irving.
R. F. Carpenter.	E. Knecht.
G. E. Davis.	W. H. Perkin, jun.
C. Dreyfus.	Sir H. E. Roscoe, M.P.
H. Grimshaw.	C. Traub.

Hon. Local Secretary:

J. Carter Bell.

Bank House, The Cliff, Higher Broughton, Manchester.

The name in italics is that of a Member of Committee that retires at the end of the current session.

The following has been elected to fill the vacancy, and will take office in July next: Committee: W. Thomson.

Meeting held in the Chemical Club on Friday April 6th, 1894.

MR. IVAN LEVINSTEIN IN THE CHAIR.

## INDIA RUBBER (A CRITICISM).

BY WILLIAM THOMSON.

ON the 5th of January a paper was read before this Section by Mr. C. O. Weber on india-rubber, but with special reference to its vulcanisation, in which he brought a considerable amount of criticism against work which I had done and which I think requires a reply. I desire therefore to lay before you to-night a paper giving my criticism of his paper.

He confirms the observation of Tilden that isoprene polymerises into india-rubber. Tilden says: "Isoprene is a very volatile liquid boiling about 35°. Its molecular formula is  $\text{C}_5\text{H}_8$ , and it forms a tetrabromide  $\text{C}_4\text{H}_6\text{Br}_4$ ." Weber says he had some isoprene which he kept for nine months, it had polymerised into india rubber; which he precipitated by methyl alcohol. The alcohol kept in solution "a thick oil boiling at 245° C., and combining with bromine with formation of a tetrabromide."

Weber does not state of what it is a tetrabromide.

He goes on to say that this thick oil is composed of a mixture of substances, 20 per cent. of dipentene (which he has previously informed us boils between 168° and 176° C.)—a large range of difference for a pure substance. The rest, he says, is a mixture of two substances, which, so far, he has failed to separate, because fractional distillation did not avail; and yet the mixture of these three substances, 20 per cent. of which boiled at 170° or thereabouts, had a boiling point, he informs us, of 245° C.

He presumably separated the dipentene from this mixture of three bodies and left two bodies. (I should like to know how he ascertained there were only two bodies present), but for the sake of argument I will assume there were only two; he states that he determined the molecular weights of the mixture of these two substances by Beckmann's freezing-point apparatus, and he remarks that the results he obtained "strongly indicate this mixture to consist of condensation products of the formula  $\text{C}_{25}\text{H}_{36}$  and  $\text{C}_{40}\text{H}_{64}$ ."

I have understood that in using Beckmann's apparatus an important condition was that the substance under test should be pure. It would be interesting to know how Weber obtained any formula at all from such an experiment, and still more interesting to know how he succeeded in finding any indication of the formulae for both.

Tilden, in his paper (Chem. News, Vol. 65, p. 265), says, the constitutional formula of isoprene is not known with certainty, but it must be selected from the five following formulae, three of which he classes as "derivatives" of

allene, " $\text{CH}_2 = \text{C} = \text{CH}_2$ ," and two as derived from crotonylene,  $\text{CH}_2 = \text{CH} - \text{CH} = \text{CH}_2$ ." I should like you to contrast Weber's statement with this. He says:—

"At the present time the constitution of isoprene itself is very doubtful. We only know that it contains two ethylene bonds, and for that reason must be a derivative either of allene,  $\text{CH}_2 = \text{C} = \text{CH}_2$ , or of vinyl ethylene,  $\text{CH}_2 = \text{CH} - \text{CH} = \text{CH}_2$ ."

Whilst pointing out the fact that india-rubber is in some way connected with isoprene ( $\text{C}_5\text{H}_8$ ), he admits that we do not understand the constitution of that body and therefore, as he says, that does not help us in arriving at any idea as to the molecular constitution of india-rubber; but after pointing out that isoprene and also di-pentene contain two ethylene bonds, he concludes that india-rubber differs very essentially from the terpenes and polyterpenes as at present understood.

I have always been under the impression that there was a great difference between the "absorption" of sulphur by india-rubber, and the chemical combination of that body with it, but Weber puts it that various writers describe vulcanisation as an "absorption" of sulphur. India-rubber is capable of absorbing sulphur without becoming vulcanised. Bolas says it will absorb 50 per cent. by putting it in a bath of melted sulphur without becoming vulcanised in the slightest degree.

After discussing the question as to whether sulphur produces an "addition" or a "substitution" product, and then—speaking of the action of chloride of sulphur on india-rubber, for which he says Burghardt gives a rational explanation by saying that the displaced hydrogen combines with the chlorine of the chloride of sulphur, forming hydrochloric acid, which Weber afterwards says really does not happen—he goes on to say "diametrically opposed to these statements is Thomson, who gives it as his opinion that the vulcanising action of chloride of sulphur is due to the chlorine, and not to the sulphur of the compound. He asserts that india-rubber vulcanised with chloride of sulphur contains only *minute traces of sulphur* as an impurity, but large quantities of combined chlorine, and that the same is the case with the oils converted into india-rubber substitute by means of chloride of sulphur."

In answer to this, I give, as follows, an extract from my paper to which he refers. "Recently I analysed three samples of rubber substitute . . . the first contained of sulphur 3.4, and of chlorine 7.6 per cent. The second contained of sulphur 4.56, and of chlorine 8.22 per cent., and the third 2.67 of sulphur, and 7.90 of chlorine." Weber does not seem to recognise that there are two chlorides of sulphur in commerce, the "yellow" and the "dark," and that the yellow chloride often contains a considerable quantity of the dark sulphur di-chloride. According to Fawcitt's figures, the dark chloride contains about 52 per cent. of the di-chloride, which would give a product containing a much larger quantity of chlorine than the mono chloride, and this body might have been used in producing the vulcanised oil which I analysed.

I think I was the first to point out that, after vulcanising with chloride of sulphur, both sulphur, and chlorine were afterwards found in chemical combination in the rubber and in the substitute, and I simply accentuated the fact that in the analyses I made I found more chlorine than sulphur.

The presence of these two bodies in chemical combination was not previously recognised, and Weber, whilst acknowledging afterwards that both bodies enter into combination with the rubber, goes on to say: "Thomson's results were considered rather startling when first published, but I think his analytical method by which he obtained them is more startling still, and I consider it a matter of great surprise that it never has been challenged. This method consists in fusing the vulcanised rubber or rubber substitute with caustic potash, dissolving the melt in water, &c." . . . "It is a matter of surprise that Thomson should have found any sulphur at all in those compounds."

I might refer him to various works on organic chemistry which actually give the process which he condemns in such unmeasured terms. I would refer him, for instance, to Berntsen's *Organische Chemie*, page 6. There it suggests

fusion of organic substances containing sulphur with caustic soda and of a small quantity of an oxidising agent, which is of course necessary when no further precautions are taken, such as by the use of hydrogen peroxide, which I adopted for conversion of the whole of the sulphur compounds contained in the soda mixture into sulphates.

He proceeds, however, to say: "at any rate before applying" that process "to the analyses of these unknown compounds, its suitability for the purpose ought to have been tested. After this, neither the method nor the startling results it furnished would ever have got into print." In reply to this criticism I may say that I tested the process against the one which I had previously employed, viz., by decomposing the organic compounds by means of fuming nitric acid in hermetically sealed tubes heated under pressure, and finding the results agreed well, I adopted the soda process as the much simpler one of the two.

I asked Dr. G. R. Winder if he would kindly repeat the process described by Weber as absurd, in comparison with the nitric acid process, and give me the results, without giving him any reasons for it. He reported as follows:—

At your request I have made determinations of the sulphur contained in the two samples of india-rubber substitute which you gave to me: (a) by the nitric acid method, and (b) by fusion with caustic soda and subsequent oxidation of the solution obtained from the melt.

(a.) The nitric acid method was carried out by heating 7 grains of the substance with 240 grains of fuming nitric acid in a hermetically sealed glass tube which was heated to 265° F. during the first day. The tube was opened at the capillary end after allowing it to cool, and the  $\text{CO}_2$  allowed to escape; it was resealed and further heated to 345° F. during the second day, again opened and resealed as above described, and again heated during the third day to 385° Fahr. On opening on the last occasion practically no pressure was found, although I had a large excess of nitric acid left. The contents of the tube were then washed out, diluted with water, and the sulphuric acid estimated by means of barium chloride in the usual manner.

(b.) The caustic soda method was carried out by mixing about 10 grains of the india-rubber substitute with finely powdered caustic soda, and fusing in a silver capsule, dissolving the melt in water, oxidising with a few drops of bromine, slightly acidifying with nitric acid, and precipitating with barium chloride. Peroxide of hydrogen would of course answer the same purpose, but I had none at hand which was quite pure. The following results were obtained:—

	Per Cent. of Sulphur India-rubber Substitute.
By nitric acid process . . . . .	7.87
By caustic soda process . . . . .	7.91

The second sample tested in the same way gave in duplicate:—

	Nitric Acid Method.	Caustic Soda Method.
	Per Cent.	Per Cent.
Sulphur, first determination . . . . .	8.29	8.66
" second " . . . . .	8.33	8.72

You will observe that the sulphur given by the caustic soda method is rather higher than that obtained by the nitric acid process; that I account for by the fact that Dr. Winder precipitated his barium sulphate from a dilute nitric acid solution, the presence of free nitric acid preventing the complete precipitation of the barium sulphate.

All I was told about the samples of rubber substitute which I analysed was that chloride of sulphur had been used in their manufacture. What kind of chloride of sulphur or what kind of oil was employed, or whether that oil was first oxidised, I had no idea. The point I wished specially to make was that, contrary to the generally accepted ideas, chlorine as well as sulphur entered into



combination with the organic compounds, producing vulcanised rubber or vulcanised oil, and this my critic entirely overlooks.

Weber, having summarised what others had done, proceeds to give his own work, as he puts it, "with a view to arrive at a definite settlement of these points, and at the same time to clear up the chemical mechanism of the vulcanisation process in general."

After considering the work of Guthrie, who showed two ethylene and two amylene molecules, after being acted upon by chloride of sulphur, as being joined together by the two atoms of the diad element sulphur, with the two atoms of the monatomic element chlorine, attached each to one carbon atom of each of the molecules, ready to go off on the slightest provocation, he goes on to say that "quite recently Ulzer and Horn, and especially Henriques, proved conclusively that these india-rubber substitutes contain sulphur and chlorine in the same proportions in which they form chloride of sulphur." He goes on to say, "These results are of course directly opposed to Thomson's figures, which, however, have been obtained by such a questionable method that they cannot be taken seriously."

Now, I wish to call your attention to Henriques' results, and as they have been translated by Weber himself he ought to be thoroughly acquainted with them. They do not show that sulphur and chlorine exist in the proportions in which they exist in chloride of sulphur. The first line of the following table gives the percentage of sulphur and chlorine respectively in chloride of sulphur ( $S_2Cl_2$ ), which Weber assumes Henriques employed. The figures underneath show the relative quantities of chlorine to sulphur found in five different samples of substitute examined by Henriques, and the third column of figures shows the difference between what was found and what ought to have been found if the assertion, which Weber says has been "conclusively proved," were correct.

HENRIQUES' ANALYSIS OF INDIA-RUBBER SUBSTITUTE.

	Quantities of Sulphur and Chlorine present.		Percentage of Differences from the Theoretical Quantities.
	Sulphur.	Chlorine.	
In chloride of sulphur ( $S_2Cl_2$ )	47.41	52.59	0
White Substitutes:—			
A .....	56.14	43.86	+ 8.73
B .....	51.28	48.72	+ 3.87
C .....	48.16	51.84	+ 0.75
Castor-oil Substitutes:—			
With minimum of $S_2Cl_2$ .	41.84	58.16	- 5.57
With maximum of $S_2Cl_2$ .	54.22	45.78	+ 6.80

These figures show from 5½ per cent. of sulphur to chlorine below the theoretical proportion to 8½ per cent. above it, and whilst Weber says that Henriques has proved conclusively that these india-rubber substitutes contain sulphur and chlorine in the same proportions in which they form chloride of sulphur, Henriques himself says: "On the action of chloride of sulphur on oils very little is known," and asserts that "part of the fatty acid of the substitute must therefore be contained in it in the form of a sulphonic acid produced by some oxidising influence upon the chloride of sulphur"; and further he says, when chloride of sulphur acts upon oils part of it is converted into sulphurous and hydrochloric acids."

Weber then goes on to explain how he made an experiment to determine whether hydrogen was liberated on treating a solution of india-rubber in benzene with chloride of sulphur, and finding that no hydrogen was liberated he remarks:—

"Already now it can no longer be doubted that Fawsitt's observations of an evolution of hydrogen in this reaction

must be erroneous as well as the statements of Burghardt, Terry, and Thomson, respecting evolution of hydrochloric acid." By this he assumes that I have said that hydrochloric acid is liberated when chloride of sulphur acts on india-rubber. I state precisely the opposite. I say in my paper "In the cold vulcanisation process it is generally supposed that the chloride of sulphur breaks up, the chlorine combining with the hydrogen producing hydrochloric acid which is liberated. This reaction is clearly not the correct one."

With a view of elucidating this subject further, Weber makes two experiments, both of the same character, in which he treats rubber dissolved in benzene in the proportion of one gram of rubber to one gram of chloride of sulphur, and he obtains a compound which on analysis he finds to be a body of very definite composition, viz.,  $C_{10}H_{16}S_2Cl_2$ . Now india-rubber consists of two distinct bodies, the one more soluble in benzene or in carbon bisulphide than the other, yet he finds these two bodies (according to his analysis) to be one having the composition above mentioned. He admits that india-rubber does not act like a definite chemical substance, because a small quantity of chloride of sulphur equal to say 5 per cent. of sulphur on the rubber employed, converts the whole into a homogeneous body insoluble in the usual solvents, whilst you can go on adding chloride of sulphur as it were drop by drop, each drop acting upon the whole rubber present producing always a homogeneous compound till you have ultimately converted the whole into a hard substance or vulcanite. He does not try the effect of two grms. of chloride of sulphur, for instance, to one gram of rubber; had he done this it is probable that he would have obtained a compound which would not have given on analysis results so wonderfully in accordance with any formula.

He then goes on to state that when pinene-nitroschloride is boiled with alcoholic potash the chlorine is eliminated and the appearance of the product is not altered, and from this he argues that when india-rubber is similarly treated the result must be the same. Fawsitt says (this Journal Vol. V. p. 640) "These samples show on examination that the yellow chloride of sulphur acts quite differently from the dark; there is no tendency with the former to harden the rubber even although used as strong as 1 to 15 (of carbon bisulphide) whilst with the latter 1 to 60 has such a tendency and renders the rubber inelastic."

This hardening of the rubber being due to the presence of chlorine in the molecule, I presume, according to Weber, that the hardness should be removed by boiling in alcoholic potash. This suggests two points: first, he might have tried the experiment before making such very definite statements; and second, what formula would he assign to this rubber which has been so hardened by excess of chlorine?

He states that chlorine does not vulcanise rubber, because, although halogen derivatives of india-rubber resemble the vulcanised material, they are clearly distinguished from it by their great and complete solubility in benzene and chloroform. From this it appears that according to Weber the object of vulcanisation is to render the rubber insoluble in benzene, &c. I have always understood that the object of vulcanisation was to keep rubber soft at low temperatures and to prevent it from becoming plastic at the higher temperatures. If the halogens effect this I should say they vulcanised the rubber, and if the rubber so vulcanised were soluble in benzene that would be a great advantage in enabling old vulcanised rubber to be remanufactured. I have, however, in this bottle of benzene some of Fawsitt's rubber vulcanised by the iodide of antimony, and you will see that it is quite insoluble. Further, I dissolved india-rubber in carbon tetrachloride and then passed chlorine through the solution which became warm and at the same time gradually precipitated the chlorinated compound as an insoluble jelly. The excess of chlorine passed through the tetrachloride solution was then conducted through a small quantity of water in a test tube to find whether any hydrochloric acid was produced. This would have been indicated by a rise of temperature in the water, but the water remained quite cold during the experiment, and when the carbon tetrachloride was examined no indication of the production of hydrochloric

acid was noticed either by the fuming produced when hydrochloric acid is present or by the smell. These results show, I think, that Weber was mistaken in his observations on the action of solvents on the halogen compounds of india-rubber.

Having proved to his own satisfaction that india-rubber combines with a maximum amount of 23.8 per cent. of sulphur and 26.1 per cent. of chlorine, Weber finds that 4.26 per cent. of sulphur and 4.71 per cent. of chlorine completely vulcanises the whole mass. This he arrives at by taking the experiments of Henriques, who showed that rubber vulcanised with chloride of sulphur so as to leave in it 5 per cent. of sulphur was rather over-vulcanised, whilst rubber containing a tenth of that quantity was under-vulcanised. On such data does he venture to predict the molecular constitution of rubber. He assumes a rubber containing 4.26 per cent. of sulphur to be properly vulcanised, and he calculates if 23.8 per cent. represents the molecule as  $C_{10}H_{16}$ , then 4.26 per cent. of sulphur would represent a total molecular weight of 1,300, and this almost exactly corresponds to  $C_{10}H_{16}S_2Cl_2$ . It might, however, with equally good reason be taken as Fawcitt gives it, that rubber vulcanised by chloride of sulphur so as to contain 0.791 per cent. of sulphur is well and properly vulcanised and this on the basis in question would bring the molecular weight up to over 8,000, thus making the molecule about  $C_{10}H_{16}S_2Cl_2$ . I am quite sure, as Weber puts it, that the molecular weight for india-rubber which he has deduced will not be accepted without comment, or even with it. In short, I think his ideas of india-rubber are altogether at variance with the character of the material whose molecular weight he has been trying to determine.

#### DISCUSSION.

DR. CARL OTTO WEBER said in reply: this paper of Mr. Thomson resolves itself into a criticism *a tout prix* of my paper, and some of his arguments appear to be based upon an utter misconception or disregard of my words. His remarks about isoprene ascribe to me claims I never raised. Why Mr. Thomson should wonder, in the face of Gladstone and Hiltbert's paper, that I express myself with some reserve with regard to the constitution of india-rubber as a polyterpene, I am at a loss to understand. After taking my remarks on Burghard's view of the vulcanising action of chloride of sulphur to be an acceptance of them, he proceeds to furnish proof of the correctness of his analytical method by quoting the results obtained by Winder in testing it against Carus' nitric acid method. That results so satisfactory as those can be obtained I do not doubt; but I maintain that in working Thomson's method without such a check, the results must always contain an element of considerable uncertainty, as they are apt to show most unaccountable variations. This is not my experience alone, for if anyone has proved it, Thomson has done so himself by his own analyses, in which he finds only from one-half to one-third the amount of sulphur observed by Dr. Winder. That commercial chloride of sulphur sometimes contains small quantities (never exceeding 6 per cent.) of  $S_2Cl_2$  is no explanation of the exceedingly low percentages of sulphur he observed, as, even by preparing india-rubber substitutes with pure  $S_2Cl_2$ , his figures could not be correct. Altogether, I insist that, in whatever manner prepared, there is not, nor ever was there, an india-rubber substitute in existence containing such small amounts of sulphur as observed by Thomson. In india-rubber vulcanised by  $S_2Cl_2$ , Thomson finds 2.6 per cent. S and 6.31 per cent. Cl, and I maintain that these figures are also absolutely impossible as far as the percentage of sulphur is concerned. In vulcanising with chloride of sulphur a loss of chlorine might under certain circumstances occur, but no sulphur can escape. The loss or deficiency of sulphur shown in Thomson's analyses is simply due to his method of estimating it. For this reason all the conclusions Thomson derived from his figures are necessarily erroneous. The severe manner in which Thomson criticised the formula  $C_{10}H_{16}S_2Cl_2$ , which I calculated for the compound obtained on the interaction of 50 grms. of india-rubber and 50 grms. of chloride of sulphur, would have been justified if he had

proved that by using 60 grms. of chloride of sulphur or more, compounds of an altogether different composition are obtained. This, however, is not the case, provided the concentrations given by me are adhered to. Nevertheless, I have reasons to believe that even compounds  $C_{10}H_{16}(S_2Cl_2)_2$  and  $C_{10}H_{16}(S_2Cl_2)_3$ , corresponding to  $C_{10}H_{16}Br_4$  and  $C_{10}H_{16}Cl_6$ , can and do exist, but this forming partly the subject of my next paper, I will not discuss this point any further to-night. Thomson missed the only undoubted chance my paper affords him for correcting me, and this is with regard to the statement I made that the whole of the chlorine can be eliminated from the compound  $C_{10}H_{16}S_2Cl_2$ , by boiling it with alcoholic potash, which should read potassium ethylate, *i.e.*, a solution of metallic potassium, not potash, in alcohol. Thomson next goes on to criticise the manner in which I arrived at the formula  $C_{10}H_{16}$  for india-rubber, taking, however, not the slightest notice of the important experiment with mixtures of  $C_{10}H_{16}S_2Cl_2$  and unvulcanised india-rubber. He also overlooks that I distinctly stated the probability of even that formula still requiring to be doubled in order to express the true molecular weight of india-rubber. By passing a current of chlorine through a solution of india-rubber, Thomson obtained a compound which he found to be insoluble in benzene, and he concludes from this that my statement as to the solubility of india-rubber chloride in benzene is incorrect. His compound, however, can no more be described as india-rubber (hexa-) chloride, than a chloride of cymene could be called a terpene chloride. Gladstone and Hiltbert already showed that by passing a current of chlorine through a solution of india-rubber, substitution products of the latter are obtained, which indeed are insoluble in benzene. Thomson's suggestion that, according to my statements, india-rubber over-chlorinated in the vulcanisation process ought to be rendered soft and pliable again by abstracting the excess of chlorine, is due to a similar misunderstanding. In abstracting chlorine from an addition product of chlorine and india-rubber, we invariably deprive the molecule of an equivalent amount of hydrogen, so that the eventual result is again a compound as different from india-rubber as cymene is from terpene. I certainly did not say that the object of vulcanisation was to render the india-rubber insoluble in benzene, but pointed out that the solubility of the halogen addition products of india-rubber, coupled with their general chemical instability and their sensitiveness against changes of temperature, sharply distinguished them from vulcanised india-rubber. Even Fawcitt's vulcanisation with iodide of antimony proves nothing to the contrary, and, from Thompson thinking that it does, I must infer that he has not studied the vulcanising action of this compound, which is most interesting, and which I will explain in my next paper on the subject. The chemistry of india-rubber offers a vast field of work to chemical investigators, whether from a purely scientific, technological, or analytical point of view.

Meeting held Friday, 4th May 1894.

MR. IVAN LEVINESTEIN IN THE CHAIR.

#### ON THE ANALYSIS OF INDIA-RUBBER ARTICLES.

BY CARL OTTO WEBER, PH.D., F.C.S.

WITHIN the last two years considerable progress has been in the analysis of india-rubber articles, chiefly owing to the successful exertions of R. Henriques (this Journal, 1893, 467) in this direction. There are, however, still a great number of moot points to be settled by further investigation before we shall have arrived at a thoroughly reliable and satisfactorily workable method of analysis of these very complex compounds and mixtures. Terry, in a paper read before this Society (this Journal, 1892, 970),

expresses an opinion that from the fact of no analytical methods for the analysis of india-rubber articles being known, it should not be inferred that they do not exist. But he does not tell us what these methods consist in, and I must confess to a deep-rooted suspicion against secret analytical methods. However, I believe that I possess a knowledge of the method Terry was hinting at, and I shall show later on that it was all the better for being kept secret, its secrecy being indeed its strongest point.

Before Henriques published his investigations on this subject, the analysis of india-rubber articles essentially consisted in the estimation of the ignition residue of the samples, the nature of the organic matter either not being considered at all, or vaguely supposed to represent india-rubber of some kind. The determination of the residue on ignition of a sample of an india-rubber article does hardly deserve the name of an analytical method; it is at best a rough-and-ready test. With regard to the estimation and separation of the organic constituents of manufactured india-rubber, it appears that no serious attempts have ever been made to overcome the numerous difficulties and solve the many vexed questions involved in this task.

It is well known that the majority of india-rubber goods are not manufactured from pure india-rubber only, but contain, besides mineral matters, more or less considerable quantities of india-rubber substitutes (oil substitutes), recovered rubber, various bituminous substances, resins, paraffin wax, resin oil, mineral oil, and sometimes even fatty oils. Almost completely overlooked was the fact that the various brands of india-rubber are by no means homogeneous compounds, but represent mixtures of hydrocarbons of the nature of india-rubber proper with variable quantities of resinous substances, which are of course also to be found in the manufactured articles. The proportions of hydrocarbon and resin vary greatly in different brands of india-rubber, but seem to be fairly constant for the same brand. The most noteworthy property of these india-rubber resins, which very likely are products of the oxidation of the india-rubber hydrocarbon, is the readiness with which they combine with large quantities of sulphur. Indeed, several of the inferior brands of india-rubber are said to be vulcanisable. This is, however, not intrinsically true, for although it may be found that they cannot be vulcanised with such quantities of sulphur as are supposed to be admissible for practical purposes, this is only on account of the resin taking hold of the sulphur before the india-rubber has time to combine with it. If we free such brands of india-rubber of their resin, vulcanisation of the rubber will no longer be found impossible.

On the resinous constituents of india-rubber, Terry (this Journal, 1889, 218) read some time ago a very interesting paper giving the percentage of resinous matter contained in various brands of india-rubber. His results were obtained by extracting the finely-cut india-rubber in a Soxhlet tube with alcohol. Having observed that this method is rather tedious, an excessively long time being required for the complete extraction of the resinous matters, I dissolved the dry samples of india-rubber (50 grms.) in 1,000 cc. of benzene, filtered through silk gauze, and added 100 cc. of the filtrate very slowly to 200 cc. of alcohol. The whole of the india-rubber was precipitated while the resin remained in solution. The latter was then filtered, the india-rubber remaining on the filter, washed with some hot alcohol, and subsequently the whole of the filtrate evaporated in a weighed flask.

As will be seen from the table in next column, some of my figures, especially those referring to the better qualities of india-rubber, agree very well with Terry's results. Most of my figures are, however, rather higher than that author's, but this may perhaps be due to variations existing between different samples of the same brand of india-rubber.

The melting points Terry observed for these resins are, as he suspects himself, of very little value; as a matter of fact in many cases the melting point can scarcely be determined with accuracy, owing to these resins without exception being mixtures of saponifiable resin acids and indifferent resins. A thorough investigation of these resins and their constituents, as well as of the resin-free india-rubbers would be most valuable, and, no doubt, also be

Name of India-Rubber.	Resin (Terry).	Melting Point.	Resin (Weber).
	Per Cent.	°.	Per Cent.
Para .....	1.2	5	1.5
Ceara .....	1.5	2	2.1
Columbian .....	2.5	..	3.8
Mozambique .....	3.0	18	3.2
Rio Janeiro .....	5.8	64	5.2
Madagascar .....	6.1	..	8.2
Sierra Leone .....	7.4	..	9.7
Borneo .....	7.9	28	11.7
Assam .....	9.3	82	11.3
Mangalore .....	10.5	18	13.1
African Ball I. ....	18.5	38	22.8
" " II. ....	22.8	40	26.1
" " Biko .....	41.2	20	63.9

productive of important technical results, although I admit that from the point of view of chemical manipulation the subject is certainly not very attractive.

All these resins being readily soluble in alcohol and alcoholic posash, it is evident that some attention will have to be paid to them in the analysis of india-rubber articles. All the more so as in certain cases it may be possible to infer the quality or average quality of the india-rubber contained in an article, from the percentage of resin present. It must, however, not be forgotten that resinous matters may be formed in manufactured india-rubber by oxidation, as shown by Burchardt and Thomson, and it is doubtful whether these resins are any different from those occurring in the crude india-rubber. For this reason I content that Burchardt's statement "that the amount of oxygen combined with the caoutchouc is an index of the amount of deterioration which it has undergone," cannot be applied all round indiscriminately, and must lead to serious errors in cases where the rubber articles have been manufactured with addition of natural resins and gums, between which and the india-rubber resins I have not been able to discover any characteristic difference.

It is clear that the separation of these resins from the insoluble components of india-rubber articles offers very little difficulty, but the same cannot be said with regard to the separation of the india-rubber from india-rubber substitutes; and great credit is due to Henriques for the at once exceedingly simple and satisfactory way he devised to attain this end. I must, however, mention that a process for the same purpose, originated, I believe, by Mr. Boias, was communicated to me some two or three years ago by Mr. Carter Bell. This process consists in treating the finely-ground or cut sample with an ethereal solution of peroxide of hydrogen, whereby the india-rubber is oxidised into a compound easily soluble in alcohol, while any india-rubber substitute present remains unaffected, so that the loss on extraction of the oxidised sample with alcohol represents india-rubber. The residue from this extraction is then heated and the india-rubber substitute distilled off, any mineral matter present remaining behind. I have spent a great deal of time in the attempt to obtain, by the application of this method, the results I was told it yielded, but have been utterly unsuccessful. Solutions of peroxide of hydrogen containing from 5 to 50 volumes of available oxygen proved equally inefficient. Oxidation, indeed, did take place, but it proceeded exceedingly slowly. As a matter of fact, I have not been able to effect complete oxidation by three weeks' treatment with an ethereal solution, containing 32 volumes of available oxygen.\* About 19 per cent. of the total india-rubber present I found to be

\* To give an idea of the exceedingly slowness with which the oxidation proceeds, I may say that after all this time the other still contained 13 volumes of available oxygen, while the greater part of the india-rubber present was still unoxidised.

soluble in alcohol after this time. The alcoholic solution contained not the slightest trace of sulphur, but the percentage of sulphur in the unoxidised part of the sample was found to be very much higher than before; in fact, the total quantity of sulphur present had remained exactly the same. This seems to show either that only the unvulcanised india-rubber present is oxidised, or that at any rate the oxidation is the mere incomplete the higher the degree of vulcanisation of the india-rubber. This observation reveals an important relation between the liability of an india-rubber article to suffer oxidation and its degree of vulcanisation, but it proves at the same time that, for the analysis of india-rubber articles, particularly for the separation of india-rubber from rubber substitute, the above-mentioned method is impracticable.

A complete analysis of an india-rubber article will have to contain the following data:—

1. India-rubber.
2. India-rubber resins.
3. India-rubber substitutes.
4. Free fatty oils, mineral oils, resin oil, resin, paraffin wax, and bituminous bodies.
5. Sulphur of vulcanisation.
6. Sulphur and chlorine in substitute.
7. Free sulphur.
8. Mineral matters.

This enumeration is of course not meant to represent the order in which these items should be estimated; this will largely depend upon the methods employed for the isolation of the various compounds. In any case the estimation of the mineral matters present will for obvious reasons always be the last operations. The other substances occurring in india-rubber articles may be divided into two groups, one of which embraces all those of the above-named compounds which at temperatures not exceeding 100° C. are practically insoluble in all organic solvents, while the second group contains substances which in most of the usual organic solvents are very easily soluble. These two groups therefore comprise respectively:—

A (insoluble).	B (easily soluble).
1. India-rubber.	(6.) India-rubber resins.
2. India-rubber substitute.	(7.) Oils, resins, paraffin wax, &c.
3. Sulphur of vulcanisation and chlorine in combination with rubber.	8.) Free sulphur.
4. Sulphur and chlorine in substitute.	
5. Mineral matters.	

Besides these analytical data, some of the physical properties of the samples may be found of importance in judging their quality. Such properties are the specific gravity, tensile strength, dry heat test at 270° F. for one hour, and moist heat test at 320° F. for three hours. I do not propose to enter upon a discussion of these physical tests, but I may say that they are undoubtedly of very high practical value, enabling one to determine within a few hours whether a given sample comes up to a certain standard or not. On the other hand, however, I may just observe that the results of these tests, while giving a fair idea of what we may call the efficiency of an india-rubber sample, give only a very crude and deceptive notion of its intrinsic quality. For this reason I hold that these physical tests should be supplemented by an analysis, which for ordinary purposes might be sufficient if giving the percentages present of india-rubber, organic bodies other than india-rubber, and mineral matter. I shall show further on that such an analysis can comfortably be made in about half a day.

Having above divided the constituents of india-rubber articles in two groups, according to their solubility in organic solvents, it is a matter of course that we shall begin the analysis by separating a sample to be examined into these two groups. Although a number of experiments on the solubility or insolubility of india-rubber, rubber substitute, and the substances mentioned above in the B group have been published, I thought it advisable to submit that question

to a systematic re-examination, as I believe some of the statements made in regard to this subject to be either misleading or obviously incorrect.

All india-rubber resins are soluble in ethyl alcohol, methyl alcohol, acetone, ether, chloroform, benzene, and bisulphide of carbon. The three last-named solvents will also dissolve unvulcanised india-rubber, which not unfrequently occurs in india-rubber articles, and for this reason they cannot be considered suitable for the intended separation. Ethyl ether can scarcely be called a solvent for india-rubber, although it seems to a slight extent capable of dissolving the non-resinous constituents of some of the inferior qualities of india-rubber. It may therefore be advisable to reject even ether as a solvent for our purpose. Mixtures of alcohol and ether, in which the volume of the latter does not exceed twice the volume of the alcohol, are quite incapable to dissolve any india-rubber, but this mixture has the drawback of not being suitable for extraction in a Soxhlet tube, owing to its boiling point not being constant. A mixture of benzene and methyl alcohol (1 : 2), which I recommended some time ago for the extraction of oily and resinous substances from india-rubber, answers very well, and has the great advantage of a constant boiling point (56° C.). I have, however, abandoned the use of this solvent in favour of acetone, which is an excellent solvent for all the substances here in question, but has not the slightest action upon india-rubber and vulcanised oils. Acetone has the further advantage, which will often be found very useful in the analysis of oily and resinous matters, to be miscible in all proportions with water, as also with all of the usual organic solvents. But whichever of these solvents be employed for the separation of the constituents of an india-rubber sample in the two above-mentioned groups, it is most important to have the sample in the finest possible state of sub-division. Cutting the sample is a very wearisome and never particularly satisfactory process, and I prefer to pass it several times between steel rollers revolving in contact. The sample is thereby reduced into a mass of exceedingly fine and easily permeable flakes.

The behaviour of rubber substitutes against solvents was examined by Holde (this Journal, 1893, 469), who found a sample, prepared by heating on the water-bath a mixture of 4 grms. of linseed oil with 4 grms. of chloride of sulphur and 4 cc. of carbon bisulphide, to be quite insoluble in ether. Unfortunately the idea does not appear to have struck that author, that the rubber substitute produced in this extraordinary manner might possibly be different from the commercial article. The fact that there exists the greatest possible difference between the two, renders his elaborate series of experiments with this compound quite valueless, and his conclusions based upon them more or less erroneous. I have analysed upwards of 400 samples of india-rubber substitute and never observed less than 6 per cent. of ethereal extract, but often as much as 11 per cent. Holde further proposes to estimate fatty oils contained in manufactured rubber by extracting 1 grm. of the finely-cut sample with 60 cc. of ether-alcohol (2 : 1) upon a filter, evaporating the filtrate, and drying and weighing the residue. Of the percentage of extract thus obtained he deducts, by way of a correction, 5 per cent., having observed that a number of india-rubber articles, which he merely supposes to be pure, give 5 per cent. of ether extract. The correctness of this method he attempts to demonstrate by impregnating rubber filings with weighed quantities of colza or cotton-seed oil, subsequently extracting them in the above-mentioned manner. Of course he obtains thus tolerably concordant results, but it does not occur to him that extracting the total quantity of oil added to filings of vulcanised india-rubber from the latter is one thing, and extracting the oil added to india-rubber during the process of manufacture another. Any fatty oils entering into the rubber mixture before vulcanisation are bound to undergo an alteration, in part at least, during vulcanisation; in fact, they are more or less converted into substances of the nature of a rubber substitute, which is no longer soluble in ether. Very questionable also is the admissibility of the above-mentioned correction of 5 per cent. which Holde applies to his gross results. Technically pure Pará rubber contains from 0.5 to 2 per cent. of resin. If this rubber is vulcanised by the cold process the extract it yields is not in

excess of the quantity of resin originally present (not taking into account any free sulphur which may be contained in the extract), provided the boiling point of any solvents employed does not exceed 110° C. If, on the other hand, the rubber be vulcanised by the hot process it may yield from 4 to 8 per cent. of extract according to the time and temperature allowed for vulcanisation, both of which depend upon the percentage of sulphur used and also upon the presence or absence of sulphur carriers in the mixture.

The addition of fatty oils to india-rubber mixtures is very little practised nowadays, mineral or resin oils being used

instead. The estimation of these unsaponifiable oils in extracts obtained from india-rubber articles offers little difficulties, but a single extraction of the sample with ether-alcohol is utterly insufficient to remove the whole of these oils. In many cases I found it necessary, and in all cases advisable, to repeat the extraction 10 times in order to completely exhaust the samples. This of course is easily done in a Soxhlet tube, and I prefer, as I stated before, to use acetone as the solvent. These points are well illustrated by the following series of experiments:—

	1st.	2nd.	3rd.	4th.	5th.	10th.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
I. White substitute .....	4.82	6.58	8.16	8.73	8.91	8.98
II. Black substitute .....	12.22	15.01	16.82	16.75	20.55	20.74
III. Mozambique rubber ( $S_2Cl_2$ vulcanised) containing 3 per cent. of resin .....	2.10	2.74	3.04	3.91	5.02	3.95
IV. Mangabeira rubber ( $S_2Cl_2$ vulcanised) containing 10.8 per cent. of resin .....	7.56	9.33	10.78	10.78	10.80	10.80
V. { 30 per cent. white substitute } { 50 per cent. Mozambique } } $S_2Cl_2$ vulcanised .....	3.28	5.24	6.41	7.19	7.65	7.97
VI. { 70 per cent. black substitute } { 30 per cent. Mangabeira } } vulcanised with 6 per cent. S .....	9.43	13.42	18.22	21.06	21.41	21.41
VII. { 10 per cent. codza oil } { 90 per cent. Mozambique } } $S_2Cl_2$ vulcanised .....	6.11	7.50	7.90	8.02	8.02	8.05
VIII. { 15 per cent. castor oil } { 85 per cent. Mangabeira } } vulcanised with 6 per cent. S .....	9.33	10.43	10.96	10.98	11.04	11.13
IX. { 10 per cent. mineral oil } { 90 per cent. Mozambique } } $S_2Cl_2$ vulcanised .....	9.81	10.65	11.58	12.08	12.60	12.82
X. { 15 per cent. resin oil } { 85 per cent. Mangabeira } } vulcanised with 6 per cent. S .....	14.08	17.55	21.91	27.51	28.49	28.58
XI. { 15 per cent. resin oil } { 40 per cent. black substitute } { 45 per cent. African ball contain- } ing 18.5 per cent. of resin } } vulcanised with 8 per cent. S .....	18.08	23.98	30.08	34.90	36.92	37.11
XII. Tobacco pouch, best quality (Messrs. D. Moseley and Sons, Manchester).	4.22	4.92	5.52	5.97	6.58	6.66

These figures unquestionably prove the insufficiency of Holde's above-mentioned method of extraction; further, the possibility of effecting complete extraction by adopting the means proposed above; and lastly, the impossibility of detecting by extraction the amount of fatty oils added to india-rubber during the process of manufacture.

The estimation of the constituents of the extracts will have to be carried out essentially by the well-known methods of separating fatty oils from mineral oils and resins. I must, however, point out that the results of this separation will not admit of any definite interpretation respecting the nature of the india-rubber contained in the sample, or regarding the quantity of fatty oils or resin employed in the manufacture of the article. The reasons for this are obvious. There exists no fundamental difference between the resins contained in india-rubber and the other natural resins, both being genetically closely related to the terpenes or polyterpenes. For this reason the smell of burning india-rubber resin is also identical with that of the other resins. The estimation of any fatty oils added during manufacture is absolutely impossible, if at the same time india-rubber substitutes have been employed. Even the amount of added unsaponifiable matters can only approximately be estimated, owing to the fact that the india-rubber resins as well as the other natural resins are mixtures of saponifiable and unsaponifiable substances in variable proportions. The estimation of the free and combined sulphur in these extracts offers, however, little difficulty, but in all cases where the estimation of the free sulphur offers no

interest, I prefer to estimate simply the total sulphur in the extract, as this figure is sometimes wanted for the estimation of the sulphur of vulcanisation, which in my opinion is one of the most important data of a complete analysis of an india-rubber article. If, however, the separation of the constituents of the acetone, is in most cases a matter of subordinate interest, the total extract nevertheless is a very useful figure, as its amount may safely be taken to be inversely proportionate to the quality of the total organic constituents of an india-rubber sample.

Lately a controversy on a point of some importance with regard to this extraction question has arisen between Henriques and myself. I stated in a former paper (*Zeitschr. f. angew. Chem.* 1893 [21], 22; see abstract this Journal, 1894, 33) that a mixture of one vol. of benzene and two vols. of methyl alcohol had no solvent action whatever upon india-rubber or vulcanised india-rubber. This statement Henriques opposes on the strength of an experiment with a sample of vulcanised Pará rubber, from which, by means of that solvent, he extracted 2.41 per cent. of substance other than sulphur. I have not the least doubt regarding the correctness of Henriques' figure, but what I certainly doubt is that these 2.41 per cent. he extracted consist of either india-rubber or vulcanised india rubber. Part of his extract is certain to consist of india-rubber resin. The remainder is a compound of a resinous nature and containing sulphur. Even when using india-rubber freed from every trace of india-rubber resin, hot vulcanisation with sulphur always results in the formation of a certain quantity of this "resin

of vulcanisation" as we may term it. If the vulcanisation process be well conducted the quantity of this resin formed appears to be very small, seldom exceeding one per cent. But whenever vulcanisation is effected at too high a temperature, or if the treatment, at what might otherwise be a suitable temperature, has been too prolonged, correspondingly large quantities of this resin are formed.

The most important part of the analysis of an india-rubber article, no doubt, is the estimation of the substances above enumerated in group A. The merit to have shown a thoroughly satisfactory way at least to separate india-rubber from india-rubber substitute, is entirely due to Henriques. His method, which consists in the treatment of the sample with alcoholic caustic potash or soda (this Journal, 1893, 467), requires no further comment. With regard to the quantity of substitute found by this method, it is evident that it stands in inverse ratio to the commercial value of the sample, but to ascribe to india-rubber substitutes, as has been done lately (Chem. Zeit. 1894, 310, 329; Lobry de Bruyn and van Leent), a general deleterious influence upon the stability of india-rubber goods is certainly not warranted by facts. The fact that improperly prepared india-rubber substitutes are sometimes the cause of the premature and spontaneous decomposition, can scarcely be considered a valid reason for the wholesale condemnation of all india-rubber substitutes. In numerous cases the manufacture of an india-rubber article from pure india-rubber would amount to a waste of material, which the consumer would be loth to pay for. Of course the cost price of an article can be reduced by addition of mineral matters to the india-rubber, as well as by addition of substitute, but it would be a great mistake to presume that, generally speaking, mineral substances can replace the india-rubber substitute in india-rubber articles. Such an indiscriminate substitution would lead to considerable modifications of the physical properties, especially of the tensile strength (co-efficient of elasticity) and of the resistance against disintegration by friction of the india-rubber articles, so as to render them in some cases quite unfit for technical uses. I am speaking from experience in saying that the use of properly prepared india-rubber substitutes does not involve any danger whatever as regards the stability and serviceability of india-rubber articles, and the prohibition of their use would be about as reasonable as the prohibition of wood pulp or esparto in the manufacture of low and medium class papers.

If a sample of an india-rubber article does not contain any mineral matter the analysis would be practically concluded after the above-mentioned extraction of the india-rubber substitute by means of alcoholic caustic potash, according to Henriques' method. The part of the sample remaining undissolved in this treatment would then simply represent pure vulcanised or partly vulcanised india-rubber. But in many cases an india-rubber sample may contain small quantities of inorganic colouring matters, the presence of which it is sometimes difficult to discover merely by the look of the sample. For this reason I consider it advisable always to dissolve this residual india-rubber, when the presence of the least quantity of mineral matter will readily be detected. For the solution of the india-rubber, vulcanised and unvulcanised, Henriques proposes to boil the residue remaining after the extraction of the rubber substitute, with that fraction of petroleum boiling from 140° to 250° C. This part of Henriques' method I consider very defective, as according to my experience, strongly vulcanised india-rubber will never dissolve in this solvent. Even in cases where complete solution is eventually obtained the time required is often excessively long. I have never been able to dissolve any sample in less than 10 hours. The solutions obtained, as against solutions effected by heating with boiling oil of turpentine, possess the advantage not to resinify on the filter, but their filtration is generally a very tedious and difficult operation. Evolution of sulphuretted hydrogen does not occur in this solution process, provided all free sulphur has been previously extracted, which is always the case if the first extraction is executed with acetone in the manner described above. The complete extraction of all free sulphur is of course of the greatest importance if it should be desired to estimate

the sulphur of vulcanisation in the india-rubber, a figure to which I attach great importance.

The unsatisfactory nature of Henriques' solvent for india-rubber induced me to examine more particularly this point of the analysis of india-rubber. Very obvious is the assumption that the employment of solvents possessing a higher boiling point might lead to more satisfactory results. This, however, was found not to be the case, neither vaseline nor paraffin showing themselves any superior to petroleum as used by Henriques. Indeed I invariably observed the appearance of black, charred-looking masses, as soon as the temperature of the solvent exceeded 230° C. This observation is perhaps not very surprising if we remember that petroleum hydrocarbons of the paraffin and olefin series are rather poor solvents even for unvulcanised india-rubber, forming with it solutions which are peculiarly devoid of the quality known as "body." By considerably diluting these solutions liquids are obtained which cannot be filtered through paper, the filtrate simply consisting of a solution of the india-rubber resin. I believe indeed that these so-called solutions of india-rubber in petroleum hydrocarbons are merely liquid jellies. This makes it perfectly clear that the solution of vulcanised india-rubber in this solvent is not to be thought of. If, therefore, by the application of Henriques' method a solution is obtained, it must evidently be a solution of the products of the decomposition of india-rubber at the temperature of the boiling solvent. The decomposition of unvulcanised india-rubber begins already at 160° C., that of vulcanised india-rubber between from 190° to 220° C., according to the degree of vulcanisation. The products of this decomposition are terpenes or polyterpenes, which are very subject to further polymerisation, thus forming black resinous substances, which, however, are exceedingly difficultly soluble in petroleum hydrocarbons. These polymerisation products will be produced in increasing quantities the higher the temperature at which decomposition begins, or, in other words, the higher the degree of vulcanisation of the india-rubber. What we require therefore is a solvent which will easily dissolve unvulcanised india-rubber as well as the decomposition products and their polymers of vulcanised india-rubber. The boiling point of the solvent should be high enough to induce the decomposition even of strongly vulcanised india-rubber, accordingly it should be above 200° C., but not exceed 210° C. Of the easily accessible and cheap solvents nitrobenzene answers all these requirements, it being an excellent solvent for india-rubber, while its boiling point (208° C.) could not be more suitable. The behaviour of this solvent against vulcanised india-rubber is most surprising. In the cold, quicker on heating the vulcanised india-rubber immersed into this solvent swells considerably, without solution taking place. On prolonged heating the india-rubber becomes transparent, and as soon as the boiling point of the solvent is reached solution ensues within a few seconds. Any mineral matter present quickly settles out, the supernatant solution being perfectly clear and only slightly darker in colour. Henriques' boiling petroleum has no action upon india-rubber substitute, while the boiling nitrobenzene dissolves these compounds as readily and completely as it dissolves vulcanised rubber, so that this solvent not only places at our disposal an efficient means for the estimation of the india-rubber, and, as I shall show later on, of the sulphur of vulcanisation, but at the same time also for the ready estimation of the quantity, and, what is quite as important, of the nature of the mineral matters present. Only the presence of red lead escapes direct detection in this process, it being very quickly reduced to oxide of lead by the boiling solution. If, therefore, the presence of red lead is suspected in a sample, a special test has to be used for its detection. For this purpose I impregnate a few grammes of the well-ground sample with a saturated solution of anhydrous aluminium chloride in pure ether and subsequently heat for two hours in an air bath at 126° C. In the presence of small quantities only of red lead the sample becomes very hard and brittle, while in the presence of larger quantities the sample is converted into a gritty dust and frequently exhibits a strong smell of chlorine. Addition of red lead to india-rubber mixings is, however, a thing very uncommon at present, most manufacturers

knowing full well the inevitable fatal influence of this compound upon india-rubber.

In carrying out this separation of the india-rubber from the mineral matters, 3 grms. of the sample, after extraction with acetone and subsequently with alcoholic potash, are placed in a flask together with about 150 cc. of nitrobenzene. This flask is attached to a vertical glass tube, which serves as a reflux condenser. Solution of the india-rubber ensues within a few minutes after the boiling point of the liquid (208° C.) has been reached. In order, however, to make quite sure of the completeness of the solution, I continue the boiling for about half an hour. This has also the advantage of rendering the solution much easier to filtrate, from which fact we may infer that on boiling the molecule of the india-rubber gradually breaks up into smaller molecules of easier soluble substances. What these are I do not know, but I intend to follow up this reaction by a special research. The solution obtained as described is eventually filtrated, the flask and filter residue being washed with warm nitrobenzene. The weight of the dissolved india-rubber including the sulphur of vulcanisation is then found by weighing the dry residue on the filter, consisting of mineral matter only.

In most cases the figures obtained by means of this process represent the quantities of vulcanised india-rubber and mineral matter actually present. There are, however, certain admixtures employed in the manufacture of india-rubber articles which may considerably detract from the value of these analytical figures. This is especially the case when the samples contain so-called "recovered rubber" or "devulcanised rubber," i.e., old rubber articles which, either by treatment with certain oils, or simply by grinding, have been brought in such a form and state as to be again suitable for incorporation into fresh rubber mixings. The proportion in which fresh rubber and such recovered rubber has been employed in the production of a new article cannot be detected by our present analytical methods, and this is, no doubt, a great defect, as the use of this recovered rubber, which, in many cases, merely consists of ground-up old rubber articles, is very much on the increase.

Peculiar difficulties arise if an india-rubber article contains starchy matters, an addition not unfrequently practised. In the process of vulcanisation the starch does not seem to suffer any alteration, which is not very surprising, considering the temperature at which the vulcanisation process is carried out. The presence of starch can, therefore, be detected qualitatively by means of the iodine test, provided the articles be light in colour. In darkly coloured articles the reaction is generally very doubtful. The quantitative estimation of this starch offers no difficulty, and can be effected without any alteration in the course of analysis as above mentioned. Several experiments showed that ordinary air-dry starch, containing about 18 per cent. of water, is not destroyed by boiling in nitrobenzene.

7.597 grms. of farina, containing 18.2 per cent. of water, were boiled for one hour with 100 cc. of nitrobenzene. No discoloration was observable. The contents of the flask were subsequently filtered, flask and filter being washed with ether; the filter residue was eventually dried at 110° C., and was then found to weigh 6.204 grms., which with the 18.2 per cent. of water contained in the air-dry starch would amount to 7.586 grms. Thus we see that this treatment with nitrobenzene, although subjecting the starch to the very high temperature of 208° C., causes no loss in weight through decomposition. On closer examination of the starch thus recovered, it was, however, found that an alteration had taken place, resulting in the conversion of the starch into soluble starch and very little dextrin. We are, therefore, in a position to estimate very accurately any starch contained in an india-rubber article, by dissolving the sample freed, in the manner before described, from extractive matter and india-rubber substitute, in boiling nitrobenzene. The starch, together with any inorganic substances present, subsequently remains on the filter. The separation of starch and mineral matter is effected simply by treatment of the filter residue with boiling water, the starch being estimated by evaporating the filtrate, drying the residue at 110° C., and weighing. The quantity of starch thus found is, of course, not the actual quantity of commercial starch added to the rubber mixing. This quantity is obtained by increasing the figure by the amount of moisture contained in commercial starch, and which, as I stated before, amounts to about 18 per cent.

Some further difficulty arises if the sample under examination contains asphaltum, the presence of which is frequently indicated by a high percentage of total sulphur, owing to the asphaltum often being employed in the form of a sulphinated product, containing as much as 20 or 22 per cent. of sulphur. Henriques suggested to estimate it by treating the sample, which of course must be free from extractive matters and substitutes, with cold nitrobenzene, in which the asphaltum is readily soluble. This process answers very well. If, however, instead of the natural asphaltum, artificial asphaltum, prepared from gas-tar and pitch, has been employed, the results obtained by this method are somewhat too low, owing to the considerable quantities of finely-divided carbonaceous matter contained in the artificial product. This carbonaceous matter is of course met with later on, together with the mineral matters.

I have prepared some samples on a large scale, in order to test the accuracy which can be attained in the analysis of india-rubber goods containing the above-mentioned components. The india-rubber used for this purpose was Assam, containing 11.3 per cent. of resin. I preferred to experiment with such a medium quality, which shows much better than the high-class rubbers the influence of the india-rubber resins upon the analytical data obtained:—

	I.		II.		III.		IV.	
	Mixed.	Found.	Mixed.	Found.	Mixed.	Found.	Mixed.	Found.
Assam (with 11.3 per cent. of resin) ..	55	46.61	55	47.37	55	47.01	55	47.63
Resin.....	..	8.21	..	7.49	..	7.74	..	7.09
Substitute, white, refined (9.27 per cent. S).	15	15.3	15	15.05	15	14.96	15	14.78
Lead oxide.....	10	11.93	10	9.91	..	..	..	..
White lead. ....	10	7.74	..	..	..	..	..	..
Barium sulphate.....	5	4.88	..	..	..	..	..	..
Vermilion .....	..	..	5	5.02	..	..	..	..
Antimony sulphide .....	..	..	10	10.12	5	4.94	10	9.92
Asphaltum (18.1 per cent. S) .....	..	..	..	..	..	..	15	14.19
Farina (18.01 per cent. H <sub>2</sub> O).....	..	..	..	..	20	16.02 (= 16.53)	..	..
Sulphur (added) .....	5	4.86	5	4.89	5	4.87	5	7.65
Temperature of vulcanisation .....	134° C.		125° C.		125° C.		125° C.	



These results show that the quantity of resin extracted is, without exception, rather higher than corresponds to the proportion of resin contained in india-rubber. The reason of this, as I pointed out before, is to be found in the formation of resinous matters from the india-rubber during vulcanisation, and I may mention that the quantities of such resins formed are smaller if vulcanisation is effected in the presence of energetic sulphur carriers, such as antimony sulphide. White lead seems to suffer partial decomposition into lead oxide and carbonic acid, which accounts for the too high figure for lead oxide and the too low figure for white lead obtained above. The fact that in the second of the above samples the percentage of both vermilion and antimony sulphide are slightly too high is probably due to the original mixture not being perfectly homogeneous. The figure for starch refers to the substance dried at 110° C.; adding to it the proportionate amount of water contained in air-dry farina, the result is seen to be most satisfactory. The sulphur was obtained by estimating the total sulphur and deducting from this the amount of sulphur contained in the india-rubber substitute.

Lately Henriques suggested estimating the free carbon (lampblack) in india-rubber articles, containing, besides india-rubber oils, rubber substitute and asphaltum only, by first separating from the rubber all these soluble constituents by the processes above referred to, and determining by combustion, the proportion of carbon and hydrogen contained in the remaining india-rubber. This suggestion is based upon the fact that all reliable analyses of india-rubber show the relation of C : H = 10 : 16. Any lampblack mixed with the india-rubber will of course lead to a higher figure for carbon, the percentage of which could then be arrived at by simple calculation. This suggestion may be workable, but I certainly prefer to estimate the free carbon by dissolving the india-rubber in nitrobenzene, filtering the solution through a weighed filter, washing with ether, and drying.

The above-described methods enable us to make a complete analysis of practically any india rubber article with regard to the percentage of india-rubber, and of all the admixtures, other than sulphur, usually employed in the manufacture of these goods. The following is a short recapitulation of the various stages of the process :—

- (1.) Extraction with acetone.
  - (a.) Fatty acids and oils in extract.
  - (b.) Saponifiable resins in extract.
  - (c.) Unsaponifiable matters in extract.
- (2.) Extraction of rubber substitutes with alcoholic potash (Henriques' process).
- (3.) Extraction of asphaltum with cold nitrobenzene (Henriques' process).
- (4.) Estimation of india-rubber by means of boiling nitrobenzene.
- (5.) Estimation of starch.
- (6.) Estimation of mineral and carbonaceous matters.

The figures for 1, 3, 5, and 6, I prefer to determine by weighing the extracts direct; 2 and 3 are best determined from the loss of weight of the substance operated upon. It is possible to estimate the quantity of india-rubber substitute present by direct weighing, but no greater accuracy of results is gained thereby. To weigh the quantity of the india-rubber dissolved in the nitrobenzene direct, I have not been able yet to do, as in distilling off the nitrobenzene, volatile decomposition products of the india-rubber escape in considerable quantity.

The estimation of the sulphur contained in india-rubber articles is one of the most important points of their analysis. The various forms in which the sulphur may occur in an india-rubber article differ very much in their influence upon the quality and durability of it, and for this reason the total sulphur, without the knowledge of the various items it is composed of, is of very little interest. The total sulphur of india-rubber articles in the first instance divides itself under three heads :—

- (a.) Free sulphur.
- (b.) Sulphur in organic compounds.
- (c.) Sulphur in inorganic compounds.

The quantity of free sulphur contained in a sample is an item of some importance, as it is well-known that a high percentage of free sulphur invariably proves detrimental to the durability of india-rubber goods—in fact, is one of the most common causes of their decay.

Even more important than the free sulphur is the "sulphur in organic compounds," as it includes the sulphur of vulcanisation. For this very reason, however, the separation of the sulphur in organic compounds into its various forms becomes further necessary. Of chief importance is the estimation of the sulphur of vulcanisation, while the sulphur contained in india-rubber substitutes or asphaltum, which may be present, is generally of subordinate interest only. Natural asphaltum contains about 10 per cent. of sulphur, but the asphaltum used in the manufacture of india-rubber goods sometimes contains as much as 20 per cent. of sulphur, so that a sample containing this admixture often shows a surprisingly high percentage of total sulphur and of sulphur in organic combination.

The sulphur in inorganic compounds is of minor interest. It is estimated in the final residue remaining after the solution of the india-rubber by the well-known methods for the analysis of inorganic sulphides and sulphates. The sulphates contained in the inorganic residue represent always deliberate admixtures. Sulphites are present whenever thio-sulphates have been added in the mixing of the rubber, although small quantities of them may also be formed by the action of the free sulphur upon litharge or zinc oxide at the temperature of the vulcanisation. Sulphides either have been added as such (antimony sulphide, zinc sulphide, vermilion), or their presence is due to the action of the free sulphur upon the before-mentioned metallic oxides. All sulphides, especially those of antimony and lead, play in the vulcanising process the part of sulphur carriers.

From the above it follows that the complete analysis of an india-rubber article requires the estimation of the sulphur in the following states :—

- |                 |  |
|-----------------|--|
| Total sulphur : | a. Free sulphur.                       |
|                 | b. Sulphur in organic compounds.       |
|                 | 1. Sulphur in the acetone extract.     |
|                 | 2. Sulphur in the rubber substitute.   |
|                 | 3. Sulphur of vulcanisation.           |
|                 | c. Sulphur in other organic compounds. |
|                 | c. Sulphur in inorganic compounds.     |

Of the various methods suggested for the estimation of the total sulphur and of the sulphur in organic combination in manufactured india-rubber, only the well-known method of Carius and the one devised by Henriques can be recommended, and I prefer the latter as being for our purposes at once the most reliable as well as the most expeditious.

As regards the analytical import of these various forms of sulphur, I have already mentioned the well-known fact that the amount of free sulphur present in a sample often affords an explanation of the bad-keeping qualities of manufactured rubber. Of course, I do not mean to say that the apparently spontaneous decay of such goods is invariably attributable to any free sulphur which may be present, although the appearance on the surface of india-rubber goods of a greyish efflorescence can always unhesitatingly be put down as due to free sulphur. It is, however, remarkable that this efflorescence does not, as has been generally assumed, consist of pure sulphur, but invariably also contains ammonia sulphate, the quantity of which increases considerably with the age of the efflorescence. The proportion of ammonia sulphate observable in these cases is particularly great in goods which have become hard and brittle owing to excess of sulphur. It occurs to me that this fact furnishes an explanation of the real cause of the danger which is involved in the presence of free sulphur in india-rubber. It is a daily observation that substances in close contact with india-rubber undergoing oxidation, also favour the oxidation of the india-rubber itself, just like the oxides of manganese and copper which, acting as powerful oxygen carriers, are justly held to be the greatest enemies of india-rubber. This action of manganese which I first observed some three years ago is not generally known yet, the perniciousness of copper was first observed and well

demonstrated by Mr. Thomson. Fatty oils have no effect upon india-rubber, provided access of air to the latter be prevented, but as soon as on admission of the air the oil becomes rancid, the decomposition of the india-rubber also begins. Evidently for this reason the quick-drying oils, and those of the non-drying oils which easily become rancid, are so dangerous to india-rubber. Now it has been known for a long time that sulphur in a very fine state of sub-division rapidly undergoes oxidation and upon this fact, I consider, rests the detrimental action of free sulphur upon india-rubber. On the other hand, however, this contention involves the conclusion that this danger of free sulphur only exists where it is present in india-rubber in such a quantity that it exhibits a tendency to migrate to the surface of the goods. This conclusion well agrees with observed facts, but it is nevertheless impossible to state in definite figures at which percentage free sulphur begins to be dangerous. This is, no doubt, due to the fact that a number of substances which may occur in india-rubber articles highly favour the above-mentioned migration of the sulphur by dissolving it. Substances of this kind are the fatty oils, mineral oils, resin oils, and particularly the heavy portions of the india-rubber solvents. Thus it may happen that a quantity of free sulphur which in one case shows no effect whatever, may in another case be responsible for the decay of an india-rubber article.

The estimation of the free sulphur is done in the extract obtained on the extraction of the sample with acetone, which solvent readily extracts the finely subdivided sulphur. It is, however, important to state that not all the sulphur contained in this extract is free sulphur, as whenever the extracted sample contains rubber substitutes, the extract will also contain sulphides of fatty acids, and it further contains sulphides produced by the action of the free sulphur upon the resins, which are always to be found in india-rubber. For this reason the amount of the total sulphur in the extract is generally very much in excess of the free sulphur which is really present. The estimation of the free sulphur I carry out by adding to the above-mentioned extract 20 cc. of a solution of pure sodium sulphide and caustic soda ( $5\text{NaOH} : 15\text{Na}_2\text{S} : 100\text{H}_2\text{O}$ ), and heating this mixture on the waterbath for about an hour. Even in the presence of a large proportion of unsaponifiable matters the whole of the extract dissolves. I then dilute with warm water and precipitate the fatty acids contained in this solution by adding a slight excess of a solution of barium hydrate. The whole is then filtered, the filter well washed, the filtrate subsequently made up to 300 cc., and the sulphur estimated in an aliquot part of it. This method is the only one by which concordant results could be obtained. The following analyses were made by it:—

I. Steam hose: manufactured from 60 per cent. of india-rubber (Madagascar with 8.1 per cent. of resin), 36 per cent. of brown substitute (11.4 per cent. solution in acetone), 4 per cent. of sulphur.

Total extract: 10.9 per cent., containing 1.65 per cent. of total sulphur.\*

Saponified with 20 cc. of sodium sulphide containing 6.154 grms. of S in 100 cc.

Filtrate after saponification made up to 300 cc., 50 cc. of it oxidized with  $\text{H}_2\text{O}_2$ , then precipitated with barium chloride.

Found: 2.1008 grms. of  $\text{BaSO}_4 = 0.2885$  grms. S = 1.731 grms. in 300 cc.

The 20 cc. of sodium-sulphide used for saponification contain 1.2308 grms. of S, consequently we have free sulphur in the extract:  $1.7310 - 1.2308 = 0.5002$ , or free sulphur in the sample analysed: 0.5 per cent. Repeated estimations yielded 0.48, 0.51, 0.48 per cent. respectively.

The examination of the substitute employed in the manufacture of the above sample yielded 11.4 per cent. of an extract containing 5.3 per cent. of sulphur. Consequently only 0.2173 grms. of the total sulphur found in the extract can be derived from the rubber substitute. This quantity, together with the free sulphur, is still by

0.93 per cent. short of the observed quantity of total sulphur in the extract. This 0.93 per cent. of course represents part of the 4 per cent. of sulphur employed in the manufacture of the sample, and it must be contained in the extract in the form of a highly sulfonated resin of vulcanisation. That this is so is also clearly indicated by the total quantity of extract obtained from the sample, which is rather higher than the aggregate of the extractive matter contained in the components of the sample.

II. 60 parts of india-rubber (Madagascar), 40 parts of brown substitute, 9.6 parts (10 per cent.) of sulphur.

Total extract: 14.97 per cent., containing 7.82 per cent. of total sulphur.\*

Found: Free sulphur 5.66 per cent.

Sulphur from substitute (calculated) 0.222 per cent.

Sulphur in resin of vulcanisation 1.94 per cent.

In accordance with what I have previously stated regarding the formation of resins of vulcanisation this analysis shows that their proportion increases with the excess of sulphur employed for the vulcanisation.

The estimation of the sulphur in organic compounds we have above divided under four heads. Of these the first combined sulphur in the acetone extract—is readily obtained by deducting from the total sulphur in the extract, the free sulphur. Articles manufactured with brown rubber substitute may contain a very considerable quantity of combined sulphur in the acetone extract.

The "sulphur in substitute," i.e., in that part of it which is insoluble in acetone, is ascertained according to Henriques' method in the extract obtained on treating the sample with alcoholic potash. This portion of the total sulphur is, however, of little interest unless it be desired to obtain some information regarding the nature of the substitute. For details on this point I refer to Henriques' paper on this subject (this Journal, 1893, 167).

Of considerable importance is the estimation of the sulphur of vulcanisation, which is, no doubt, of the greatest influence upon the serviceability and the durability of an india-rubber article. The question as to the quantity of sulphur combining with india-rubber has been repeatedly discussed, but is very far from being settled. Thus, I believe, is due to the fact that in these discussions the vital point of this question, i.e., the chemical nature of the sulphide or sulphides as which vulcanised india-rubber must be considered, has been altogether neglected. Terry states that by repeated treatment with solution of caustic soda he has been able to reduce the sulphur of vulcanised india-rubber to 0.6 per cent. This I do not doubt, but I certainly do not believe that this sample would be as well vulcanised with 0.6 per cent. sulphur as say with 2 or 2.5 per cent. Lobry de Bruyn and van Leent, strange to say, confirm Terry's statement, although the sulphur minima they observed are from three to four times as high as Terry's figure. Although we know with tolerable certainty that india-rubber is not a homogenous hydrocarbon, we may on the other hand safely assume that the molecular weights of the various hydrocarbons composing it are very little different. Indeed, the chemical behaviour of india-rubber suggests that they are merely isomeric. Still less doubtful is the fact that the vulcanisation of india-rubber, as I have shown in a previous paper, consists in the formation of sulphides or disulphides of definite composition. If, therefore, 0.6 per cent. of sulphur should be considered as the minimum of sulphur required for the complete vulcanisation of india-rubber, we are bound to accept its molecular weight as 5,300, which leads to the formula  $(\text{C}_{10}\text{H}_{16})_{95}$  or  $\text{C}_{950}\text{H}_{1520}$ . This formula will surely find its champion to defend it. Even with the assumption that the sulphur in the vulcanised india-rubber exists in the form of a double atom, as is indeed the case in those goods which have been vulcanised with chloride of sulphur, the above formula would be  $\text{C}_{475}\text{H}_{760}$ , which still is certainly by far too large. Applying the same calculation to de Bruyn and van Leent's figures, a molecular weight of  $\text{C}_{1100}\text{H}_{1760}$  appears as the most probable, and I take it as very significant that this is the same which I deduced for india-rubber from the investigation of its chlorosulphides.

\* Calculated upon the original weight of the sample extracted.

\* Calculated upon the original weight of the sample extracted.

It is certainly a mistake to assume that india-rubber will combine with one definite quantity of sulphur only to form vulcanised rubber. There is no doubt that the vulcanisation of india-rubber with sulphur will, on closer examination, be found to be analogous to the vulcanisation with chloride of sulphur, and that starting from a minimum, the sulphur of vulcanisation can be increased until all the available affinities of the india-rubber are saturated and products obtained which probably form the bulk of what is technically known as ebouite, a compound which certainly contains not less than about 20 per cent. of sulphur of vulcanisation. I have analysed a very great number of flexible vulcanised india-rubber goods and found them to contain from 1·7 to 8 per cent. of sulphur of vulcanisation, but all these samples containing less than 2 per cent. of combined sulphur, showed on fresh cuts unmistakable signs of tackiness which increased very much on heating them at temperatures not exceeding 100° C. I have not seen yet a thoroughly sound specimen of vulcanised india rubber containing less than 2·2 per cent. of sulphur. With an increasing percentage of sulphur of vulcanisation the tenacity of the articles increases very much. Goods containing above 5 per cent. of sulphur possess an enormous tensile strength which, however, appears to be developed at the expense of their elasticity.

In order to estimate the sulphur of vulcanisation, it is of course absolutely necessary first to remove every trace of free sulphur. I stated above that extraction with acetone answers very well for this purpose. I am well aware that the usual way to free vulcanised india-rubber from uncombined sulphur is to boil the sample repeatedly with solutions of the caustic alkalis. But I consider this method to be quite inadmissible as the alkaline sulphides which of course are formed in the first instance possess the property of reducing disulphides into monosulphides. This reaction may be responsible for the very low percentage of sulphur of vulcanisation observed by Terry. Of course, the possibility to prepare vulcanised india-rubber containing only 0·6 per cent. of combined sulphur cannot be denied, but I maintain that in such a product only part of the india-rubber can be vulcanised. But since it is an acknowledged fact that with an increasing percentage of combined sulphur the chemical and physical stability also increases, we must demand of a sound india-rubber article, that it should contain no unvulcanised india-rubber. This is only possible where there are at least 2 per cent. of combined sulphur. In many cases, as for articles which have to stand high pressure steam or high temperatures, a still larger proportion of sulphur will be necessary.

In every case the sulphur of vulcanisation should be estimated direct. For this purpose the solution of the india-rubber in nitrobenzene, is distilled under reduced pressure. The flask containing the non-volatile residue consisting of a treacly mass is then dried at 140° C. and subsequently oxidized with fuming nitric acid. The whole of the residue becomes eventually dissolved and the solution is then transferred to a platinum dish, the flask being rinsed with warm nitric acid. After evaporating the solution on the water bath, the residue is fused with carbonate of soda, the melt dissolved in water oxidized with bromine, acidulated, and the sulphuric acid precipitated with barium chloride. The estimation of the sulphur contained in the asphaltum, which is sometimes present, is performed in a similar manner in the solution of the asphaltum in cold nitrobenzene. The sulphur present in the form of inorganic compounds is estimated in the ultimate residue remaining behind after the solution of the india-rubber, by the well-known methods for the estimation of the sulphur in inorganic sulphides and sulphates.

The above discussed methods enable us to detect and estimate in india-rubber articles the following components:

- I. Fatty oils, mineral oils, resins, paraffin wax.
  - (a.) Free sulphur.
  - (b.) Sulphur in organic combination.
- II. India-rubber substitutes.
  - (a.) Sulphur in substitute.
- III. Asphaltum.
  - (a.) Sulphur in asphaltum.
- IV. India-rubber.
  - (a.) Sulphur of vulcanisation.
- V. Starch.
- VI. Mineral matters, free carbon (lampblack).
  - (a.) Sulphur in inorganic compounds.

In most cases such a detailed analysis is not called for, and the estimation of the acetone extract, rubber substitute, india-rubber and mineral matter, will answer for most purposes. The results obtained are remarkably accurate. Nevertheless, it will be found that even the most complete analysis in some cases does not give the desired information. This most often happens when the article to be analysed has undergone some deleterious change, the cause of which the analysis is expected to disclose. That excess of fatty oils, copper oxide and manganese oxide may cause such a change is known well enough, but often enough this "decomposition" takes place in the absence of those dangerous metallic oxides or oils. In such cases the analysis of these goods is apt to be disappointing, as it

#### I. EXTRACTION WITH ACETONE (10 RUNS).

Solution.	Residue.
Sulphinated oils from substitute.	II. EXTRACTION WITH ALCOHOLIC POTASH.
Fatty oils.	Solution.
Mineral oils.	India-rubber, substitutes.
Resin oils.	III. EXTRACTION WITH COLD NITROBENZENE.
Natural resins.	Solution.
India-rubber resins.	Asphaltum.
Resin of vulcanisation.	IV. EXTRACTION WITH BOILING NITROBENZENE.
Paraffin wax.	Solution.
Free sulphur.	India rubber
Combined sulphur.	Chlorine in india-rubber.
	Sulphur in substitute.
	Sulphur in asphaltum.
	Sulphur of vulcanisation.

#### V. EXTRACTION WITH BOILING WATER.

Solution.	Residue.
Starch (Farina).	Mineral matter, free carbon, fibrous materials.
	Sulphur in inorganic compounds.

enables us to form an opinion neither of the cause nor of the amount of the decomposition which has taken place. But I believe that this is due not so much to the imperfectness of our analytical methods, than rather to the incompleteness of our knowledge of the decomposition products of india-rubber under the influence of various agents. What is required is the analysis of a great number of decomposed articles, the original composition as well as the cause of the decomposition of which is known. Such analyses will show the effect of this decomposition upon the various components of india-rubber articles, they will also enable us to detect and isolate its products and thus we may hope to be able eventually to pronounce with certainty upon the cause of such a decomposition from the results of the analysis. At present this is, generally speaking, impossible. Anyone who doubts this has only to peruse the scientific evidence produced in the law courts in those rather frequent cases where india-rubber manufacturer and cloth manufacturer attempt to fix one upon the other the responsibility for the decay of water-proof cloth.

The course to be followed in the analysis of india-rubber goods can be indicated in the manner shown on preceding page.

#### DISCUSSION.

Mr. HEYS said he should like to correct Dr. Weber's statement in reference to Hutchinson's method. He stated that it was obtained from Mr. Bolas—that it was very slow and gave very inaccurate results in practice. This method was discovered by Mr. Hutchinson and communicated to Mr. Bolas in his office, but it gave very different results in their hands to those described by Dr. Weber.

Mr. BELL corroborated Mr. Heys' remarks.

Mr. TERRY said that looking at the paper as a whole there would have been a greater element of novelty in it if it had appeared before the recent papers of Henriques and of De Bruyn and van Leent. With regard to the method for the estimation of substitute, he must repeat a former observation, that although detailed methods might not before have been published, yet they had been worked out by those who required them. He had known, but had not used the method, ascribed to Bolas, but had preferred to work more on the lines laid down in the paper, and by which satisfactory results could be obtained. Some of the solvents employed by Dr. Weber were new to him in this application, and he hoped to be able to experiment with them on bodies of known composition, which as Dr. Weber had pointed out was the only reliable guide. He agreed with the observation that it was possible to attach too much importance to the physical tests unless such tests were supplemented by analysis; and he would also like to add that some practical experience with rubber on the part of the operator was very desirable also.

Dr. WEBER said with regard to the remark of Mr. Heys he would be glad to substitute the name of Mr. Hutchinson for that of Mr. Bolas. It was quite possible that his information with regard to his process for the analysis of india-rubber was not complete. Such information as he had was given in the paper, but he was able to dissolve a fraction of the india-rubber only. Mr. Bell confirmed Mr. Heys' statement as to the reliability of the method, but that confirmation would carry little weight, unless he could show that he tested the process by analysing samples of known composition. If they got an india-rubber article to analyse, they did not as a rule get at the same time a description of its component parts, and any method would give some result whether correct or not. He had not given a single analysis in this paper of an article the composition of which he did not exactly know. There was something more important than the mere analysis. The difficulty really began just when one required to interpret one's own results.

#### ON RESIST AND DISCHARGE STYLES WITH ANILINE BLACK.

BY CHARLES DREYER.

THERE are few colours that have rendered greater services to the calico printer and to the dyer than aniline black, the invention of which is due to John Lightfoot, of the Broad Oak Printworks at Acerrington, who patented his invention in the year 1863.

The depth of shade, the beauty of the colour, its great fastness to all reagents and to light, called immediately on its appearance the attention of chemists and colourists all over the world to its production.

It is not our intention to-night to give you a full history of the progress made in the application of this black for dyeing and printing; this has been done amongst others by the inventor himself in "The Chemical History and Progress of Aniline Black," published in 1871; by my esteemed friend Professor Noeltling in a work published in collaboration with Dr. Lehne entitled "Anilinschwarz und seine Anwendung in Färberei und Zeugdruck," published in 1892, a book we can highly recommend to all chemists and colourists who occupy themselves with the production of this colour, and of which it is to be regretted that no English translation has as yet appeared; and finally by Dr. A. Kilmeyer in a publication dated 1893, and entitled "Die Entwicklung des Anilinschwarz in der Druckerei und Färberei." The information contained in these publications is of such value that no calico printer and no dyer should be without them. It will be sufficient if we tell you that a high degree of perfection has been attained in the application of this colour in printing and in the dyeing of yarn, raw cotton, and piece goods. We believe it is no exaggeration to state that the weight of goods dyed and printed with this colour at present is equal if not superior to that made with any other colour.

Our object to-night is to call your attention to the progress made in the production of styles in printing with aniline black; at first it was used in the place of logwood blacks in most styles where such blacks were used; it was useful to print it with mordants which had to undergo dyeing with madder or its derivatives. Next the colourists aimed at producing effects by printing colours that would resist the black, namely, prevent the black from being formed at all points where such resist colours were printed, allowing the resist colours to appear in their own shades. It is believed that finer effects, clearer cut outlines of patterns are produced by the discharge method, consisting in printing or padding the cloth with the colour which after oxidation or steaming produces the black, but immediately after the printing or the padding of the colour for black a colour or colours are printed containing substances which prevent the formation of the black.

In connexion with aniline black, the expression of "discharge color" is not to be understood in the sense that the black is discharged after it has been formed, the nature of the colour prevents that. Take for example the case of indigo blue: such blue is discharged, and white is produced in the places where a color containing chromates of alkalis is printed and passed afterwards in a solution of sulphuric and oxalic acids.

In the case of aniline black, the discharge colour must be printed before the black has had time to oxidise, it should be considered as a resist-discharge, as we shall see later on in speaking of "Prudhomme's" method.

To understand the action of "reserves" and "discharges" on aniline black, it is necessary that a short sketch of the theory of the formation of the black be given.

Aniline black is formed by the elimination of hydrogen from aniline, this can be done by various oxidizing substances, such as chlorates, chromic acid, &c., in the presence of a trace of free acid, or in the case of the chlorates, in presence of a metallic salt, the chlorate of which is easily decomposed; a solution of pure chlorate of aniline may be boiled without any aniline black being formed, but immediately a drop of acid or of a copper salt is added to the solution the formation of black takes place at once. In the

same manner pure bichromate of aniline is fairly indecomposable into black, but if a drop of acid be added to a solution of bichromate of aniline the black is formed immediately; we may therefore conclude that black is only formed in the presence of a trace of free acid, and on this fact are based the various methods for resist printing with aniline black. We have, therefore, to look to alkaline substances or substances capable of neutralizing free acids, as capable to be employed as resists for aniline black, this is the opinion of most writers on the subject; we would add that besides these should be named also reducing substances or a mixture of the two; Witz recommends a mixture of 300 gr. acetate of soda and 300 grms. sodium hyposulphite per litre, or thickening as a good resist. To produce white on aniline black grounds, the following have been used:—

Caustic soda, carbonate of soda, silicate of soda, arsenite of soda, hyposulphite of sodium (100 grms. per litre), pyrogallie acid, recommended by Horace Koechlin; acetate of soda, chalk, zinc dust, albumen, aluminate of soda, citrate of soda, and sulphocyanides, recommended by Storek and Strobel (Wagners Jahresberichte, 1879), 50 gr. of sulphocyanide per litre are sufficient; by this process resists in all colors can be produced by simply adding either to an albumen color or to a tannic acid colour 50 to 60 grms. per litre of a sulphocyanide, and in the case of acid colours a suitable quantity of lead sulphocyanide.

#### *Discharge method of Prudhomme.*

We mentioned above the discharge method of Prudhomme, which for reasons given we preferred to call a "resist-discharge" method. This method has been used so successfully that we will give it in detail. The colour for padding used by him is made as follows:—

1,500 grms. chlorate of potash.

40 litres water.

4,000 grms. solution of ferrocyanide of potassium

28 per cent.

2,600 grms. aniline salt, and

X aniline oil.

The aniline salt (aniline chloride) should be as neutral as possible, and the quantity X of aniline oil to be added to the colour will depend on the composition of the aniline chloride.

Kertesz has published an improvement on Prudhomme's process; his colour is made as follows:—

{ 3,500 grms. chlorate of soda.

{ 20 litres water.

{ 5,500 grms. aniline oil.

{ 6,250 " hydrochloric acid at 19½ Baumé.

{ 10 litres water.

12 litres ferrocyanide of ammonium, bring the whole to 63 litres.

The ferrocyanide of ammonium for above is made as follows:—

A { 18 kilos. ferrocyanide of potash are dissolved in 32 litres water.

B { 9 kilos. ammonium sulphate are dissolved in 13 litres water.

Mix A and B in cooling, sulphate of potash crystallises out, and the clear solution is used.

The piece goods are padded with this solution and dried carefully. The temperature of the drying room should not go higher than 38 per cent., from this careful drying depends the success of the discharge operations.

The goods should be printed as soon as possible with the various discharge colours, of which we give a few:—

#### *Discharge White.*

3,000 grms. acetate of soda.

2,000 " soda ash.

4,000 " starch paste after cooling, add

250 c.cm. bisulphite of soda at 38° Baumé.

All pigment colours can be used as discharges by simply adding to them some soda ash, and aniline colours have also been used as discharges with suitable substances added to them.

After printing of the discharge colours the goods are steamed through the Mather and Platt steaming apparatus once or twice and passed through a hot bath of bichromate of potash (1—1,000), then washed, soaped, washed again, and dried.

A new departure in styles with aniline black has been made in the year 1892, and strange as it may seem it is again the Broad Oak Printworks of Accrington who have been the pioneers in this field of research. The patent taken in the names of Francis Frederick Grafton and William Browning is dated 18th June 1892, and states that:—

"The invention relates to an improved process or combination of processes for the production and fixation of colours, in conjunction with aniline black upon calicoes and other woven fabrics."

the claims are:—

"The simultaneous production and fixation of coloured designs in conjunction with aniline black grounds upon calico or other woven fabrics, by the combined process of combination of processes that follow":

"Firstly, prepare or mordant the cloth with an astringent solution and tartar emetic.

"Secondly, pad with a mixture of aniline oil and other materials thickened or otherwise, suitable for the production of aniline black by the ordinary known methods.

"Thirdly, print the design with colouring matters (such as are ordinarily fixed with astringent solutions) dissolved and thickened and mixed with acetate of soda or other well-known materials such as are used to prevent the formation or development of aniline black on the parts so printed.

"Fourthly, develop the aniline black (on the parts not printed) by steaming or ageing as usual which will at the same time cause the mordant or astringent matter to fix the colouring matters and resist or prevent the formation of the aniline black upon those parts which have been printed."

These are the claims set forth in that patent, they differ as you see from Prudhomme's method in so far that tannate of antimony is fixed on the cloth, previously to padding with the aniline black colour, such tannate of antimony acting as a mordant for basic aniline dyes and fixing them effectively after the steaming operation to which the goods are subjected afterwards.

We have seen goods produced by the above method which are excellent in design, in effectiveness and clearness of the finest lines in the pattern.

The next patents in order of date, which seem to be closely allied to the above, we find taken in America both on the same day, namely, the 14th February 1893, G. Donald of North Adams claims first, the padding of the cloth with an astringent or a metallic salt, if he pads with the one he adds the other to the aniline black colour with which he pads the cloth afterwards. He then prints the colour containing a substance or substances capable of resisting the fixing of the black (like Grafton) and a colouring matter combining with the tannin mordant previously fixed on the cloth, finally he steams to fix the colours and to develop the aniline black.

We leave you to judge where the improvement in Grafton's method can be seen in the above process.

F. P. Pearson of North Adams pads first in a colour capable of producing aniline black, then he prints the colour mixed with an astringent and a resist for the black, he steams or ages and passes finally through a solution of tartar emetic or tin salt to fix the basic aniline dyes.

We have not mentioned in its order of date the patent taken on the 12th January 1893 by the Thornliebank Company, Limited, Glasgow, and William Edward Kay, the two previous patents of Donald and Pearson having such obvious resemblance with that of Grafton's that we considered it better to give them in succession to the latter.

The claim of the Thornliebank Company and Kay are:—

1st. Preparation of the cloth with a mixture of aniline oil and other ingredients capable of developing aniline black, one of such ingredients being a ferrocyanide.

2nd. Printing of the design with a thickened paste containing (a) a solution of a suitable aniline or coal tar colour, and (b) acetate of soda, acetate of zinc, or other suitable reagent capable of preventing the development of aniline black.

3rd. Steaming or ageing the fabric in the manner usually followed for the development of aniline black.

The remarkable part in this invention is the observation made by the inventors that certain aniline dyes (we presume the basic dyes) are sufficiently fixed without the addition of any mordant when printed on the cloth prepared with an undeveloped aniline black mixture, one of the ingredients of which is a *ferrocyanide*; this observation should be followed out to its logical conclusion and experiments should be made to know if ferrocyanides do actually act as mordants for basic aniline dyes and fix them as effectually as tannic acid and emetic mordants.

Finally the inventors state that the order of the padding and the printing processes may be reversed.

On the 11th of April 1893, William Thomas Whitehead, of Magog, Canada, took out an English patent for improvements in resists or discharges for use in printing or dyeing cotton, &c. with aniline black; the invention consists in the use of oxides of zinc, &c. as a resist or resist discharge for aniline black, alone for producing whites, or mixed with pigment colours, aniline dyes, or dyewood extracts, for producing coloured patterns on aniline black grounds.

This inventor also made the curious observation that pigment colours and some aniline dyes, principally the basic dyes, are fixed by the ingredients contained in the aniline black colour in conjunction with the oxide of zinc, &c. without the addition of albumen to the pigment colours or of any other mordant in the case of the aniline dyes; this is, in our opinion, a valuable part of this invention and leads us to ask the question: What is the chemical or physical action that takes place to enable the oxide of zinc, and the other substances contained in the aniline black colour to fix pigment colours?

The fixing of the basic aniline dyes is already explained, partly at all events by the observations of Kay, and we shall take an early opportunity to try and ascertain the nature of the fixing action of the above substances on pigment colours.

We have been informed that Whitehead's process which is in use in many printworks in America has given satisfactory results.

Mr. John Riley of Thornliebank fearing that he could not be present this evening wrote to us as follows:—

"In May 1880 I introduced oxide of zinc as a resist for aniline blacks, and it has been in use at Thornliebank regularly since that date, for whites and pigment colours resists. The introduction of this insoluble resist enabled us to print the designs and steam one hour to fix the colours before padding the aniline mixture; it was used by us as a resist and not as a fixing agent."

It is possible that further patents exist on these interesting styles, but we have not had the leisure to search for them all.

We have endeavoured to lay before you, as fairly as possible, the methods lately discovered for producing valuable styles with aniline black, and so call specially the attention of those interested to the possibilities of further discovery and improvements in them, and I see present amongst you the leading chemists and colourists of the district, so that I can feel certain that the idea will be properly taken up and that the seed which I have sown will not fall into barren ground. This country has certainly made immense progress in the art of calico printing these last 20 years, and I hope it will continue to occupy the front rank in this difficult industry. I think papers bearing on some subject of the industry should be brought oftener before our section, and I hope we shall see more of them by members who are specialists in this branch brought before us next session.

## DISCUSSION.

Dr. Winkler said one of the mordants Dr. Dreyfus had mentioned was ferrocyanide, the mordanting properties of which he (Dr. Dreyfus) seemed somewhat doubtful about. He could, however, thoroughly bear out the property of ferrocyanide, or rather of hydroferrocyanic acid, to form lakes with the basic dyes, so that if we have either cotton prepared with ferrocyanide and treated then in a solution of a basic dye; or if the cotton is first impregnated with a solution of the basic dye and then treated with ferrocyanide in both cases the colour becomes fixed upon the cotton, the fastness of these lakes to light and washing is however very small, and cannot be increased by the forming of a compound lake as it could by treatment of a tannin lake with tartar emetic.

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### Hon. Local Secretary:

H. R. Procter, Yorkshire College, Leeds.

The names in italics are those of Members of Committee who retire at the end of the current session.

The following have been elected to fill the vacancies, and will take office in July next:—*Chairman:* C. F. Tetley. *Vice-Chairman:* J. J. Hummel. *Committee:* T. Fairley, W. Leach, A. G. Perkin, and Thorp Whitaker.

Meeting held at the Queen's Hotel, Leeds, on Monday, May 7th, 1894.

PROFESSOR SMITHELLS IN THE CHAIR.

## ON THE QUALITATIVE DETERMINATION OF TANNING MATERIALS.

BY H. R. PROCTER, F.L.C., F.C.S., LECTURER ON LEATHER INDUSTRIES AT THE YORKSHIRE COLLEGE, LEEDS.

THE following paper is an amplification of one which was read at the Chicago "World's Congress of Chemists" last year, and my excuse for bringing it before you to night is that it has not as yet appeared in any English paper, and that I would rather that it did so with some emendations and additions which further experience has suggested.

It can hardly be said that any systematic attempt has been made to formulate a scheme for distinguishing the various tanning materials by their qualitative reactions, except perhaps a very slight table published by the writer, although, in view of the constantly increasing number of new tanning materials, and especially of new extracts which are offered to tanners, the matter has become one of considerable practical importance. The commercial value of an extract is dependent, not only on the percentage of

tanning matter as determined by analysis, but on the nature of the tannin present, which influences the character of the leather produced.

It is, of course obvious, that, failing a clear knowledge of the tannins themselves any really scientific scheme of separation is for the present impossible; since, although we have good reason to believe that the tannins are a class, we are quite ignorant of their number, which may easily be a comparatively small one, since many members of the group which at present receive individual names are probably mere mixtures. All that can be done at present is to separate the tanning materials into groups or individuals by reactions which have been empirically determined; of which in many cases we do not know the chemical significance, and which are often due to other constituents of the material than the tannins themselves. The question is still further complicated by the fact that woods, barks, and fruit of the same tree often yield tannins of distinctly different character. Thus the bark of *Acacia arabica* [Babul] gives the general reactions of the Australian acacias, and evidently contains as its main constituent a tannin of the catechol group, while the pods of the same tree have a tannin which differs from any other which I have examined, but which is apparently a pyrogallol derivative. The differences between the tannins of oak bark, galls, and wood are equally striking. And while cutch from the wood of *Acacia catechu* gives the phloroglucol reaction intensely, the bark of the same tree hardly contains a trace.

Perhaps before proceeding to discuss means of identification, I may be permitted briefly to sketch what is known of the chemistry of the tannins, since, though it is as yet impossible to determine them on a really scientific basis, it is well to keep this steadily in view.

The tannins form a widely distributed class of vegetable products of varied constitution, but which is characterised by the power of precipitating gelatin and allied bodies from their solutions, of tanning animal skin, and of giving blackish colorations with ferric salts. So far as is known, all are derivatives either of the dihydric phenol, catechol, or of the trihydric, pyrogallol, while many of them contain in addition phloroglucol, which is isomeric with pyrocatechol, and is generally regarded as being also a trihydric phenol. The tannins frequently exist in some sort of connexion with glucose, but not as true glucosides, since by purification the glucose may be removed without apparent change in their properties.

The only tannin with the constitution of which we are even fairly acquainted is gallotannic acid, the ordinary tannin of the shops, prepared from galls, and, synthetically by Schiff's method of treating gallic acid with phosphorus oxytrichloride. Gallic acid is derived from pyrogallol by the introduction of a carboxyl group, as is salicylic acid from ordinary phenol; and gallotannic acid appears to be an anhydride of gallic acid, bearing a relation to it somewhat similar to that of the lactones to the oxy-fatty acids. From either gallic acid or its derived tannins, pyrogallol is obtained by the simple action of heat, which removes the CO<sub>2</sub> of the carboxyl group. By hydrolysis of gallotannic acid, gallic acid is restored.

There is another tannin, known as ellagettannic acid, which is evidently closely allied to gallotannic acid, and almost invariably exists in conjunction with it in tanning materials, and which, when hydrolysed, yields a pale crystalline solid, scarcely soluble even in boiling water, and which is apparently analogous to gallotannic acid, but less two atoms of hydrogen. This is ellagic acid, which is of considerable technical importance as constituting the pale deposit of "bloom" which occurs in leather tanned with these materials. The constitution of the tannin is unknown, but a rather striking colour-reaction with nitrous acid appears to be characteristic of all tanning materials which contain it.

Of the actual constitution of the large class of tannins which yield catechol on heating we know very little. Instead of hydrolysing when heated with water or dilute acids, they appear mostly to yield series of red anhydrides, the lower members of which are dark coloured and

difficultly soluble tannins, and the higher, still of an acid nature, and quite insoluble in cold water, but soluble in alkaline solutions, are "phlobaphenes," or "reds," possibly allied to the resins. It is only after pretty severe treatment of these with caustic alkalis, that they split up into protocatechuic acid, which is related to catechol as gallic acid is to pyrogallol, together with phloroglucol and other bodies.

It would obviously be most scientific and satisfactory to classify the tannins by these decomposition products, but unfortunately the latter are so unstable, and so difficult to separate, that this is impracticable, and we have to fall back on reactions which do not demand actual isolation of the constituents. One of the oldest of these is the colour reaction with ferric salts, with which catechol and protocatechuic acid give dark green colorations, while pyrogallol and gallic acid give blue-blacks, and these peculiarities usually extend also to their derivatives. As a reagent for this purpose I employ—

**Ferric Alum, Ammonia Ferric Sulphate.**—A one per cent. solution of this salt has been chosen as easily obtainable, neutral, and in a state of approximate purity. Ferric chloride, which is generally used, is almost invariably strongly acid, and of a very persistent yellow. Acid ferric chloride in excess will give a green-black reaction with gallotannic acid itself, and its results as a test are often misleading. Ferric acetate, which has also been largely used, gives heavy precipitates with most materials, and is very strongly coloured, so that it was found less convenient than the iron alum. As the colorations are generally very powerful, they are best seen by diluting the tanning infusion freely, and adding the iron solution very cautiously. In many cases the coloration first produced, and which is the characteristic one, rapidly fades to a dull olive or brown in presence of excess of iron, probably by oxidation and destruction of the tannin.

It may be pretty safely assumed that any tannin which gives a distinctly greenish black with iron is a catechol derivative, but there are a large number of materials, especially among the acacias or mimosas, which give purplish blacks, but are pretty certainly catechol tannins; and on the other hand the oak barks which Trimble has proved to be catechol tannins, and most of which give green blacks with iron, also yield bloom or ellagic acid, and are therefore also allied to gallic acid. Another reagent is therefore employed in the preliminary classification, viz., Br. water.

**Bromine Water Reaction.**—It is best to add the bromine water drop by drop to 2–3 cc. of the infusion in a test tube, until the solution smells strongly. In some cases the precipitate is slight, or forms slowly, and occasionally it is crystalline and on this account less conspicuous, but it is usually a distinct yellow or brown flocculent one. In general terms it may be said to be a reagent for the catechol tannins; precipitating all which give green blacks with iron, and many which give blue or violet-blacks, which are reasonably suspected of containing catechol. It does not precipitate any recognised pyrogallol tannin, but several which yield ellagic acid (bloom) such as oak barks.

Tannins may therefore be divided into three classes, as in table I, viz.:—(1) those which give a precipitate with Br. water, and green blacks with iron alum, and which are pretty certainly catechol tannins; (2) those which give a precipitate with Br. water, but bluish or violet blacks with iron, and which for the present must be regarded as mixed or uncertain; and (3) those which give no precipitate with Br. and blue-blacks with iron, and which, so far as we know are all derivatives of pyrogallol.

By far the largest number of tannins fall into class 1, but these may be pretty satisfactorily subdivided by a reaction with—

**Cupric Sulphate and Ammonia.**—When solution of cupric sulphate is added to a tanning material infusion, it sometimes gives a precipitate, and sometimes not. This is a useful indication in some cases, but must not be regarded as of first rate importance, as the presence of any salt of a weak organic acid would in all cases cause a precipitate, by neu-



tralisising the sulphuric acid of the sulphate. Cupric acetate produces a precipitate with all tannins so far as I have observed, and it is interesting to note that in this case, as in many others of a like character, the tannin combines with, and precipitates the salt as a whole. On the addition of ammonium carbonate, effervescence takes place, and the precipitate darkens, and becomes a true tannate.

When ammonia is added to the mixture of a tannin and cupric sulphate, a precipitate is invariably formed in the first instance, consisting of a tannate of copper with variable quantities of blue cupric hydroxide. With excess of ammonia, the whole of this precipitate is in many cases dissolved, while in others, an insoluble tannate remains. The latter is the case with all tannins derived from gallo-tannic acid, and with many containing protocatechuic acid, and forms a useful means of classification, though as yet the difference of structure to which it corresponds is unknown. The fact that hemlock and other pine barks, as well as cutch and gambier, yield cupric tannates soluble in ammonia, is an instructive commentary on processes which have been proposed for quantitative estimation by precipitations by ammonio-cupric solutions.

Where the precipitate redissolves, it generally does so with the production of either a greenish brown, or a purplish brown coloration, which in presence of a small amount of the blue ammonio-cupric sulphate, appears either as a green or a reddish violet. It need hardly be pointed out that to see this coloration well, the copper should be in the least possible excess. Hence it is desirable to use a very weak copper solution, say at most one per cent.

We thus obtain two sub-classes: 1  $\alpha$  (Table II.) in which copper produces a precipitate soluble in ammonia and 1  $\beta$  (Table III.) in which an insoluble precipitate is formed.

No attempt has been made to arrange systematically the results of the further reactions which are suggested to determine individual members, since in the main they correspond to no broad chemical demarcation, and in others they shade off by imperceptible degrees, so that it is only by taking all characteristics into account that any safe conclusion can be drawn. This is specially the case with the vanillin reaction for phloroglucol, which is given strongly by cutch and gambier, markedly by several other materials, and so faintly by many others that no sharp line can be drawn. The following reactions are those which I have employed for further distinction:—

**Nitrous Acid Reaction.**—This reaction, which is believed to be a new one, is obtained by adding to a few cc. of the very dilute infusion in a porcelain basin a distinct excess of freshly prepared solution or a few crystals of sodium or potassium nitrite, and then 3–5 drops of decinormal sulphuric or hydrochloric acid. In typical cases the solution instantly turns pink or crimson, and slowly changes through purple to a deep indigo-blue, but in others, as sumach, where the reaction is feeble, and masked by other changes, the final colour is green or even brownish. In a large number of cases, nitrous acid produces a yellow or brown coloration or precipitate, but "reaction" in the tables invariably means a series of colour-changes as above-described. Nothing can as yet be said on the nature of the chemical changes produced. The reaction is given by all tanning materials which yield ellagic acid or "bloom," but not by ellagic acid itself, nor by pure gallo-tannic acid. It is therefore probably a reaction of ellagitannic acid, and is valuable for subdividing the mixed and pyrogallol tannins. It may also be obtained faintly from some of the oaks in Class 1  $\beta$ .

**Stannous Chloride and Hydrochloric Acid.**—This reagent consists in a strong solution of stannous chloride in concentrated hydrochloric acid. If about 10 cc. of this are added to 1 cc. of the tanning material infusion, in a porcelain basin, and allowed to stand for 10 minutes, coniferous tans, mimosas, and some others give a very marked pink coloration. This is specially distinct in the case of larch bark. If a small piece of larch-tanned leather be steeped in the reagent, the coloration appears very strongly.

**Deal Shaving and Hydrochloric Acid.**—A shaving or slip of any pine wood is moistened with the infusion, and then, either before or after drying, is again moistened with concentrated hydrochloric acid. In the case of cutch and gambier, and a few other materials, and of solution of phloroglucol itself, the spot becomes at once a bright red or violet, but in many cases the reaction is faint, and only appears after some hours. It probably always indicates the presence of phloroglucol. The statement that catechol gives a similar reaction appears to be an error.

**Sodium Sulphide.**—This was mentioned in my previous manuscript on the subject, but the printer unfortunately substituted sodium sulphide, which does not answer in the laboratory!

A few drops of the solution are placed in contact with a crystal of sulphite on a tile or in a basin. With valonia, a bright purplish pink is rapidly developed, apparently by oxidation. Many other tanning materials produce red or pink colorations, but in no case so marked a reaction as valonia.

**Concentrated Sulphuric Acid.**—A test tube is rinsed out with the infusion and drained so that only about a drop remains, and about 1 cc. of concentrated acid is cautiously poured into the sloping tube, so as to form a layer underneath the tanning solution. The ring of colour at the junction of the two liquids is noted, and then they are mixed by shaking and diluted with water. About half the materials give a deep purplish crimson, called simply "crimson" in the tables, and in many cases this is decidedly pink on dilution, while in others it is obscured by brown products formed by the heat of mixture. A large number of other materials give browns or yellows only, which, when intense, may appear red, but dilute to yellows or yellow-browns.

**Lime Water** is a most useful reagent, being very varied in the colour of the precipitates it produces, and the changes they undergo by oxidation. The reaction is best seen in a shallow porcelain basin, and time must be allowed for the changes to take place. It may be safely added in considerable excess.

**The infusions of Tanning Materials employed** are understood to be of about the strength used by the writer in determinations by hide-powder, viz., about 0.6 gm. of dry soluble matter in 100 cc., but moderate divergencies from this do not influence results. Of course, where reactions are feeble, with very dilute solutions they may be imperceptible, and on the other hand, strong solutions will sometimes give precipitates where only colorations are noted with weak ones. As some of these reactions are dependent, more or less, on the concentration of the infusion employed it is desirable to experiment in the first instance with known materials in order to acquire a little experience of exact conditions.

In some cases these reactions will serve to determine the constituents of mixtures, but in others this will continue impossible till further and more distinctive reactions are discovered.

They can of course be applied to the recognition of tannages, when excess is present in leather, which can be dissolved out with water; but the effect of dilute alkalis and other solvents on the reactions is worth further experiment.

It was thought that some information as to the meaning of the reactions employed might be obtained by examining the behaviour of the simpler derivatives under the same circumstances, and the results are given in Table X. It will be at once obvious that the blue-black reaction with iron is characteristic of the pyrogallol group, and the green-black equally so of the catechol derivatives, while phloroglucol, contrary to the statements of the text-books, gives no marked reaction with iron, but is the only member of the group characterised by the very delicate reaction with the vanillin present in deal. On the remaining reactions little light is thrown.

TABLE I.  
PRELIMINARY CLASSIFICATION OF TANNING MATERIALS.

Fe. Water produces a Precipitate. Iron-Alum gives Greenish Blacks. (Catechol Tannins.)		Br. Water produces a Precipitate. Iron-Alum gives Blue or Purplish Blacks. (Mixed and Doubtful.)		Br. Water produces no Precipitate. Iron-Alum gives Blue Blacks. (Pyrogallol Tannins.)	
Copper Sulphate followed by Ammonia in Excess.		Sodium Nitrite and 5 drops $\frac{N}{10}$ HCl.		Sodium Nitrite and 5 drops $\frac{N}{10}$ HCl.	
Precipitate redissolves.	Precipitate does not redissolve.	No reaction, or, at most, darkening.	Some Colour-Change through Red towards Blue or Green.	Colour-Change through Red to Blue.	No reaction.
1 $\alpha$	1 $\beta$	2 $\alpha$	2 $\beta$	3 $\alpha$	3 $\beta$
Table II.	Table III.	Table IV.	Table V.	Table VI.	Table VII.

TABLE II.

Class 1a.	Ferric Alum.	Bromine Water.	Nitrous Acid.	$\text{CuSO}_4$ + $\text{NH}_4\text{OH}$	$\text{SnCl}_2$ + HCl	Deal Shaving and HCl.	$\text{Na}_2\text{SO}_3$	$\text{H}_2\text{SO}_4$	Lime Water.
Cutches from <i>A. catechu</i> wood.	Green-black.	Pp.	No react. darkens.	Pp. redissolves generally red-violet coloration.	No react.	Deep violet-red.	Reddens somewhat.	Red-brown coloration.	Reddish pp. slowly formed.
"Tham leaf" extract (a cutch substitute).	Olive-black pp.	"	"	Pp. redissolves brownish coloration.	"	No react.	No react.	Crimson, dilutes pink.	No pp.
"Turwar" bark ( <i>Cassia auriculata</i> L.).	Green-black.	"	"	Pp. redissolves red-violet.	"	Trace	Pink coloration.	Crimson	Reddish pp.
"Gambene" extract (a gambier substitute).	Green-black coloration.	"	"	"	"	No react.	Slight pink coloration.	Crimson, dilutes pink.	"
"Tengah" bark ( <i>Cecropia Candelaria</i> L.).	"	"	No react. darkens pp.	"	Pink coloration.	"	Pink coloration.	Crimson	Bright red pp.
Bark ( <i>Acacia leucophlea</i> )	"	"	No react.	"	"	Slow violet react.	"	Crimson, dilutes pink.	Dull brown pp.
Bark ( <i>Syzygium febrifugum</i> ).	"	"	"	Pp. redissolves red-brown.	"	No react.	"	Crimson	Red-brown
Oak bark ( <i>Quercus suber</i> ).	"	"	Reacts somewhat.	Pp. redissolves brown.	No react.	"	Reddens	Crimson, dilutes pink.	Reddish brown pp.
Green oak (Ital.) ( <i>Quercus ilex</i> ).	"	"	Reacts faintly, if at all.	"	"	"	"	"	"
Garonille (root bark of Kermes oak ( <i>Quercus Coccifera</i> )).	"	"	Reacts?	"	"	"	"	"	"
§ Quercitron bark ( <i>Quercus Trectoria</i> ).	"	"	Reacts somewhat.	"	Light green	"	Doubtful	"	"
Gambier (ext. of leaves of <i>Naucleria glabra</i> ).	Deep green coloration.	"	No react. darkens.	Pp. redissolves olive-green.	Yellow	Deep violet-red.	Yellow	Crimson, dilutes brown.	No pp.
* "Prun bast." (leaves of <i>Coppon</i> or <i>Ostrya compressa</i> ).	Green-black.	"	No react.	Pp. redissolves green.	No react.	Pink	"	"	Light yellow pp.
* "Koko," Natal (leaves of <i>Cladrastis bursifolia</i> ).	"	"	"	"	"	No react.	"	Dark brown.	Bright yellow pp.
Larch bark ( <i>Larix Europaea</i> ).	Green-black coloration.	"	No react. darkens.	Pp. redissolves olive-green.	Pink coloration.	"	No react. darkens.	Deep red-brown.	Rusty pp.
Hemlock bark ( <i>Tsuga</i> or <i>Abies Canadensis</i> ).	Olive-green reddish pp.	"	No react. pink with $\text{NaNO}_2$	Pp. redissolves neutral tint.	"	"	Reddens	Crimson, dilutes pinkish.	Red-brown pp.
"Larch" extract from ( <i>Abies excelsa</i> ).†	Green-black or brown.	"	No react.	Pp. redissolves green.	"	"	Darkens	Deep red-brown.	Brown pp.

\* Used at Cape of Good Hope as sumach.

† Used in Natal as sumach substitute.

‡ *Fichte, Rothbume*, Norway or common spruce. *Abies pectinata* the *Weiss* or *Edel-Tanne* or silver fir is said to give a blue-black with iron.

§ Dyes yellow with Al and Sn mordants.

TABLE III

Class 13.	Ferrie Alum.	Bromine Water.	Nitrous Acid.	CuSO <sub>4</sub> + NH <sub>4</sub> OH	SnCl <sub>2</sub> + HCl	Deal Shaving and HCl	Na <sub>2</sub> SO <sub>4</sub>	H <sub>2</sub> SO <sub>4</sub>	Lime Water.
Willow bark Russian, Sp. unknown.	Green-black.	Pp.	No react.	Dense pp.	No react.	Very faint.	Pink coloration.	Reddish-brown.	Slight yellow pp.
<i>Agave Americana</i> - <i>Piper-lanceolatum</i> bark extract.	"	"	"	Dense chocolate pp.	"	"	Redders somewhat, dilutes pink.	Crimson dilutes pink.	Reddish-brown pp.
<i>Acacia catechu</i> bark ....	"	"	"	Dense violet-black pp.	"	Trace.	Pink color.	Red-brown.	Pinkish color pp.
" Thorny tree " bark ( <i>Acacia horrida</i> ) (Cape).	"	"	No react, darkens.	Dense pp.	No react.	Doubtful.	"	Duller no so. not robust.	No pp.
Mangrove bark extract ( <i>Rhizophora mangle</i> ).	"	"	No react.	Reddish-black.	Slight reddening.	No react.	Slight reddening.	Red-brown.	Red pp. darker red in excess.
Quebracho wood extract ( <i>Quebracho</i> - <i>L. xylocarpum</i> - <i>L. pyramidalis</i> ).	Green-black coloration.	"	"	Dense pp.	Pink color pp.	Trace.	Doubtful.	Crimson color dilutes pink.	Light brown pp.
" Sugar bush " bark (Cape) ( <i>Protea mellifera</i> ).	Green-black.	"	No react, darkens.	Dense pp.	No react.	"	"	Red.	Yellowish-brown pp.
" Waagboom " (Cape) ( <i>Protea magnifica</i> ).	"	"	"	"	"	"	Pink color.	Crimson dilutes pink.	Slight yellow pp.
" Kruppboom " (Cape) ( <i>Leucospermum cordatum</i> ).	"	"	"	"	"	Violet distinct.	"	"	Slight greyish pp.
Silver tree " (Cape) ( <i>Leucodendron argentea</i> ).	"	"	"	"	"	No react.	Pink coloration.	"	Light color pp.
* Chestnut Oak ( <i>Quercus Castanea</i> ).	Olive-green coloration.	"	Reacts distinctly.	Decided pp. Insoluble in excess.	"	"	Reddens.	Crimson dilutes pinkish.	Reddish-brown pp.

\* Infusions fluoresce, especially with ammonia.

TABLE IV

Class 2a.	Ferrie Alum.	Bromine Water.	Nitrous Acid.	CuSO <sub>4</sub> + NH <sub>4</sub> OH	SnCl <sub>2</sub> + HCl	Deal Shaving and HCl	Na <sub>2</sub> SO <sub>4</sub>	H <sub>2</sub> SO <sub>4</sub>	Lime Water.
" Shen's " Cypress - <i>Schinus molle</i> - possibly <i>Cortaria myrtifolia</i> .	Blue-black pp.	Pp.	No react.	Dark pp.	No react.	No react.	Yellow.	Yellow-brown.	Yellow pp. darkening.
Kliphaant Bark* ( <i>Rhus Thunbergii</i> ).	Blue-black	"	"	Dense dark pp.	"	"	Pink.	Dull crimson dilutes orange.	Pinkish pp.
Canaigre Root of <i>Rhus hydnosolens</i> .	Blue-black pp.	"	"	Dense dark pp.	No react. clouds.	Trace violet.	Slight darkening.	Yellow-brown.	Pink coloration greyish pp.
" Taiwan " or " Elands-bontjes " (Red Elephant) ( <i>Persea Burchellii</i> ).	"	"	No react. Darkens.	"	No react.	"	Pink.	Red.	Reddish-brown pp.
Mimosa or Wattle barks (Various Austral. <i>Acacia</i> ).	Dirty violet pp.	"	No react.	Dense purple-brown pp.	Slight reddening.	Sometimes trace.	Reddens.	Crimson dilutes pink.	Reddish-brown pp.
Babool bark, India ( <i>Acacia Arabica</i> ).	"	"	"	Dense dark pp.	Slight trace.	Faint trace.	Slight darkening.	Crimson dilutes orange.	Dark reddish-brown pp.
Dark red Austr. bark (Probably an <i>Acacia</i> ).	"	Pp. needle crystals.	"	Deep violet pp.	No react.	"	Orange-pink.	Crimson dilutes pink.	Bright violet pp.
* White bark " <i>Albizia blanda</i> - South America ( <i>Albizia leucacantha</i> ).	"	Pp.	"	Reddish-black pp.	"	Violet.	Redders slightly.	"	Red pp. turning violet.

\* Used at Cape of Good Hope.

TABLE V.

Class 2β.	Ferric Alum.	Bromine Water.	Nitrous Acid.	CuSO <sub>4</sub> and Ammonia.	SnCl <sub>2</sub> + HCl	Deal Shaving and HCl	Na <sub>2</sub> SO <sub>3</sub>	H <sub>2</sub> SO <sub>4</sub>	Lime Water.
English Oak ( <i>Quercus Robur</i> .)	Blue-black (green with excess).	Pp.	Reacts somewhat.	Slight pp. Dark brown pp.	No react.	Faint react.	Reddens	Crimson dilutes pink.	Reddish-brown pp.
Jaft or Dehit.* Supposed oak product.†	Blue-black pp.	"	Reacts red-blue.	Brown pp. Dark brown pp.	No react. Dark brown pp.	"	Some darkening.	"	"

\* A Persian product, dark scales very rich in tannin (about 40 per cent.).

† Strong infusions, dry whitish and iridescent.

TABLE VI.

Class 3α.	Ferric Alum.	Bromine Water.	Nitrous Acid.	CuSO <sub>4</sub> + NH <sub>4</sub> OH	SnCl <sub>2</sub> + HCl	Deal Shaving and HCl	Na <sub>2</sub> SO <sub>3</sub>	H <sub>2</sub> SO <sub>4</sub>	Lime Water.
Aleppo galls (of <i>Quercus infectoria</i> .)	Blue-black pp.	No pp. slight scum.	Reacts red to blue.	Dark pp. insoluble.	Light yellow pp.	No react.	No react.	Greenish to dirty yellow.	Pale pp. turning bluish-green.
*Sumach (leaf of <i>Rhus coccinea</i> .)	"	No pp.	Reacts feebly.	Dark brown insoluble pp.	No react.	"	"	Yellow	Yellow pp. turning bright green.
*Myrobolans ( <i>Terminalia chebula</i> .)	"	"	Reacts red to blue.	Dark insoluble pp.	"	"	Yellow	"	Yellow pp. turning greenish.
Pomegranate rind ( <i>Punica granatum</i> .)	"	"	"	Dark brown insoluble pp.	"	"	No react.	Orange-brown.	Bright yellow pp. turning red with excess.
Algarobilla (pod of <i>Cassipouira hircifolia</i> .)	"	"	"	Dense dark pp.	"	"	Deep yellow.	Deep yellow-brown.	Bright yellow pp. darkens somewhat.
+ Divi-divi (pod of <i>Cassipouira coriaria</i> .)	"	"	"	"	"	"	No react.	Crimson	Yellow pp. turning red-purple.
Algaroba (probably pod of <i>Prosopis dulcis</i> .)	"	"	Reacts red to olive.	"	"	"	Yellow	Yellow to olive.	Yellow pp. turning black.
Valonia (cup of <i>Quercus Agilops</i> .)	"	"	Reacts red to blue.	Dark red-dish pp.	"	"	Purplish pink.	Deep yellow.	Yellow pp. turning red-purple.
**Oakwood" extract (oak or chestnut wood).	"	"	"	Purplish-brown pp.	"	"	Reddens	Yellow brown.	"

\* Dyes yellow on Sn mordants.

+ Moderately strong potassium nitrite solution precipitates divi, but not *Algaroba* solutions; pp. soluble in hot, or much cold water.

\*\* *Crude* chestnut wood extract may be distinguished from oakwood by its violet reaction with ammonium sulphide (see "Gerber," No. 261, p. 157).

TABLE VII.

Class 3β.	Ferric Alum.	Bromine Water.	Nitrous Acid.	CuSO <sub>4</sub> and Ammonia.	SnCl <sub>2</sub> + HCl	Deal Shaving and HCl	Na <sub>2</sub> SO <sub>3</sub>	H <sub>2</sub> SO <sub>4</sub>	Lime Water.
Pure gallo-tannic acid ...	Blue-black pp.	No pp.	No react.	Dark pp.	No react.	No react.	No react.	Yellow	Pale pp. turning blue.
Babool pods ( <i>Acacia Arabica</i> .)	Blue-black	"	No react. darkens.	Dark green colour.	"	Faint violet.	"	Reddish-violet.	Pink colour. No pp.

TABLE VIII

—	Ferric Alum.	Bromine Water.	Nitrous Acid.	CuSO <sub>4</sub> and Ammon.	SnCl <sub>2</sub> + HCl	Dec. Stannous and HCl	NH <sub>4</sub> SCN	H <sub>2</sub> SO <sub>4</sub>	Lead Water.
Catechol.....	Dark green coloration.	No pp.	Turns yellow.	Green colour.	No react.	No react.	No react.	No pp.	No pp.
Protocatechuic acid.....	"	"	Turns brown.	No pp.	"	"	No react.	No pp.	"
Phloroglucol.....	No react.	Bulky white pp.	Turns olive green.	"	Reddish colour.	"	Slight precipitate.	"	"
Pyrocatechol.....	Blue-black turning green and brown.	No pp.	Turns yellow.	Brown colour.	"	No react.	Brown precipitate.	"	Amorphous coloration rapidly turning brown.
Gallie acid.....	Blue-black coloration.	"	Turns brown.	"	"	"	No react.	"	White pp. rapidly turning blue.

TABLE IX

REACTIONS OF PROFESSOR TRIMBLE'S PURIFIED OAK BARK TANNINS.

(A MONOGRAPH OF THE TANNINS, VOL. II., P. 88.)

	Ferric Alum.	Bromine Water.	Nitrous Acid.	CuSO <sub>4</sub> and Ammonia.	SnCl <sub>2</sub> + HCl	Dec. Stannous and HCl	NH <sub>4</sub> SCN	Lead Water.
Black oak ( <i>Q. tinctoria</i> )	Green colour and pp.	Yellow pp.	Brownish-yellow pp.	Pp. Green colour.	Yellow with some pink.	Violet colour.	Yellow colour.	Pp. turning pink then red.
Pink oak ( <i>Q. palustris</i> )..	"	"	Pinkish colour changing to brown pp.	Pp. Brownish-green colour.	Pink colour.	"	Pink.	"
Scarlet oak ( <i>Q. coccinea</i> )	Bluish-green colour. Green pp.	"	Brown pp.	Pp. Green colour.	Pinkish colour.	"	Pinkish colour.	Pp. turning reddish.
Spanish oak ( <i>Q. falcata</i> )	Green colour and pp.	"	"	Pp. Red-brown pp.	Yellow colour some pink.	"	Yellow with pink streaks.	"
White oak ( <i>Q. alba</i> )....	"	"	"	Pp. Brown-green colour.	Pinkish colour.	"	Pinkish colour.	Pp. turning pink.
Willow oak ( <i>Q. phellos</i> )..	"	"	"	Pp. Red-brown pp.	Very yellow colour.	"	Yellow with pink streaks.	Pp. turning green, liquid reddish.
Chestnut oak ( <i>Q. prinus</i> )	"	"	"	No pp. Greenish-brown colour.	"	"	"	Pp. turning pink.
Swamp white oak ( <i>Q. bicolor</i> )	"	"	"	"	"	"	"	"
English oak ( <i>Q. robur</i> )..	Bluish-green colour. Green pp.	"	Pink colour changing to brown pp.	Pp. Red-brown pp.	Dotted pink colour.	Violet colour.	Pink.	"
Indian oak ( <i>Q. sepiariaefolia</i> )	Green colour and pp.	"	Brownish-yellow pp.	"	Pink colour.	"	Yellow with pink streaks.	"

## NOTE ON THE ESTIMATION OF TANNING MATTER.

BY H. R. PROCTER, F.I.C.

THERE is much for which the leather trade should thank the Imperial Research Station for Leather Industries at Vienna, and not the least of these benefits is the invention of the indirect gravimetric method of tannin estimation published by Simand and Weiss (Dingl. Polyt. J. 260, 564; this Journal, 6, 94) in 1887, and now used in various modifications by leather chemists practically to the exclusion of every other, the Löwenthal method only retaining a certain place for special purposes. In its original form the process consisted in evaporating a given quantity (50 or 100 cc.) of the tannin infusion to determine total solids, while to another portion, carefully purified powdered hide is added in five or six successive small quantities at intervals of six or eight hours, so as to avoid the hardening effect which strong tannin solutions exert on hide if added all at once; and the filtrate from this is again evaporated to determine the solids remaining, which represent the "non-tannins," the difference between the two determinations giving the actual weight of the tanning substances absorbed. The principal drawback is the rather considerable time required for complete absorption (24–36 hours) and the consequent liability to putrefactive action in hot weather. This is reduced by the device used by Dr. Hellon, who sets the absorption flasks in a vessel of running water.

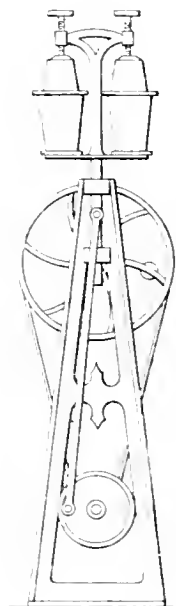
Almost immediately after the publication of the method, the present writer (this Journal, *loc. cit.*) suggested the use of a filter-tube charged with the hide-powder as a means of detanning. This was at once adopted by the Vienna Research Station with some improvement in the form of the filter, and is still used there with quite satisfactory results, reducing the required time to 2 or 3 hours.

A further modification was recently published in the "Leather Manufacturer" by Dr. Fiebing, of Milwaukee, Wis., with which I was much struck when I saw it in operation in his laboratory, and since that time have found it so satisfactory at the Yorkshire College Leather Industries Laboratory that we there use it for almost all tannin determinations. The principle of the new method is identical with that of Simand's original process, but instead of merely digesting with occasional shaking, the liquid is violently agitated for 10 minutes each time with three successive quantities of hide-powder, which has previously been thoroughly washed in at least two changes of water, and well squeezed. The shaking is best done with a "milk-shaker," a little machine constructed for the preparation of American drinks, and consisting of a vertical rod carrying clamps for two glasses with covers, which receive a rapid reciprocating movement (not less than 320 to 420 rotations of the crank per min.) from a crank driven by a multiplying wheel. In place of glasses I frequently use ordinary bottles of suitable size, with india rubber corks; and, as in my laboratory we have the convenience of an electric motor, I usually run rather over the time named for extra safety, although with 6 grms. of hide-powder in all to 100 cc. of infusion not containing more than 0.6 gram. of total solids, 30 minutes is sufficient to remove all tannin.

It is obvious that some correction must be applied to the results for the water introduced in the damp hide-powder. This is determined by pressing the latter in folds of cloth in a copying press, and re-weighing to ascertain gain in weight, each gram. of which is reckoned as an addition of 1 cc. to the volume of the solution employed. Even if this is not strictly accurate, any small error has only a vanishing influence on the result.

In addition to the saving of time, the process has the advantage that it gets rid of the rather dubious correction for solubility of the hide-powder sometimes used in Simand's original method, and hence will give good results with powders which are useless either for this or the filter method. Thus, an experimental powder was prepared

from the sawdust of buffalo-hide pickers (a manufacturing waste worth about 5d. per lb.) by washing first with dilute sulphurous acid, then with abundance of tap-water, and finally with distilled. This was dried by spreading on paper in a warm room, and ground through the Burroughs and Wellcome's drug mill, which we use for samples of tanning materials. It formed a somewhat greyish-white



powder, much heavier and less bulky than the Vienna powder, and proved quite useless in the filter from its compactness, and the difficulty of wetting it, probably due to traces of fatty acids. Used in the "shaker," the results agreed most satisfactorily with the Vienna powder, and we now rarely use anything else, as the saving in cost is very considerable. Vienna powder costing here about 10s. 6d. per lb.

The following experiments made by Mr. E. Dewez, one of my students, to determine whether the speed of the motor had any influence on the amount of matter dissolved from the powder, show also very clearly to how small an extent it can influence the results of an analysis. 6 grms. of the powder were shaken for half an hour at the highest speed of the motor with 100 cc. of water, when 50 cc. of the washings filtered through paper and evaporated left 18 mgrs. of solids, of which 9 mgrs. was inorganic. It was then washed a second time for 10 minutes, when 50 cc. gave only 4 mgrs. residue. A second experiment at the slowest speed gave for the first washing 14 and for the second 2 mgrs. residue. The washed powder of the first experiment, which must have given up much less than 4 mgrs. to a third washing, was now used to detannise 100 cc. of an extract liquor as before described, and gave a result of 7.91 per cent. of non-tannins in the sample of extract, while a parallel determination with Vienna powder and the siphon filter gave 7.81 per cent.

In my opinion the only satisfactory way of dealing with the soluble matter in hide-powder is to eliminate it to such an extent that its influence may safely be disregarded, any attempt to determine its quantity and apply a correction being rendered uncertain by the fact that it consists to a large extent of partially altered hide, which is precipitated by tannin, and consequently is less soluble in tanning infusions than in pure water. With a good powder in the hide-powder filter it is practically eliminated by rejecting the first portion (of say 30 cc.) which comes over, but such a powder should be carefully tested before use to ascertain that after running 50 cc. the residue left by the next 50 is negligible. To give an idea of the limit allowable, I may

point out that on a 20 per cent. material each milligramme will be approximately equal to 0.1 per cent.

In an important paper, *Ueber Eine wichtige Fehlerquelle der gewichtsanalytischen Methode der Gerbstoffbestimmung* (Ding. Polyt. Jour. 280, Heft 6 and 7), Dr. R. Koch points out the very varying quality of commercial hide-powders, and mentions one, of which 7 grms. gave no less than 0.258 gm. of soluble matter! He further draws attention to the fact that in his hands, powders which had undergone some degree of incipient putrefaction, and which consequently gave much soluble matter, gave different, and indeed higher results than normal powders to the extent of sometimes 2 per cent., even after thorough washing. This variable absorptive power has also been confirmed by Dr. Fiebig, and especially by Mr. Westenfelder, of Cincinnati (both, I am glad to say, members of our Society), the latter of whom furnished me with the results of a long series of analyses bearing it out. Dr. Koch states as necessary characteristics of a good hide-powder for the filter method (which was used by all three chemists named), that the powder must be white, rather inclining to yellow than grey, fine and woolly, not powdery, and especially without any unpleasant putrefactive smell. The Vienna powder possesses these characteristics in a high degree, as also does one supplied me by Messrs. Reynolds and Branson, while my buffalo powder presented a perfect example of all the objectionable features condemned, and even after purification does not recommend itself by appearance; and yet, as I have shown, gives results by the "shake" method which agree perfectly with those of standard powders. This bears out the assertion that the "shake" method is far less dependent on the character of the powder than the filter method; but as it was quite impossible to use the buffalo powder in the filter, I am unable to test it directly. It is possible that the use of sulphurous acid in the purification of my powder may have removed the objectionable features complained of, but in the meantime I am inclined to attribute the error rather to the mechanical than the chemical state of the putrefied powders. I am not at all sure that with the American chemists it was the worse powders that gave the higher results, as with Dr. Koch. It is in any case important to employ the best possible powders, at least on check analyses, and to make quite sure, by means of comparative tests, before using one of more doubtful appearance, that it has no adverse influence on the accuracy of results.

#### DISCUSSION.

Mr. A. H. ALLEN said that he had been much interested in Mr. Procter's paper, and looked forward to seeing it in print. The subject of the recognition of tannin matters presented great initial difficulties. He thought that the classification of various kinds of tannin together simply because they precipitated gelatin resulted in the grouping of substances which did not necessarily belong to the same class, any more than copper, chromium, and nickel could be analytically classed together simply because their salts formed green solutions. He was interested to know that the nitrous acid test had yielded good results, though it was disappointing to find that Mr. Procter had not yet ascertained all the conditions of success. He thought that Mr. Procter had done wisely in substituting iron alum for ferric chloride, which certainly gave very erratic results, the ordinary laboratory reagent being of very indefinite nature. He was interested in Mr. Procter's results with copper acetate, as he had recently been employing it for an entirely different purpose. With the exception, perhaps, of lead acetate, it had a more general precipitating power than any other metallic salt. Some 20 years since he had described a reaction for gallotannic acid, or rather the tannin of tea, which was very delicate, and consisted in the red coloration produced with a solution of potassium ferrieyanide to which excess of ammonia had been added. He did not at that time extend his examination to many other kinds of tannin, and should be glad to learn if Mr. Procter had made any experiments in that direction. He should also like to know if Mr. Procter had succeeded in detecting phloroglucin in the product obtained by fusing

catechol and allied substances with caustic potash. He had tried many experiments in this direction, but had never succeeded in obtaining such a result as the books would lead one to expect. This was doubtless owing to his expecting to obtain a violet coloration with ferric chloride, which Mr. Procter has shown was not yielded by pure pyrogallol. As to the coloration produced by lime-water, experiments he made many years ago led to very erratic results when the test was applied in the presence of air. He had found, however, that interesting and more reliable results were obtained by operating in the absence of air. This he did by introducing the tannin solution into a nitrometer-tube filled with mercury. He then added through the tap lime-water or barite water, and observed the change. Then on gradually admitting air the changes due to gradual oxidation were conveniently observed. This method of manipulation might be advantageously employed in other cases where it was desirable to operate in the absence of air, or to admit air in regulated amount. Mr. Allen congratulated Mr. Procter on having found a simpler method of estimating tannins than that of Löwenthal, into which the personal equation entered far too largely. The examination of tannin materials was always a difficult problem, and different analysts did not always obtain concordant results.

Mr. RICHARDSON was glad that the use of hide-powder had proved satisfactory. What was understood by the term "tanning substance"? Did it include all substances that were absorbed by hide-powder? Surely other substances than tannins were so absorbed. He was surprised that nothing had been said of the use of basic acetate of lead. What was the action of mordants on tannins? He was under the impression that the action of tannins upon hide-powder was like that which they exercised in the dyeing process. Had Mr. Procter observed whether the action was different in the case of an iron and of a tin mordant?

Mr. BROADBENT was anxious to know how the hide-powder mentioned was prepared; could it be made from ordinary leather? What was the strength of the lime-water used, and had it been standardised?

The CHAIRMAN said he felt sure he might convey to Mr. Procter the thanks of the Section for his papers. The subject of the tannins was very complex and forbidding, but it was yielding, and would, no doubt, yield entirely to patient inquiry by the skilled specialist.

Mr. PROCTER, in replying, said that the number of the tannins was not so large as was generally supposed. Trimble had indeed been led, by his recent investigations, to think that there were only two. He had not studied the action of ammoniacal potassium ferrieyanide upon many tannins. Copper acetate probably precipitated all tannins perfectly. The nature of the precipitate was worth examining, the copper salt as a whole apparently combining with the tannin. The same was true of lead nitrate, which precipitated tannins without any liberation of free nitric acid.

Both neutral and basic lead acetates were general precipitants of tannins, and so hardly came within the scope of his inquiry, which concerned itself with qualitative recognition only.

He had done little work on the products of fusion of tannins with caustic potash; their instability under such severe treatment, and the great difficulty of separating them, were not encouraging. Fortunately the test for phloroglucin with deal shaving was so satisfactory that resort to fusion seemed unnecessary. The similar reaction reported of protocatechuic acid in the text books was probably due to impurity; a specimen he had from Kala Nam gave no trace of it, and, on the other hand, pure phloroglucin appeared to give no reaction with ferric salts, as would be seen by reference to Table VIII.

The use of the nitrometer for oxidisable solutions was an excellent device, of which he should certainly avail himself.



Hide-powder certainly did not absorb tannin only, but also acids, and many metallic salts, especially of the type of alum and aluminium sulphate, as well as other substances; as regarded mineral impurities, the nature of the ash of the extract gave some clue as to what would be absorbed by the powder. He was glad that the results of the hide powder and of the dyeing tests were similar; there was no *a priori* reason for expecting it.

The "buffalo-hide powder" was probably actually made from buffalo hide—not of the American but the Oriental buffalo; he had used it because it was the cheapest and most convenient material; it was otherwise not specially suitable for his purpose. The lime-water he used was a saturated solution, of strength about half decinormal.

### A RAPID METHOD OF DETERMINING THE SHADE OF ALIZARINE PASTES.

BY A. C. PERKIN, F.R.S.E., F.I.C.

THE following simple test, though little known to the outside world, is frequently used in most alizarine factories, and also to some extent in the Turkey red dyeworks in this country. As it provides a very accurate means of judging and comparing the shades of the various pastes rapidly, it should be useful to all users of this dyestuff, so that I have therefore thought it worthy of a communication to this society. It is well known that solutions of the alkaline hydrates readily dissolve alizarine, anthrapurpurine, and flavopurpurine, giving respectively blue-violet, crimson, and orange-red coloured liquids, containing the corresponding salts. This method of distinguishing between one or other of these substances has however this defect, that these solutions, necessarily dilute, rapidly alter and fade on standing, owing to oxidation. By employing the alkaline carbonates, preferably potassium carbonate, in the place of the hydrates, this difficulty is avoided, and coloured solutions of tolerable permanency obtained. The determination is carried out as follows:—Five grms. of the 20 per cent. paste is made up to one litre with distilled water, 5 cc. of this mixture treated in a flask of about 550 cc. capacity with 5 cc. of a 2 per cent. solution of potassium carbonate and then made up to half a litre with distilled water.

The coloured solution thus obtained can then be compared as desired with similarly prepared solutions of other pastes.

It is obvious that in the comparison of one paste with another by this means, flasks of similar diameter must be used, and further it is best that the solutions should be examined before a ground glass window, in order to avoid the shadows cast by outside objects.

With a little practice it is easy to detect very small differences in shade between two pastes, for instance an addition of 3 to 5 per cent. of anthrapurpurine to alizarine is discernible.

The composition of a paste may be also to some extent determined, for whereas the coloured solutions so obtained containing mixtures of alizarine and anthrapurpurine are clear and bright, on the other hand mixtures of alizarine and flavopurpurine are considerably duller, due to the yellow tint present in the alkaline solution of this latter.

With mixtures containing alizarine and both anthra and flavopurpurines, the analysis is naturally far more complex, many trials having often to be performed, and in cases of this kind the accuracy of the result should be further confirmed by a dyeing trial, using the usual mordanted calico. It is obvious, however, that by this method of testing the number of dyeing trials otherwise necessary is considerably reduced. Among other classes of alizarine colours with which this method can be used are the alizarine cyanines, and alizarine Bordeaux pastes, several shades of each being now in the market.

It is probable also that it would be of service with several mordant dyestuffs, which yield coloured liquids with carbonate of potash solution, and even possible that it could be used with advantage for determining by comparison the amounts of developed colouring matter in samples of log-wood and Brazilwood.

### DISCUSSION.

Mr. RICHARDSON asked why a tintometer had not been used with glasses to cut off the different shades.

Mr. SEATON remarked that the solutions could not, unfortunately, be kept permanently as standards, as they tended to fade.

Professor HUMMEL, in reply, said that the process as described by Mr. Perkin had proved in practice to be both rapid and accurate, and there would be no real advantage in making it more elaborate.

Mr. PERKIN, who was unable to be present at the meeting, desires to state, in reply to Mr. Richardson, that some advantage might be gained by the use of the tintometer, but it is doubtful whether the colour of the solutions was permanent enough for examination by this means. As regards the standard solutions, these are not kept in an alkaline condition, but, as stated in the paper, the requisite quantity of the standard solution is treated with potassium carbonate when required for comparison. The test as it stands is, to his knowledge, in frequent use, and is found to be of much service for the purpose.

### NOTE ON AN IMPROVEMENT IN DYEING LEATHER WITH BASIC COLOURS.

BY J. J. HUMMEL AND H. R. PROCTER.

IN applying basic coal-tar colours to cotton, it is well known that the latter requires to be mordanted with tannic acid. In the case of leather tanned with sumach and other similar tanning matters, such preparation is of course unnecessary, not only because the leather already contains tannic acid, but because the substance of the leather itself as a protein compound has a natural attraction for the colouring matter. Nevertheless, very poor results are frequently obtained in dyeing leather, *e.g.*, skivers, with the basic colours, the colours being pale or irregular. An examination of the dye-liquors in these and other cases revealed the fact that the colouring matter was very largely precipitated, due no doubt to tannic acid dissolving off the leather.

Two methods of getting rid of the defect naturally occur to one, *viz.*, to remove the excess of tannic acid present by previously steeping the leather in tepid water, or to render the excess inert by fixing it upon the leather in an insoluble form.

Both methods were tried with the result that the second proved to be the most reliable and effective. To this end it is merely necessary to work the leather in a tepid bath (45° C.) containing the requisite quantity of tartar emetic for  $\frac{1}{4}$ — $\frac{1}{2}$  hour, then to wash, before proceeding to the dyeing operation.

By adopting this simple precaution, the dye-liquors are maintained in a perfectly clear condition, and since they are invariably unexhausted, they can be used for dyeing further quantities of leather; moreover, the dyed colours are perfectly level, and many shades darker than without this preliminary treatment. The leather itself is in no wise injured, and since the antimony is in a perfectly insoluble form as a tannate, there can be no fear of injurious consequences arising in the ordinary use of such leather.

We are not aware that the method is adopted in practice, but after repeated trial on the small scale, we have every confidence in recommending it to the trade.

We may add in conclusion that our belief is that a judicious application of tartar emetic in limited quantity along with basic colours in the process of staining leather by brushing on, would in many cases be advantageous.

#### DISCUSSION.

Mr WILKINSON asked whether antimony fluoride or oxalate could not be used instead of tartar emetic.

Professor HUMMEL said they would no doubt do equally well.

### Scottish Section.

*Chairman:* R. Irvine.

*Vice-Chairman:* E. J. Mills.

*Committee:*

G. Beilby,	W. I. Macadam.
W. J. Chrystie,	J. S. Macarthur.
J. Clark,	T. L. Patterson.
C. J. Ellis,	J. Patterson.
G. G. Henderson,	J. B. Readman.
R. A. Inglis,	E. C. C. Stanford.
R. Irvine,	D. R. Stewart.
J. Falconer King,	R. R. T. Tuck.

*Hon. Secretary and Treasurer:*

J. Stanley Muir, Chemical Laboratory, University of Glasgow.

The names in italics are those of Members of Committee who retire at the end of the current session.

The following have been elected to fill the vacancies, and will take office in July next:—*Chairman:* Robt. Irvine. *Vice-Chairman:* J. Clark. *Committee:* R. Cox, C. A. Fawsitt, D. Harris, E. Ostlere, R. Pullar, and H. Rose.

#### SESSION 1893-4.

*Meeting held in the Philosophical Society's Rooms, 207 Bath Street, Glasgow, on Tuesday, March 7, 1894.*

THE HON. CLARK, F.R.S.E., in the Chair.

### ON THE SUITABILITY OF WATERS FOR DIETETIC PURPOSES AS SHOWN BY CHEMICAL ANALYSIS.

BY R. R. FAULKNER AND R. L. THOMSON.

The question of the suitability of waters for dietetic purposes is determined by the results of their analysis, is one on which every chemist is called upon to give his decision, and the interpretation of these results, and even the very methods in use, have been the subject of difference of opinion. This discussion has been once more reopened in the "Analyst" by Mr. F. W. Stoddart whose aim has principally been to plead for the inclusion of nitrates as one of the elements of proof of the presence of sewage in a water. We have therefore thought it timely to lay before the Society the principles by which, during a period of some 20 years, we have been guided in determining the suitability of waters for dietetic purposes so far only as the question of the presence or absence of sewage is concerned.

To attain our object it is plainly necessary in the first place to ascertain the composition of sewage, and from the data obtained to select those which are specially characteristic of it, and which are likely to give the best indication of the pollution of waters. It is obvious that the free or saline and the albuminoid ammonia are ingredients of the first importance, and we are also inclined to place nitric nitrogen or nitrates in the same category, although of course these can only be derivatives of sewage. Of subsidiary importance is the amount of oxygen absorbed from permanganate of potassium, and of less value still is the proportion of chlorine. Before proceeding with remarks on these data, we would draw attention to the accompanying table, which contains various analyses of waters polluted and unpolluted, and which will be necessary for reference as we proceed.

TABLE I.

ANALYSIS OF VARIOUS SAMPLES OF WATER, THE RESULTS BEING STATED IN GRAINS PER GALLON.

	1	2	3	4	5	6	7	8	9	10	11
Free ammonia.....	0.002	0.002	0.002	0.008	0.012	0.020	Neg.	0.001	0.005	0.004	0.028
Albuminoid ammonia.....	0.004	0.009	0.009	0.011	0.012	0.014	0.005	0.008	0.008	0.012	0.009
Nitric anhydride.....	..	..	..	..	..	..	0.013	0.021	1.570	10.98	From 0.14
Equal to ammonia.....	..	..	..	..	..	..	0.004	0.006	0.194	3.174	..
Free ammonia originally present.....	0.002	0.012	0.002	0.008	0.012	0.020	0.004	0.007	0.149	3.247	..
Ratio of albuminoid ammonia : free ammonia = 1.....	2.0	4.5	4.5	1.4	2.7	1.7	1.2	1.1	6.24	27.09	..
Oxygen absorbed in 1 hour.....	0.038	0.072	0.074	0.139	0.192	0.170	0.072	0.112	0.117	0.0	..
Ratio of oxygen absorbed to albuminoid ammonia = 1.....	9.5	8.0	8.2	12.6	18.5	13.8	14.4	11.9	2.1	4.0	..
Chlorine.....	1.53	..	1.85	2.86	4.30	..	0.52	1.68	1.2	7.80	..
Average sewage, per cent.....	..	..	..	..	..	..	..	..	10.0	64.7	..

NOTE.—The free ammonia originally present is the actual free ammonia found and the equivalent of the ammonia which has been added together.

In considering the data referred to, it will be well to take the free and albuminoid ammonia together.

*Free and Albuminoid Ammonia.*—It may be as well to explain here that Wanklyn's albuminoid ammonia determination has been selected in preference to Frankland's organic nitrogen estimation, simply because the former is

so much more easy of manipulation than the latter, and although it does not give absolute results, yet they are decidedly reliable for the purpose in view.

Undoubtedly the quantities and relative proportions of the free and albuminoid ammonia are the data which are of greatest value in determining the presence or absence of

sewage, but, so far as we can gather, no good estimate of these has been published. The best estimate of the maximum, minimum, and average strength of sewage as regards free and albuminoid ammonia was made for the late Dr. Wallace, who obtained samples from the Glasgow sanitary inspector. These were taken every hour between 6 a.m. and 11 p.m. simultaneously from a sewer in the eastern district and from another on the south side of Glasgow. Each of these 36 samples was analysed separately, and in the following table will be found the maximum and minimum free ammonia, along with the albuminoid ammonia in the same samples, and the maximum and minimum albuminoid ammonia as well as the free ammonia in these particular samples, all the results being recorded in grains per gallon.

TABLE II.

FREE AND ALBUMINOID AMMONIA IN SEWAGE, showing the Maximum and Minimum in Grains per Gallon.

Glasgow.	Maximum Free Ammonia.	Minimum Free Ammonia.	Maximum Albuminoid Ammonia.	Minimum Albuminoid Ammonia.
East End:—				
Free ammonia	9.300	1.550	5.400	4.480
Albuminoid ammonia.	0.240	0.266	0.686	0.154
South Side:—				
Free ammonia	8.480	2.856	4.900	3.640
Albuminoid ammonia.	0.210	0.210	0.630	0.182

Besides expressing these facts, it will be well to give the average composition of the sewage of each district, and from that deduce the average of Glasgow sewage, and these are recorded in the following table:—

TABLE III.

AVERAGE COMPOSITION OF GLASGOW SEWAGE.

	Average of Sewage for entire Day from East End of Glasgow.	Average of Sewage for entire Day from South Side of Glasgow.	Average for Glasgow Sewage.
Free ammonia .....	5.190	4.960	5.07
Albuminoid ammonia ..	0.327	0.333	0.33

Other useful data which may be drawn from these results are the highest, lowest, and average proportion of free to albuminoid ammonia in individual samples, and these may be stated as follows, taking one part of albuminoid ammonia as a fixed quantity:—

	Maximum Proportion.	Minimum Proportion.	Average Proportion.
Free ammonia .....	40	6	16
Albuminoid ammonia ..	1	1	1

More recently we have analysed various samples of Glasgow sewage, and the results so far corroborate those recorded. Thus, a water contaminated with sewage ought to have on an average about 16 times as much free as albuminoid ammonia, provided it has not undergone nitrification and does not contain vegetable matter in solution. Practically the same view is held by Mr. Stoddart, but the fact has been lost sight of by some chemists who have depended too much upon a single item of analysis which

appears to point to contamination, whereas it might not do so if all the items had their due weight attached to them. Our contention, then, is that the quantities of free and albuminoid ammonia, and the ratio of these to each other in a water which has not been subjected to nitrification, are the chief rational measures of the pollution of waters with sewage. Once the presence of sewage is proved, its proportion in the water may be calculated from the grains per gallon of free ammonia, taking 5 grains per gallon as representing 100 per cent. of average sewage. Thus, for instance, a water containing 0.05 grain per gallon of free ammonia will represent a contamination of the water with 1 per cent. of average sewage.

At this point the questions arise, what ratio do the free and albuminoid ammonia bear to each other in waters not containing any animal organic matter, but only vegetable or mossy matter in solution? And how much free and albuminoid ammonia are we to regard as naturally present in unpolluted waters? In Table I. will be found the analyses of six pure natural waters, which have not undergone any appreciable nitrification, and in which the proportion of organic matter of vegetable origin varies from one of the lowest we have observed in No. 1 to the highest in Nos. 5 and 6. These have been selected as representative specimens of hundreds of analyses of waters from different localities, with the view of showing the range of purity as regards mossy matter that may be expected in natural waters uncontaminated with sewage. From an examination of these results it is apparent that the albuminoid is in every case in excess of the free ammonia, varying in the proportion of 1.4 to 4.5 of the former to 1 of the latter. It will thus be seen that there is an essential difference in the ratio between the two ammonias according as they are derived from animal or vegetable matters. In fact it may be taken as a general rule that when the organic matter present is purely of vegetable origin the albuminoid exceeds the free ammonia in amount, but we do not insist that this holds good in all cases, although it is exceptional to find it otherwise. The second question has reference to the absolute amount of free ammonia which may be allowed in a water, and our experience points to 0.01 or at most 0.012 grain per gallon if the albuminoid ammonia does not exceed that figure. When the water, however, is of a more mossy character, the albuminoid ammonia is naturally higher, and we have found that the free ammonia also increases in such cases even up to 0.02 grain per gallon, and, as far as our observation goes, invariably more albuminoid ammonia, an example of which is given in No. 6 analysis on Table I. These figures are based upon many hundreds of analyses of waters, mostly from different districts, made by us, and by going more carefully into about 400 samples analysed during the last two or three years, we find that about 66 per cent. come within these somewhat stringent limits stated, and even 58 per cent. pass the lower of these limits, thus showing how pure it is possible to get waters. These results indeed seem to be inconsistent with the idea that rain water gathers any appreciable quantity of ammonia unless it has been collected from sooty and otherwise dirty roofs.

It should be pointed out here that there is a decided peculiarity about chalybeate waters as regards the presence of free ammonia, which is higher than usual, and also bears a high proportion to the albuminoid ammonia, as will be seen by reference to analysis No. 11 on Table I. This is not a solitary instance, but we have found the same repeatedly in samples from deep bores in which it was said there could not possibly be any sewage contamination. In one particular sample, of which we do not know the source, and which contained 0.38 grain per gallon of iron in solution, there was found 0.448 grain of free and 0.042 of albuminoid ammonia. In another sample which was obtained from South America there was no less than 3 grains, each of free and albuminoid ammonia per gallon, accompanied with 13 grains of iron in solution. It is almost impossible to assign a good reason for the presence of these large quantities of ammonia unless by the presence of animal matter, but there is without doubt a peculiarity about chalybeate waters, however it may be accounted for, inasmuch as they contain sensibly more ammonia than ordinary unpolluted waters.

**Nitrates and Nitrites.** It is scarcely necessary at this point to go over again the arguments for including nitrates and nitrites as factors in judging of sewage contamination, as this has been done so recently by Mr. Stoddart, who has also demonstrated the rapidity of the oxidation of ammonia by filtration through soil. This we also observed some years ago in the case of a sewer which was leaking, and after the sewage had got mixed with more water and passed through a few feet of soil the 0.7 grain per gallon of ammonia it contained was almost entirely converted into nitrates and nitrites. This rapid nitrification can no longer be denied, nor can the fact of the danger of contracting disease from even almost perfectly oxidised sewage water, of which several cases are now on record. One very important case has been described by Dr. Milroy in his excellent paper entitled "How Typhoid Fever Spreads." He there shows how typhoid fever appeared in Fergushill and Bensley, villages near Kilwinning, and continued there for eight years, or so long as the people were allowed to drink waters containing only 0.002 grain of free and 0.003 grain of albuminoid ammonia per gallon, but at the same time they contained 2.75 grains of nitric anhydride, being condemned by Dr. Clark. In the face of such facts it is quite wrong to persist in ignoring nitrates and nitrites, or to fix a limit of 0.77 grain per gallon of nitric anhydride, as even Mr. Stoddart seems reluctantly to do. In judging the analysis of water it is a great mistake to allow the very highest proportion of nitrates and nitrites that *might possibly* be present from other sources than animal matter, and only condemn them for anything above that. It is surely more reasonable to argue as to their purity from the number of really good waters that can be obtained in comparison with the bad or suspicious ones. In the following table we give the percentages of waters of various kinds obtained from different districts:—

	Per Cent.
Unpolluted waters with less than 0.012 grain per gallon of free ammonia .....	58
Unpolluted waters containing 0.012 to 0.002 grain per gallon of free ammonia .....	8
Waters containing more than 0.002 grain per gallon of free ammonia, several known to be polluted .....	8
Waters containing under 0.77 grain per gallon of $N_2O_5$ ..	9
Waters containing 0.77 to 2.00 grain per gallon of $N_2O_5$ ..	9
Waters containing above 2 grains per gallon of $N_2O_5$ ..	8
	100

Of these waters 34 per cent. were reported as polluted with matter of the nature of sewage, and in some cases we were informed that sewage was undoubtedly getting in. There can be no valid reason for supposing that the 9 per cent. under 0.77 grain of  $N_2O_5$  per gallon are likely to be polluted with anything but sewage when we consider that two-thirds of the whole number are so very pure. In fact the probabilities seem rather to lie in the other direction. Taking everything into account, we have come to the conclusion that if the nitric anhydride found is calculated to ammonia and added to the free ammonia actually found, in order to find the free ammonia originally present, the final figure thus obtained ought not to exceed those laid down under the free and albuminoid ammonia section.

The rapid nitrification of the free ammonia in waters by simple keeping should be borne in mind. A mixture of Loch Katrine water and sewage containing over 0.3 grain of free ammonia per gallon, lost 0.04 grain per day by conversion into nitrate and nitrite. Thus, a water despatched on the one day and arriving the next might lose a comparatively large proportion of ammonia before it could be analysed. Analyses of waters containing nitrates are given in Table I., Nos. 7 and 8 being unpolluted, and 9 and 10 contaminated with sewage.

**Oxygen absorbed.**—Tidy's modification of the Forschammer oxygen process is a useful subsidiary test, which may help to throw some light on the character of a water. Our results in the waters in Table I. were calculated from experiments allowed to stand for one hour only, as there is no advantage gained for our purpose by allowing to stand for a short and a long period. If nitrites are present, the test is only valuable if they are estimated separately, but not by

permanganate, and deducted from the total result. No oxygen experiments were made with the polluted samples, but the free and albuminoid ammonia tests in the following table gives a comparison of the albuminoid ammonia and the oxygen absorbed in over 20 samples of sewage.

	Oxygen absorbed per gallon of water	Albuminoid ammonia per gallon of water
No. 1	1.27	0.10
No. 2	0.70	0.05
Average	1.06	0.08

These results show that the lowest oxygen absorbed is 2.4 times that of the albuminoid ammonia corresponding to it, the highest 8.1, and the average 4.6 times. These figures agree with the ratio of these two items in waters Nos. 9 and 10, which we found reason, in the presence of nitrates, to believe to be polluted with sewage, and which is confirmed by this test also. The difference from the ratio between the oxygen absorbed and the albuminoid ammonia in pure waters is apparent when the analyses of Nos. 1 to 8 are examined, these showing the maximum difference to be 18.5 times, the minimum 8.0 times, and the average 12.3 times more of the oxygen than the ammonia. It will be apparent, without further explanation, that these figures may also be of value in corroborating other results. It may be noted in passing that the colour of a water usually gives a fair indication of the presence of mossy matter.

**Chlorine.** The chlorine has often been insisted on as a useful proof of sewage contamination, but it is obviously of practically no value, seeing we can have pure waters varying from 0.52 to 1.3 grains per gallon of chlorine. The maximum chlorine we have found in sewage was 9.11, and the minimum 2.51, with an average of 4.92 grains per gallon. A water therefore which had got contaminated with, say, 1 per cent. of sewage would have on an average 0.05 grains per gallon added to its chlorine, and it is at once apparent that such a small quantity could never be discovered in an item which is so variable. In Table I., indeed, we have a contaminated water (No. 9) containing much less chlorine than such a pure natural water as No. 1. The very high chlorine in the grossly contaminated water No. 10 is noticeable as being probably derived from sewage, but its indication of pollution is of little value when it is impossible to miss finding it by the means which must always be applied. The estimation of chlorine for the purpose we have in view may therefore be dispensed with.

These, then, are the various points which we consider as absolutely necessary to be attended to in giving a decision on the character of a water. In many instances the evidence, based on analysis, of contamination of water by matter of the nature of sewage, is palpable, and does not require any fine discrimination; while in some cases much judgment and experience are necessary to determine the presence or otherwise of sewage impurity; and the latter remark applies not only with regard to the proportions of normal or abnormal ingredients found, but also to the ratio in which these stand to each other. But which analysts may differ along the border line, so to speak, as to whether or not certain results indicate or establish sewage contamination, there can surely be but one opinion as to the unsuitability for dietetic purposes of any water which admittedly does contain sewage matter, or even that gives evidence of previous sewage contamination, although the ammonia has practically disappeared and all its nitrogen exists as combined nitric acid, having been oxidised to its fullest limit. It by no means follows that because the dead nitrogenous matter has been fully oxidised the lethal microbe and its spores have been destroyed, and it has been noticed (as in the case brought forward by Dr. Milroy) that the clearest well waters, which are often the most palatable, are frequently responsible for the propagation of zymotic diseases. A water containing an appreciable proportion of nitrates due

originally to sewage matter may be free from objection, but that can only be either because the sewage was not infected sewage, or because the water, after receiving the impurity, has filtered through a sufficient thickness of soil, chalk, sand-stone, or other medium to ensure the absence of the minutest solid particles, such as micro-organisms and their spores. In the former case the uninfected sewage may become infected at any moment, on the outbreak of disease at its source, and in the latter case it may not be known to the analyst, and very seldom is, what natural or possibly artificial treatment the water may have received with the result of eliminating disease-producing germs. His only safe course, then, is to condemn as dangerous for dietetic use any water containing an appreciable amount of nitrates, until it is ascertained from the history of the water, confirmed, if possible, by bacteriological examination, that there is no possibility of the presence of micro-organisms of an objectionable character, and that these, even if infected sewage were percolating into the water, would always be perfectly retained by the filtering medium. Chemical analysis, therefore, is a safe and reliable guide in the question of the suitability of a water for drinking and dietetic purposes, as it will practically always detect sewage or sewage residue, and it only remains to avoid the use of such water. It is not sufficient that bacteriological examination shows the absence of pathogenic germ life in a particular sample of water, as such water might be contaminated by sewage free from disease germs, but which might become infected at any time. On the other hand, chemical analysis may fail, although rarely, to detect sewage matter or to trace it, as, for example, when all the nitrate originating from sewage nitrogen has been taken up by plants growing on soil through which the water has percolated. Then a bacteriological examination is of value, and indeed is the only means available for the determination of the suitability of the water for dietetic use. Numerous field experiments of our own have shown that rain water allowed to percolate through bare soils without vegetation dissolve in a few weeks nitrates contained in the soil amounting to several grains per gallon of the water, but that where there is vegetation growing the nitrates are entirely removed by the plants, none whatever being found in the percolate.

#### DISCUSSION.

The CHAIRMAN thought that Mr. Thomson had brought forward a subject which, to do it justice, would require a special meeting for consideration. He agreed practically with everything Mr. Thomson had said, and in reporting upon waters for many years past, he had been in the habit of relying mainly upon the data to which Mr. Thomson had referred. These data being the ammonia, the nitric acid, and the oxygen absorbed. In England much greater difficulty was experienced in valuating the results of analysis than in Scotland. That arose from the fact that in England there were deep bores, especially in the chalk strata, which contained a considerable quantity of nitrates. In Scotland there were very few, if any, deep-bore waters containing nitrates. Nearly all the waters containing nitrates were found near the surface or in very shallow wells, and practically in every case where considerable quantities of nitrates were found, they could be traced to a sewage origin. For a long time it was held by chemists that the presence of nitrates, being themselves innocuous, was no objection. It had been shown in recent years that while the nitric acid itself was not objectionable, its presence was a proof of sewage contamination or at least sewage infiltration, and, if the sewage was contaminated with any specific disease, germs of that disease were capable of multiplying in the water. As a matter of fact, a considerable number of wells which, judged by the free ammonia, or albuminoid ammonia, or the oxygen absorbed, seemed to be perfectly pure and harmless, were capable of producing specific diseases such as typhoid fever, and in these waters the only substance that could be found by analysis likely to indicate the cause of this specific disease, was the nitric acid which had been formed from the oxidation of sewage matter. Mr. Thomson had referred to a water at Kil-

winning which was reported as contaminated with sewage, and was found to be the means of propagating typhoid fever. The Cross well water at Kilmaurs was found to have communicated typhoid fever to a great many of the inhabitants. This Kilmaurs water contained free ammonia per gallon 0.001 grain, albuminoid ammonia 0.004 grain, and oxygen required 0.03 grain. These were proportions of free ammonia, albuminoid ammonia, and oxygen required, which by themselves would indicate purity; but side by side with these small quantities of ammonia and oxygen required, there was as much as 2.8 grains of nitric acid per gallon, and upon these data, basing the calculation on the presence of 5 per cent. of ammonia in average town sewage, about 17 per cent. of town sewage had been filtering into this well. In this case no attempt was made to subject the water to a bacteriological examination; but in a more recent case in a water of a different character, which contained very much more nitric acid—9.8 grains—equivalent to about 60 per cent. of oxidised town sewage, and in addition free ammonia 0.042 grain, and albuminoid ammonia 0.012 grain, on being subjected to a bacteriological examination, typhoid germs were detected. He agreed with Mr. Thomson that in judging of the character of a water there was probably nothing which gave a clearer indication, especially in a shallow well, of sewage infiltration than the presence of a considerable quantity of nitric acid. There was, however, a difficulty in laying down any particular limit above which a water might be said to be contaminated with sewage. In a country where the soil was extensively cultivated, the surface drainage of the land caused the water to contain a little nitric acid. So far as his own experience went, the amount of nitric acid in such water, even where the lands were heavily manured, seldom exceeded from 0.5 to 0.7 grain of nitric acid per gallon, and, unless where there were special reasons to indicate that this nitric acid was not from the ordinary cultivation of the land but from sewage, it could hardly be said that such a water was dangerous for domestic purposes. Where the percentage of nitric acid was appreciably greater he thought that, unless it was accountable for otherwise, there was a clear indication of sewage infiltration and consequent danger.

To the presence of chlorine in the water, he attached no importance, as the difference in the amount of chlorine was not such as would give any real indication of the quality of the water.

Regarding the amount of ammonia present in water, it must be remembered, more especially with peaty waters, that a considerable quantity of albuminoid ammonia might be found in them. In many peaty waters, such as were used for distillery purposes, there was as much as 0.04 grain per gallon of albuminoid ammonia. He did not mean to say that peaty water was good for drinking purposes, but it was certainly excellent for the production of high class whisky. Deep pit waters, especially those which contained carbonate of soda, and waters that were derived from shale strata and deep borings, frequently contained a considerable quantity of ammonia, but this ammonia was not necessarily an indication of sewage contamination and might be due to the strata.

He had occasion to examine a deep-bore water from Ayrshire, intended to supply a town, and it contained so much free or saline ammonia that, under ordinary circumstances, it would have been described as highly contaminated, but there was no sewage in the neighbourhood. He attributed the ammonia present to the shale strata.

The PRESIDENT understood Mr. Thomson to differentiate between organic matter from an animal and from a vegetable origin, although he did not quite gather the way in which the difference was proved.

He did not quite agree as to the absolute connection between "previous sewage contamination" and nitrates, and could not help thinking that the words "previous sewage contamination" in connection with nitrates had done a great deal of harm. He would certainly have looked upon the presence of nitrates as being an element of change and as dangerous in water. No doubt in shallow wells the nitrates could only come from one source, but he remembered

examining a water which came from a bore in the lower chalk about 500 feet deep, which contained 4 grains of nitrates to the gallon. It was absolutely free from organic matter and was delightful water to drink. Now, who could prove that these nitrates were derived from "previous sewage contamination"? The Chairman, when he found chloride of ammonium in water in undue proportion, did not put it down to previous sewage contamination. He very properly thought that it must have been derived from the strata. Why should not the nitrates in the deep well water be derived from the same source?

Professor HENDERSON desired to thank Mr. Thomson for having had the boldness to fix a standard, or at any rate what he believed to be a standard, of purity for water. He spoke with the utmost diffidence in the presence of experts in water analysis, but his experience was that anyone, other than an expert, had considerable difficulty in determining from the results of his analysis the suitability of waters for domestic use; for, if he referred to the standard writers on the subject, he found generally that the information he obtained contributed rather to increase his perplexity than to remove it. He wished that water experts could come to some agreement on the subject, or could at least agree to differ in an intelligible way. He observed that Mr. Thomson had discarded the determination of the ratio of organic carbon to organic nitrogen, on which Frankland laid so much stress, and he wished to know if Mr. Thomson had any reason for this, other than the desire to simplify the experimental work. As regards the occurrence of nitrates in water, it seemed to him that it was not necessary to conclude that this indicated "previous sewage contamination," because it was perhaps possible that nitrates might be formed directly from atmospheric nitrogen. He was under the impression that he had recently seen it stated that an observer had succeeded in isolating a micro-organism which possessed this power, and would be glad to know if this observation had been confirmed.

Mr. TARLOCK said that the principal point in connection with the paper was the fact that it was necessary to insist upon the nitrates present in water being interpreted as indicative of sewage contamination, unless there was information to the contrary. Whether the nitrates were of animal origin or not was a matter of some importance, but when a chemist received a sample for analysis, he did not know anything regarding its history. It was, for example, possible to suppose that the nitrates in the deep chalk waters had originated in other ways than from animal matter; but most probably they were derived from antediluvian animal matter which had got converted into nitrates.

In the case of Altona, it was clearly shown that mechanical means were quite capable of dealing with contaminated water. So long as the filters were in good order, cases of cholera diminished day by day, but on some accident happening to the filters, a fresh outbreak of the disease at once occurred.

So much recent evidence had been brought forward with regard to the presence of nitrates, indicating, if not recent, at least, previous sewage contamination, that he was surprised that any waters should be classed as pure which had not been thoroughly filtered through chalk soil or other medium sufficient to remove pathogenic germs. He thought the proper course to adopt was to report such waters as risky or dangerous—not necessarily as unfit—but as dangerous. A water might be unfit or it might be dangerous where the source of the nitrate was unknown, but might be perfectly fit if filtered sufficiently to exclude all germ contamination.

Mr. THOMSON, while he differentiated between animal matter and vegetable matter, did not mean to say that this problem was solved. The methods employed of estimating the oxygen absorbed and the albuminoid ammonia gave some indication, especially when the other results were taken into consideration, whether the organic matter was due to vegetable or animal origin matter.

The President thought Mr. Thomson's method was more likely to solve the problem than Frankland's method of comparing the carbon and the nitrogen.

Mr. THOMSON, continuing, said that the oxygen absorbed in one experiment was 8.1 times as much as the albuminoid ammonia in sewage, and with the vegetable matter in one instance the same relationship was apparent, so that they crossed each other. The composition of sewage showed that its organic matter was not of purely animal origin. Some of the sewage analysed by Mr. Wallace contained a large amount of vegetable matter, particularly about 12 or 1 o'clock in the day, when the washings of vegetables were present in large quantities. These could not be actual results from animal matter alone, for they must always include vegetable matter. It might be possible to differentiate between pure vegetable and pure animal matter, but they could not differentiate with the same reliability between the organic matter of sewage, which is a mixture of animal and vegetable, and organic matter of purely vegetable origin. There were two microbes said to be in existence, the one producing nitrates and the other producing nitrites, and the one might not be a stage of the other.

The President: Was it not the case that the nitrate-microbe only acted upon nitrite?

Mr. THOMSON doubted this. Warrington, though his opinion was not expressed in a way quite free from ambiguity, seemed to think that nitrates could be formed without the intermediate production of nitrites.

Mr. HENDERSON: Warrington's American Dictionary definitely stated that the nitrate organism only acted on the products of the nitrite one, and would not act upon anything but the nitrite.

Mr. THOMSON said the article he referred to was published about two years ago by Warrington in Thorpe's Dictionary of Applied Chemistry (Vol. II. page 699).

Mr. HENDERSON: Both organisms had been isolated and cultivated in the pure state.

The President: One would produce the nitrite and the other only the nitrate from the nitrate.

#### POSTPONED DISCUSSION ON "THE PRODUCTION OF OIL-GAS FROM PARAFFIN OILS AND FROM PURE MEMBERS OF THE PARAFFIN AND TERPENE SERIES OF HYDROCARBONS." (This Journal, 1894, p. 231-237.)

Mr. BENBY thought that it would add to the value of an investigation such as this if the elements of time, temperature, and heating surface were so clearly discriminated that it would be easy to give each its due value; so that it might be possible in each experiment to say not only the temperature to which the oil vapour had been exposed, but also the time during which the exposure had lasted.

Dr. T. R. MARSHALL inquired regarding the destructive distillation of Ozokerite, as that substance was one of considerable commercial importance.

Mr. FARWELL considered that the greatest drawback to oil-gas was its tendency to condense in the mains. The illuminating power was considerably lowered after the passage of the gas from the works through the mains to the place of consumption. By introducing a percentage of oxygen the gas was found not to condense, but to retain its illuminating value. Had Mr. Teich conducted any experiments such as would bear on this point?

Mr. Töcher, in reply, said, oil gas had been spoken of as being unstable, but as a matter of fact that was erroneous. On subjecting properly made oil-gas and coal-gas to the same variations in temperature, condensation was less from oil-gas than from coal-gas. He had not experimented with mixtures of oil-gas and oxygen although he at one time thought of doing so. Dr. Thorne claimed for oxy oil gas a greater enrichment figure than for oil-gas, while Professor Lewes and others said that equally as good results could be obtained by using oil-gas alone as an enricher. Oil-gas, when used as an enricher, showed an enrichment figure far in excess of its actual illuminating power, no matter what method was employed to manufacture the gas.

The enrichment values of mixtures of oil-gas (15–20 per cent.) and coal-gas (80–85 per cent.) had been repeatedly determined. Beyond this he was not aware of any other researches having been conducted, and it had therefore occurred to him to determine the intrinsic illuminating value of oil-gas in proportions varying from 5 to 75 per cent. The enrichment figure for oil-gas increased as the percentage taken decreased, until 118 candles were reached, equivalent to an increase of about one candle per cent. of oil-gas taken. The destructive distillation of Scotch oils was undertaken in order to determine the products of decomposition at various degrees of temperature. Professor Lewes had given the results of carefully-conducted experiments on the cracking of petroleum, and all were familiar with the valuable work done by Dr. S. Macadam on Scotch oils. Continuing, Mr. Töcher explained that his experiments, as embodied in Table I., might be considered as supplementary to those hitherto published and to determine with more precision the function which temperature played in the manufacture of gas from paraffin oils. Gas engineers had at their disposal two systems of oil-gas manufacture: (1) the cracking up of the oil at a moderately high temperature and in one operation, with the formation of about 10 to 15 per cent. of tar, and (2) the cracking up of the oil at a low temperature and subsequent re-cracking of tarry matter formed together with undecomposed oil, the ultimate products being gas and coke. The latter was the principle of Mr. Young's process, and although Mr. Young claimed that the small ratio of square surface and consequent greater free space in his retorts were important factors, still it was evident that the value of Mr. Young's process lay in the fact of its being very economical owing to the low temperature employed and to the complete conversion of the oil into gas and coke. A wide retort was preferable to a narrow one because it prevented the roasting of newly-formed gases, and for the same reason the use of iron filings or coke ought to be avoided. He quite agreed with Mr. Beilby that the principal affecting oil-gas manufacture were by no means fully understood and that there was ample field for further investigation. In instituting comparisons between, and making experiments with, retorts of various length and surface ratios, and in dealing with different rates of flow, he had endeavoured to combine the various factors in the production of oil-gas, as he thought too much stress had been hitherto laid on the question of free space alone. In the experiment with mineral oil (0.884), where he had expressed the decomposition of the oil in the form of an equation, the results would be very similar, no matter whether a Young, Patterson, or Pintsch retort were used, as the rate of flow would of course be regulated to the capacity of the retort. He had not conducted any experiments with Ozokerite, but, being an olefine, it ought to yield, on destructive distillation, a very rich gas. Being a solid hydrocarbon, there would be some difficulty in using it on a practical scale.

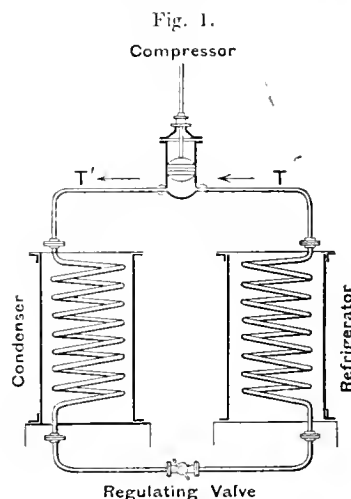
## Journal and Patent\* Literature.

Class.	Page
I.—General Plant, Apparatus, and Machinery .....	592
II.—Fuel, Gas, and Light .....	595
III.—Destructive Distillation, Tar Products, &c. ....	597
IV.—Colouring Matters and Dyes .....	598
V.—Textiles: Cotton, Wool, Silk, &c. ....	512
VI.—Dyeing, Calico Printing, Paper Staining, and Bleaching .....	513
VII.—Acids, Alkalis, and Salts. ....	513
VIII.—Glass, Pottery, and Enamels. ....	518
IX.—Building Materials, Clays, Mortars and Cements..	519
X.—Metallurgy .....	520
XI.—Electro-Chemistry and Electro-Metallurgy .....	527
XII.—Fats, Oils, and Soap Manufacture. ....	529
XIII.—Pigments and Paints; Resins, Varnishes, &c.; India-Rubber, &c. ....	532
XIV.—Tanning, Leather, Glue, and Size .....	532
XV.—Manures, &c. ....	533
XVI.—Sugar, Starch, Gum, &c. ....	533
XVII.—Brewing, Wines, Spirits, &c. ....	533
XVIII.—Chemistry of Foods; Sanitary Chemistry and Water Purification; Disinfectants .....	536
XIX.—Paper, Pasteboard, &c. ....	538
XX.—Fine Chemicals, Alkaloids, Essences, and Extracts	539
XXI.—Photographic Materials and Processes .....	543
XXII.—Explosives, Matches, &c. ....	543
XXIII.—Analytical Chemistry .....	543

## I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

*Refrigerating Apparatus.* C. Linde. J. Soc. Arts. 42 (1894), 322–330.

THE author introduces his subject by describing the method used by Prof. Dewar in liquefying oxygen and air. Carbonic acid is allowed to pass from a drum into a vessel (Fig. 1) in which atmospheric pressure is maintained.



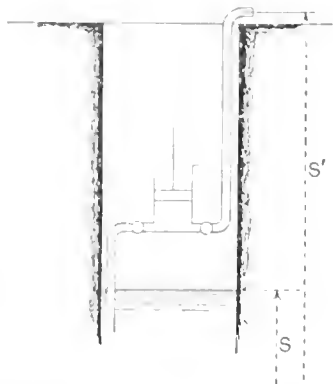
The carbonic acid boils at  $-80^{\circ}\text{C}$ . ( $-112^{\circ}\text{F}$ .). Ethylene vapour is compressed into a second vessel placed inside the first; it liquefies and is then allowed to enter a third vessel

\* Any of these specifications may be obtained by post by remitting *sd.*—the price now fixed for all specifications, postage included—to Sir Henry Reader Lack, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.



under atmospheric pressure. The ethylene boils at about  $-100^{\circ}\text{C}.$  ( $-141^{\circ}\text{F}.$ ). At a lower pressure the ethylene would boil at  $-140^{\circ}\text{C}.$  ( $-220^{\circ}\text{F}.$ ). If oxygen be compressed into a closed vessel placed within the ethylene vessel, it is liquefied, boiling under atmospheric pressure at  $-180^{\circ}\text{C}.$  ( $-290^{\circ}\text{F}.$ ); by reducing this pressure, the temperature can be lowered to  $-200^{\circ}\text{C}.$  ( $-328^{\circ}\text{F}.$ ) at which atmospheric air condenses. Although methods are thus available for producing and maintaining temperatures not far distant from the absolute zero, the lowest temperature hitherto employed for industrial purposes is  $-110^{\circ}\text{C}.$  ( $-166^{\circ}\text{F}.$ ) which has been used for obtaining very volatile organic substances in a state of purity. The author proceeds to consider what efficiency is obtainable; what working processes are the best; and what is the position of existing machines with reference to these considerations. He compares the action of a cold-producing machine to that of a pumping-engine (Fig. 2), which has to keep water in a

Fig. 2.

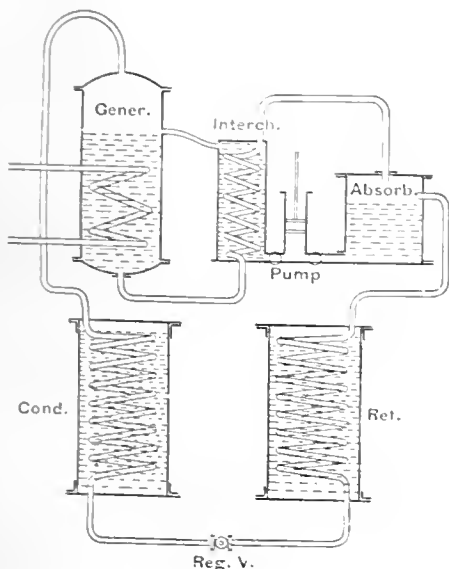


shaft at a certain level by raising all the in-flowing water to a height from which it may flow away. Thermo-dynamic reasoning shows that in a perfect engine—

$$AW = \frac{H}{T} (T' - T) \dots \dots \dots (1)$$

where  $A$  is the thermal equivalent of work,  $T$  the absolute temperature at which a certain quantity of heat  $H$  is abstracted, and  $T'$  the (higher) absolute temperature at

Fig. 3.

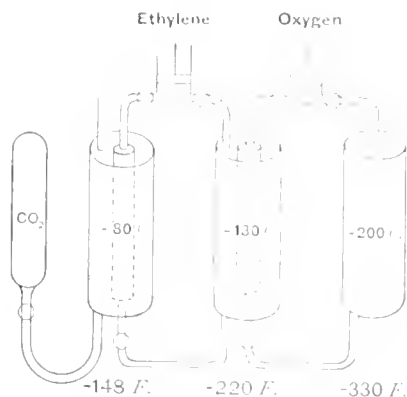


which a certain quantity of heat  $H$  is rejected. A working process will only enable us to attain a favourable efficiency provided that at no single point heat be abstracted at lower temperatures than those strictly determined by the given problem, and that the heat be not any higher than is absolutely necessary for its rejection by the cooling water.

Refrigerating machines may be classed in three groups. Fig. 3 exhibits the action of the first type, in which the reduction in temperature depends upon the expansion of air. The compressor draws in air from the freezing chamber and compresses it to about 3 atmospheres. The compressed air is passed through a cooler, where its temperature is reduced by cooling water. It is then admitted into an expansion cylinder, whence, after cooling by expansion, performing a certain amount of work, it re-enters the freezing chamber.

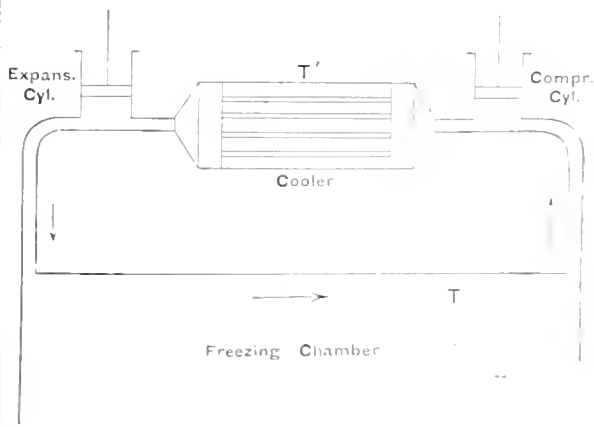
The second and third types of refrigerating machines depend upon the reduction of temperature by the evaporation of a volatile liquid, the return to the liquid state being effected either by compression or by absorption. Fig. 4 illustrates the action of a machine of the second type

Fig. 4.



(compression and evaporation). The vapour from a volatile liquid is aspirated by a compressor and forced into a condenser at a pressure corresponding to the temperature  $T'$ , at which the transfer of the latent heat to the cooling water is rendered possible. The condensed liquid returns by the regulating valve into the refrigerator. Such machines have been termed "cold steam-engines" by Zenner, as they represent simply the reversal of a steam-engine. For a perfect process the nature of the volatile liquid is quite immaterial, but in practice it is disadvantageous to employ volatile liquid possessing a critical point close to the temperature range within which the working process is performed. It has been proved that

Fig. 5.



when ammonia is used, as much as two-thirds of the heat determined by equation (1) can actually be abstracted under ordinary conditions of working.

The third type of refrigerating machine—the absorption machine—is shown in Fig. 5. Like the compression machine, it has a condenser and a refrigerator. The ammonia vapours are absorbed by a weak solution of ammonia in water; after this is saturated it is pumped into the generator, exchanging on its way its heat with that of the weak solution coming from the generator. The solution in the generator is then heated so as to drive off the ammonia, and the pressure of the vapours developed is sufficient to liquefy them in the condenser.—D. E. J.

*The Influence of Temperature on the Tensile Strength of Metals.* M. Rudeloff. Mitt. Königl. tech. Versuchs. 11 [6], 292.

See under X., page 520.

*Plant for Granulating Slag.* R. H. Terhune. School of Mines Quarterly, 15 [2], 108.

See under X., page 522.

#### PATENTS.

*Improved Combined Digester, Dryer, Washer, and Disinfecter, for Bones, Fat, Meat, Glue, and other Materials.* H. G. Warner, Wandsworth, Surrey. Eng. Pat. 6485, March 27, 1893.

AN upright boiler is provided with a steam-jacket, and fixed therein is a cylindrical perforated casing to contain the material to be treated; the boiler is also fitted with perforated ring pipes for spraying steam, hot water, or disinfecting fluid into the casing, while another pipe supplies hot air for drying. Manholes are provided for introducing and discharging the contents, and a shaft provided with vanes may be used for stirring up the contents. The apparatus may be used for digesting drying, washing, or disinfecting bones, meat, glue, clothes, or other substances.—E. G. C.

*Improvements in Apparatus for the Manufacture of Metallic Pigments or Compounds, and in the Production of Lead and Zinc Pigments or Compounds and Combinations of these, by means of such Apparatus.* A. C. J. Charlier, Glasgow. Eng. Pat. 7119, April 6, 1893.

See under XIII. A., page 532.

*Improvements in Separating Oleaginous or Fatty Matters from Boiler Feed-Water or other Water.* J. and G. Weir, Cathcart, Renfrew, North Britain. Eng. Pat. 10,664, May 31, 1893.

WITHIN a box-like casing, layers of an absorbent material are arranged, such as animal fibre, hair, sponge, or the like, with channels between each layer. The feed-water to be purified is introduced at a suitable point, and forced through tortuous channels, in which it gives up its fatty matters to the absorbent material, emerging in a purified condition. The fatty matters are removed from the absorbent material by squeezing between rollers, washing with a solvent, or by similar means. In a modified arrangement the water flows through an annular cylinder with curved guide blades, the inner surface of the annular cylinder being formed by a roll of flannel, backed by perforated sheet metal; the fatty matters are absorbed by the flannel and fall into the central

cavity of the cylinder, from which they are discharged in any suitable manner. Other modified arrangements are described and illustrated.—E. G. C.

*Improvements in Calcining Apparatus for the Conversion of Bicarbonate of Soda into Carbonate of Soda and other Uses.* La Société Marcheville Daguin and Company, Paris. (Under Internat. Convention, date claimed December 3, 1892.) Eng. Pat. 12,997, July 3, 1893.

See under VII., page 518.

*A Process for Preventing Incrustation in Steam Boilers.* V. Gane, Paris, France. Eng. Pat. 14,211, July 22, 1893.

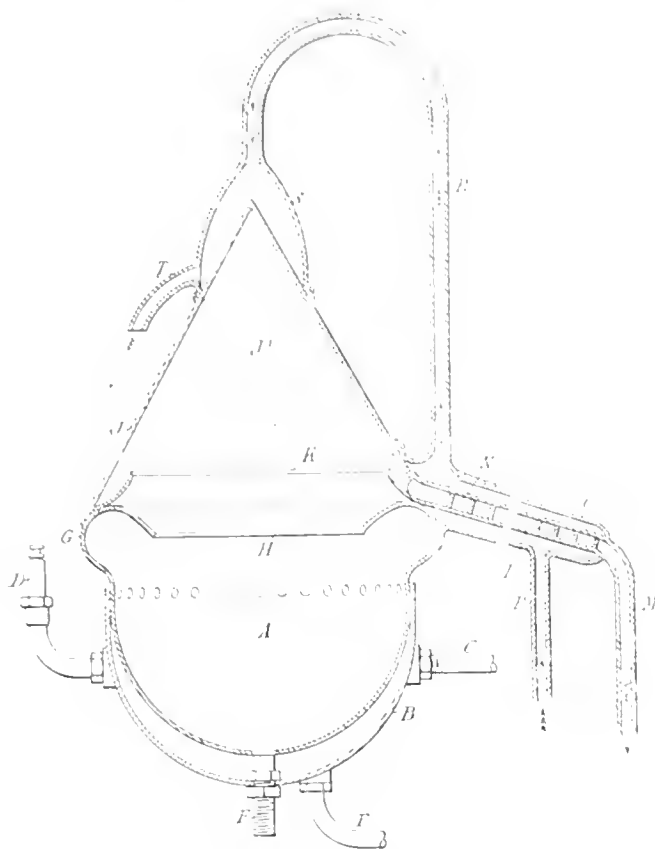
INCRUSTATION in boilers is prevented by causing the shell to absorb petroleum, shale oil, or other hydrocarbon under pressure in the following manner. The boiler is filled with petroleum and left for some hours under pressure, when, it is said, the petroleum penetrates into the iron or steel plate and incrustation is thereby prevented from adhering. To remove the incrustation from old boilers the petroleum is introduced from above and the boiler is slowly filled with water from below, thereby causing the lighter petroleum to slowly come into contact with the porous incrustation and penetrate it; the petroleum is finally expelled at a suitable outlet at the top for use over again. The absorption of petroleum by the incrustation causes the latter to scale and fall to the bottom of the boiler, from whence it is readily removed.—E. G. C.

*An Improved Still or Apparatus for Distilling Spirits and all kinds of other Liquids.* G. Morris and W. Edney, both of Bristol. Eng. Pat. 16,063, August 25, 1893.

THE figure (see next page) is a sectional elevation of the still, which consists of a hemispherical body A, bellied or swelled out at G, with a recessed lip H. Over this body is mounted a conical dome J, having an outlet L, containing baffle-plates N, to increase the surface area of the condensed liquid which flows through. The spirit or other liquid is introduced into the bowl A by means of the orifice J<sup>1</sup>, and heat is applied by means of a steam-jacket B or other suitable contrivance. The vapour is compelled to rise centrally by means of the curved lips H K, and impinges upon the dome J, which latter is cooled by means of the stream of water passing from the water-channel O R through the chamber S. The condensed liquid flows down the sides of the dome J, and passes away between the baffle-plates N in the worm L, being cooled by the stream of water passing in the reverse direction through the annular channel O. The distilled liquid finally escapes through the pipe M. The steam-jacket has a steam-inlet C, a safety-valve D, and a condensed water outlet E, while an outlet F is provided for cleansing. The steam-jacket may be dispensed with and other suitable modes of heating used, while in large pans two or more collecting domes J may be used.—E. G. C.

*Improvements in Valves for Tar, Ammoniacal Liquor, and other Liquids.* F. S. Cripps, Sutton, Surrey. Eng. Pat. 17,737, September 21, 1893.

See under III., page 508.



AN IMPROVED STILL.

*Improved Apparatus for Filtering or Purifying Feed-water for Marine or other Steam Boilers.* J. Wotherpoon, Forest Hill, Kent, and J. Davie, Wallsend-on-Tyne. Eng. Pat. 1910, January 29, 1894.

This specification claims "the combination of a filter-body made of an open-ended, parallel-sided formation, with a depending mud-collector adjacent to each end; a stationary perforated diaphragm, fitting within such filter-body and between the mud-collectors; filtering media arranged between such diaphragms; covers adapted to close the ends of the filter-body and to limit the outward position of such diaphragms," &c. The parts are forced together by means of a movable back-plate actuated by a hand-wheel and screw, so as to force the collected impurities into the mud- or sediment-collectors, from which they are discharged through mud-cocks. The water to be filtered is supplied to one end of the filter-chamber and forced through the layers of filtering media, issuing by a pipe which leads to the boiler.—E. G. C.

are: (1)  $C_2H_4 = C_2 + 2H_2$  when the decomposition takes place at a very high temperature; and (2)  $C_2H_4 = C + CH_4$  at a lower temperature. According to Day, at about 350° condensation without breaking up only occurs; whilst at 400°, after a sufficiently long time, entire decomposition is brought about, with formation of marsh-gas, ethane, and liquid products. The author prepared ethylene from rectified methylated spirit and strong sulphuric acid (25 parts to 150 respectively), and stored it over alkaline pyrogallate solution; so that the gas used contained 98·80 per cent. of ethylene and 1·20 of nitrogen. After heating for 100 hours to 400° C. the composition was as follows, a little air having obviously been admitted:—

Carbon dioxide, 0·82; free oxygen, nil; unsaturated hydrocarbons, 7·00; saturated hydrocarbons, 61·82; carbon monoxide, 1·17; hydrogen, 22·18; nitrogen, 7·01; total, 100·00.

In analysing such a mixture, after removal of  $CO_2$  by sodium hydrate and oxygen by alkaline pyrogallate, the unsaturated hydrocarbons are absorbed by a solution of bromine in potassium bromide, and the  $CO$  by acid cuprous chloride. The residue, on agitation with petroleum, is deprived of most of the saturated hydrocarbons, the residual portion thereof, chiefly marsh-gas, being determined by explosion and  $CO_2$  absorption (see this Journal, 1891, 107). If the saturated hydrocarbons be not treated with petroleum before explosion, the quantity of  $CO_2$  formed on combustion exceeds 100 per cent. of the combustible gas acted upon, showing that higher homologues of marsh gas, especially ethane, are present.

On passing ethylene through a platinum tube heated to from 600° to 1,500° C. (temperature ascertained by Le Chatelier's thermo-couple, platinum and platinum-

## II.—FUEL, GAS, AND LIGHT.

*The Action of Heat on Ethylene.* V. B. Lewes. Proc. Roy. Soc. 55, 90—107.

FROM the work of earlier observers the reactions accepted in the text-books as those occurring when ethylene is heated

rhodium), it was found that the decomposition produced varied with the temperature and rate of flow: thus, with a

particular tube and current of about 10 cc. per minute, the following figures were obtained:—

Temperature—centigrade.	600°	700°	800°	900°	1,000°	1,200°	1,500°
Percentage of ethylene in gas used.	96.78	96.73	96.78	94.8	94.8	98.91	98.91

#### ANALYSIS OF GAS AFTER HEATING: PER CENT.

Unsaturated hydrocarbons.	96.42	96.39	96.46	94.77	18.02	10.54	0.43
Containing acetylene..	0	0	Trace	0.82	0.60	3.60	0
Saturated hydrocarbons.	0	0	0	59.73	76.48	55.26	27.80
Hydrogen.....	0	0	0	0	0	25.11	62.68

#### CARBON DEPOSITED AND OIL FORMED IN GRMS. PER 100 CC.

Carbon.....	0	0	0	0	0	0.0273	Large quantity
Oil.....	0	0	0	0.0024	0.0048	0.0038	0
Change of volume.....	0	0	0	Decrease	Decrease	Increase	Large increase

When the rate of flow was reduced to less than one-half (4.2 cc. per minute), a higher member of the paraffin series, probably ethane, was formed at temperatures up to 900°, with but little methane; at 1,000° the quantity of the former diminished, and that of the latter increased; and at 1,200°, little but methane was formed; accordingly a series of observations was made by passing ethane through the hot tube, the gas being prepared from ethyl iodide by the copper-zinc couple in presence of water, and containing 96.38 per cent. of actual ethane. The following figures were obtained:—

Temperature.....	900°	1,000°	1,200°	1,500°
Unsaturated hydrocarbons produced.	31.00	28.42	11.58	1.69
Containing acetylene.....	Trace	0.30	1.80	0.91
Saturated hydrocarbons—				
Paraffin.....	12.82	8.34	3.88	0
Methane.....	12.01	12.73	21.83	20.62
Hydrogen.....	49.64	46.78	57.45	73.35
Carbon formed per 100 cc. of gas.	0	0	0.0126	0.0314
Oil formed per 100 cc. of gas.	0	Trace	Trace	0

Hence, even below 900° C., ethane decomposes with liberation of hydrogen and formation of unsaturated hydrocarbons, chiefly ethylene, which latter is decomposed as before at higher temperatures; accordingly, the text-book equation No. 2 (*already given*) is incorrect; carbon and hydrogen appear simultaneously, and are probably both derived from acetylene.

On passing acetylene through the tube heated to 1,000°, it was found that about a quarter remained unchanged, the remainder chiefly becoming polymerised to benzene; a little ethylene was formed, probably by direct combination of part of the acetylene with hydrogen liberated by the decomposition of another portion. On the other hand, on passing a mixture of 3 vols. hydrogen and 1 of ethylene through the tube at 1,250°, the quantity of acetylene produced was somewhat larger than when ethylene not mixed with hydrogen was used.

Methane was prepared from methyl iodide by means of the copper-zinc couple and water (gas contained 99.2 per

cent. of methane); on passing through the hot tube the following results were obtained:—

Temperature Centigrade ...	1,000°	1,200°	1,500°	1,500°
Unsaturated hydrocarbons produced.	Trace.	0.07	0.39	1.20
Containing acetylene.....	Trace.	0.07	0.39	0.96
Saturated hydrocarbons....	37.65	90.00	88.52	19.22
Hydrogen.....	1.55	8.53	10.37	78.66
Carbon deposited per 100 cc. of gas.	0	0	Trace.	0.015
Oil formed per 100 cc. of gas.	0.0023	0.0025	0.0005	0

Hence methane is much more stable under the influence of heat than any of the other gaseous hydrocarbons examined; it probably decomposes first into acetylene and hydrogen, thus:—



and then the acetylene either polymerises or decomposes into carbon and hydrogen, according to the temperature. These results explain why the flame of methane, when burning at an open tube, is practically non-luminous, the maximum temperature of the flame being below 1,100° C., so that no acetylene is formed; whilst, when burnt in a regenerative burner at 1,500°, the light emitted is of considerable illuminating value.

A mixture of hydrogen and benzene vapour (5.28 per cent. benzene) gave the following results on passing through the tube:—

Temperature Centigrade.....	900°	1,100°	1,300°
Unsaturated hydrocarbons produced	5.00	3.33	2.43
Containing acetylene.....	0	Trace	0.083
Saturated hydrocarbons.....	0	2.87	5.62
Hydrogen.....	95.00	93.80	92.47
Carbon deposited per 100 cc. of gas	0	Trace	0.01
Oil deposited per 100 cc. of gas....	Trace	0.012	0

Hence the diluted benzene breaks down to acetylene, methane, and carbon, and finally to carbon and hydrogen.

On the whole, the conclusion drawn from the various series of experiments is that the primary action of heat upon ethylene is represented by the equation



whilst the final decomposition is, as represented by previous observers, —



In between, a variety of reactions occur, owing to the polymerisation of the acetylene thus formed from the ethylene, and also at higher temperatures from the methane, according to the equation—



—C. R. A. W.

#### PATENT.

*A Process and Appliance for Electrically treating Gaseous and other Bodies, a New Compound Gas made by such Process, and Apparatus for Producing and Consuming said Gaseous Product.* T. G. Hall, Chicago, county of Cook, U.S.A. Eng. Pat. 6551, March 23, 1893.

See under XI., page 528.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*The Sulphur Compounds in Ohio Petroleum.* Charles F. Mabery and Albert W. Smith. Amer. Chem. Jour. 16 [2], 83—89.

In this paper the authors respond to the adverse criticism of Kast and Lagai (Dingler's polyt. Journal, 284, 69; this Journal, 1892, 598), upon their former work (Amer. Chem. Jour. 13, 232; this Journal, 1891, 629, and 1890, 275).

The authors continue to have reliance in the Carius method of determining sulphur if thoughtfully carried out, sealed tubes being employed; and certainly they prefer it to that employed by Kast and Lagai, involving, as this latter is said to do, an oxidation of explosively-decomposable sulphur compounds with fuming nitric acid and potassium chlorate in an open dish. Further to test the Carius' method, analyses of pure ethyl sulphide have been made by it and S determined, 35.46 and 35.48, against calculated quantity, 35.55. Again, a crude burning oil distillate gave by Carius' method 0.46 per cent. S, while, by ignition with soda-lime, 0.40 per cent. was obtained.

With regard to the lead and calcium salts obtainable by neutralising the sulphuric acid solution of the sulphur-containing bodies, which is produced in commercial refining, it is admitted by both sets of observers that these are not salts of sulpho acids derived from the paraffin alkyl sulphides dissolved in the acid; but that, whilst these latter are present and are obtainable by dilution of the concentrated acid liquor, there are other sulphur-containing bodies in sulphur-petroleum which are capable of forming salts.

Messrs. Kast and Lagai were unable to obtain the definite crystalline compounds with mercuric chloride; the authors now describe at some length two of them, derived from the "crude naphtha distillate" of Findlay crude petroleum and from the "crude burning oil distillate" of Lima petroleum respectively.

The crystalline bodies are obtained by precipitation of the oil with aqueous solution of mercuric chloride, filtration, washing with gasoline, pressing, and drying in the air. They are decomposed by treatment with alcoholic solution of sodium sulphide or by hydrogen sulphide, yielding oils

(the distillation of which is described) containing 49.72 per cent. and 47.68 per cent. of sulphur respectively. From 800 grms. of Findlay distillate 70 grms. of the mercury compound were obtained.

The authors do not agree with Kast and Lagai that the odour of the crude oils is due solely to unsaturated hydrocarbons; on the contrary, they consider they have afforded grounds—by the separation of "several classes" of odorous substances, not otherwise specified, however—for the statements that it is due to "a mixture of bad-smelling compounds." They state that, while the crude distillates may be completely deodorised by mercuric chloride, only about two-thirds of the S-containing bodies are so removed in the case of Ohio petroleum, and about two-fifths in the case of a distillate from Canadian petroleum. Kast and Lagai state that sulphur oils may be deodorised by sulphuric acid, one-quarter only of the sulphur-containing bodies being removed, and this result appears to be confirmed by the author, as to the amount of the bodies removed, though the deodorisation was not quite so perfect.

It is noteworthy that the Ohio oil used by Kast and Lagai was obtained in Bremen and contained 1 per cent. of sulphur; the authors state that Ohio oil never contains more than 0.6 per cent., and quote in their support Prof. Orton and Lord (U.S. Geological Survey, eighth annual report), who found 0.553 per cent. S in crude Trenton limestone oil; other analysts, who report about 0.5 per cent.; and the experience of commercial manufacture, which indicates on the average 0.55 per cent.

Canadian sulphur petroleum contains more sulphur than do the Ohio petroleum.—L. R. B.

*Preliminary Examination of Canadian Sulphur-Petroleum.* Charles F. Mabery. Amer. Chem. Jour. 16 [2], 89-93.

THE author is engaged upon a comprehensive study of this subject, but publishes the pre-ent preliminary notice, as he has found that Kast and Lagai are also working in the same field.

The material investigated was the "sludge acid" obtained in refining the burning oil distillate of a crude Canadian petroleum. This crude oil had a specific gravity of 0.86 at 20°, and contained 0.98 per cent. of sulphur, exclusive of a further quantity present as hydrogen sulphide. Upon distillation, under a tension of 250 mm., 11 per cent. by weight was collected below 150°, 24 per cent. between 150° and 300°, and the residue amounted to 65 per cent. These three fractions contained respectively 0.195 per cent., 0.64 per cent., and 0.98 per cent. of sulphur.

The "sulphur oil" held in solution in the "sludge acid" was precipitated by the addition of the acid to water, and, after washing, measured half the bulk of the "sludge acid" used. It had a specific gravity of 0.86 at 15°, was dark brown in colour, and contained 9.94 per cent. of S. Distillation with steam (unattended by serious decomposition) gave a distillate with a powerful odour, containing 5.46 per cent. of S, while the residue contained 10.41 per cent. The distillate redistilled below 200° and had sp. gr. 0.82 at 20°. Further fractionation was attended with great difficulty owing to the peculiar persistency with which water was retained, and in some cases decomposition with separation of sulphur, occurred. However, the steam distillate was redistilled six times (tension = 150 mm.); the fractions, the weights of which are not given, contained sulphur as follows: fraction, 32—40° (0.13 per cent.); 40—70° (0.65 per cent.); 110°—120° (11.51 per cent.); 120—130° (13.94 per cent.); 140°—150° (13.44 per cent.); 160—170° (13.93 per cent.). Each fraction reacted with bromine, leaving no residual oil. The lower fractions were united with hydrobromic acid and polymerised upon standing, in one case noted, with rise in temperature of 150°. The fractions are regarded as composed of unsaturated hydrocarbons, and the  $C_{12}H_{22}$  hydrocarbons are considered to be absent.

The residue of the steam distillation was similarly fractionated, but the sulphur contents of only the 65—72° and 96°—97° fractions were recorded, being 9.12 per cent. and 12.96 per cent. respectively. All the fractions gave

crystalline or viscous precipitates with mercuric chloride, leaving oils with intensely penetrating odours.

The author has now resumed the investigation upon a much larger scale, but finds that the "sulphur oil" he now obtains, unlike the parallel product he formerly obtained from the refinery, undergoes decomposition during steam-distillation, with production of hydrogen sulphide, sulphur, sulphuric and sulphurous acids. Decomposition is also induced by the presence of air. He now therefore dries the "sulphur oil" as far as possible, by letting it stand for a week over recently-fused calcium chloride, and then distils (under tension of 15 mm.) in a porcelain still and gets but little decomposition.

It is interesting to note that, as in the former case, great difficulty is experienced in separating water from the oil: after long standing, 15 litres still contain about 10 cc. of water.

With the large quantities of material now being worked the author expects to be able definitely to isolate all the various odorous bodies which have been met with.

—E. R. B.

#### PATENTS.

*Improvements in obtaining Sulphonic Acids and Sulphone-like Compounds, Free from Inorganic Salts, from the Neutralised Product of the Action of Sulphuric Acid upon Mineral and similar Oils, and particularly Hydrocarbons, Artificially or Naturally Sulphuretted.* L. O. Helmers, Hamburg. Eng. Pat. 6044, March 21, 1893.

THE methods in use for purifying the sulphonated compounds produced as above are unsatisfactory, that consisting in dissolving the sulphonised product in water and separating by the addition of a salt (Eng. Pat. 10,523, July 24, 1884) yielding an impure result, while dialysis entails a loss of the sulphonic acids or their salts. The improved methods described consist in the employment of alcohol and chloroform, singly or together, as solvents. If the neutralised sulphenised product, freed as much as possible from water, be treated with strong alcohol (ethylic or other alcohols), the sulphonic acid salts will be dissolved out, leaving a mixture of sulphones and sulphone-like bodies and inorganic salts, from which the last can be extracted by washing with water. The alcoholic extract will contain small quantities of sulphone-like bodies, which treatment with chloroform will remove. The alternative method of purification consists in treating the neutralised sulphonised product with a mixture of alcohol and chloroform (or ether, petroleum, naphtha, &c.), which dissolves out everything but the inorganic salts. By evaporating off the solvent and treating the residue with chloroform, the sulphonic bodies will be extracted, or treatment with alcohol will dissolve out the sulphonic acid compounds.—J. G. W.

*Improvements in Valves for Tar, Ammoniacal Liquor, and other Liquids.* F. S. Cripps, Sutton, Surrey. Eng. Pat. 17,737, September 21, 1893.

THE valve is of the ordinary rectangular slide type, working in a centre casting provided with two side flanges. These latter flanges are bolted on to the adjacent pipe flanges, having the slide casting between them, the facing strips being surfaced to form sound joints when the centre casting and side flanges are bolted together. The valve is actuated by a screw and can be locked in any position by means of lock nuts.—E. G. C.

#### IV.—COLOURING MATTERS AND DYES.

*A Japanese Ponceau-Red Dye.* Scheurer. Chem. Ind. 17 [2], 39.

THIS ponceau-red appears to consist of the crocin obtained by the decomposition of the glucoside of the saffron, at all events a red material is obtained, which in China, by some unknown method, is prepared from saffron blossoms. This raw material is dyed in an acid bath upon hemp, and then the colouring matter thus fixed is again abstracted by means of potash solutions. Probably by acidifying this solution, the Japanese red is then obtained ready for use as a paste. It is interesting to note that the weak affinity for vegetable fibres probably containing tannin, is thus made use of for the isolation and purification of the dyestuff.

—W. S.

*Diazo Compounds and Nitrosamines.* C. Schraube and C. Schmidt. Ber. 27, 514—523.

THE fact that, by the action of diazobenzene chloride upon ketones and aldehydes, hydrazones are produced, first led various investigators to the hypothesis of the capacity of diazobenzene to react in the isomeric nitrosamine form,  $C_6H_5.NH.NO$ , as well as in the diazo form,  $C_6H_5N:N.OH$ , yielding hydrazine compounds in the former case and azo compounds in the latter.

The authors have succeeded in proving not only the existence of the nitrosamine form of diazobenzene, but also in obtaining phenylnitrosamine in the shape of its stable alkaline salts. If para-nitrobenzene chloride is added to a dilute solution of caustic soda, an excess of the latter being maintained, a clear solution is obtained, without evolution of nitrogen, in which the presence of the diazo compound can no longer be shown. After a time crystals separate from this solution, especially after the addition of common salt. These crystals were found to consist of the sodium salt of para-nitrophenylnitrosamine. The free nitrosamine is separated as a sparingly soluble crystalline precipitate by the addition of an acid to the cold alkaline solution.

The supposition that the diazobenzene potassium compound first described by Griess might also be a nitrosamine derivative was, by experiment, proved to be unfounded, for it gives at once, like diazobenzene chloride, a colour reaction when brought into contact with phenols under the necessary conditions, whilst the nitrosamines do not show this property. The conversion of the diazo compound into the nitrosamine may, however, be accomplished by heating the alkaline mixture above  $100^\circ$ . The nitrosamine then separates.

Owing to the extraordinary tendency of para-nitrodiazobenzene to be at once converted into the nitrosamine, even with the most dilute solutions of alkali and at a temperature of  $16^\circ$  the potassium compound of that body could not be obtained. Another great difference exhibits itself in the behaviour of the two nitrosamine salts on the addition of an acid to their solutions. While para-nitrophenylnitrosamine is separated as a fairly stable compound, phenylnitrosamine does not appear to exist in its free state. Even with the greatest care reconversion into diazobenzene takes place immediately, and can instantaneously be completed by supersaturation with acetic acid.

A mixture of the alkaline solutions of para-nitrophenylnitrosamine and  $\beta$ -naphthol gradually produces a precipitate of a dyestuff on being warmed, or even on being allowed to stand sufficiently long in a closed vessel. When spread on paper and thus exposed to the action of the carbonic acid of the atmosphere, the formation of colouring matter from a freshly prepared mixture takes place very slowly. Phenylnitrosamine under the same conditions at once forms a colouring matter in consequence of the action of the carbonic acid, whilst no colour reaction takes place either on heating or continued standing. These facts, together with the negative results of experiments made with a view of combining phenylnitrosamine sodium in an alkaline

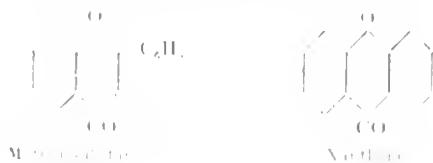
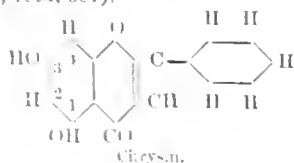
solution with acetone, acetoacetic acid, or acetone diacetic acid, are inconsistent with the theory of phenylhydrazine being an intermediary product in the formation of the hydrazones. —F. M.

*A Colouring Matter occurring in the Leaves of the Grape-Vine.* E. Schunck, E. Knecht, and L. Marchlewski. Ber. 27, 187-188.

THE green portions of plants contain besides chlorophyll as a rule only a yellow colouring matter, called carotin, chrysophyll, or erythrophyll, which is insoluble in water. Several investigators find, however, that some kinds of leaves give aqueous extracts of a more or less impure yellow colour, an observation which is explicable from the fact that in most of these instances mature leaves were used. Young leaves yield an almost colourless extract. Yellow autumn leaves, however, contain considerable quantities of soluble colouring matters. Thus the authors found that fallen beech and horse-chestnut leaves give deeply coloured aqueous extracts. They have also succeeded in isolating a yellow colouring matter from vine leaves, the investigation being suggested by the use of these leaves for dyeing purposes in Persia. Like most vegetable coloring matters, this substance is a glucoside. It can be prepared by the addition of lead acetate to the decoction of the finely-powdered leaves, treatment of the precipitate formed with sulphuretted hydrogen, and subsequent extraction of the dried lead sulphide with boiling alcohol. The residue, obtained by evaporation of the alcohol, is freed from sulphur by means of carbon bisulphide, the glucoside remaining as an indistinctly crystalline brownish-yellow substance. By boiling with dilute sulphuric acid it is split up into a sparingly soluble brown body and glucose. This colouring matter may, after washing with water, be purified by adding to its alcoholic solution an alcoholic solution of lead acetate and treating the previously washed and dried bluish-green precipitate with ether containing hydrochloric acid, by which the impurities are taken up. The remaining colouring matter is then dissolved in alcohol and precipitated from this solution by the addition of water. It forms a reddish-brown powder, soluble in alkalis with a brown colour. Its aqueous solution produces upon chrome-mordanted wool fine brown shades, and dyes wool mordanted with tin a fine yellow. The colouring matter may possibly be of practical value. The vine leaves were also found to contain up to 2 per cent. of potassium hydrogen tartrate. —F. M.

*Chrysin.* St. v. Kostanecki. Ber. 26, 2901-2905.

PORTULACA buds contain two yellow colouring matters, chrysin and teetochrysin. The latter is the methyl ether of the former. Piccard, the discoverer of these substances, found that chrysin possesses the formula  $C_{15}H_{10}O_2$ , and is by alkalis split up into phloroglucinol, benzoic acid, and acetic acid, a small quantity of acetophenone being formed at the same time. Chrysin forms a diacetyl derivative, and therefore must contain two free hydroxyl groups. Only one of these, however, can be replaced by methoxyl. Sodium compounds of the alkyl ethers of chrysin can be obtained by carefully adding caustic soda to their alcoholic solutions until a slight turbidity ensues. These compounds are almost insoluble even in hot water, but are split up by it into their constituents. The methyl ether forms the acetyl compound  $C_{15}H_{10}O_2 \cdot (OCH_3) \cdot (OC(=O)CH_3)$ , which crystallises from alcohol in bright, colourless needles, melting at 149°. The formula which the author arrives at as the one best representing the constitution of chrysin, shows its close relationship to the hydroxy-xanthenes, chrysin being a phenylated pheno- $\gamma$ -pyrone, and xanthone a diphen- $\gamma$ -pyrone. Fisetin and quercetin may be regarded as hydroxy-derivatives of the mother-substance of chrysin (this Journal, 1894, 381).

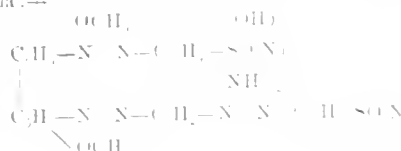


F. M.

## PATENTS.

*The Manufacture or Production of Dyestuffs.* H. E. Newton, London. From "The Farbentabrikeu vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 7339, April 10, 1893.

IN Eng. Pat. 13,113 of 1890 (this Journal, 1891, 760) mixed tetrazo dyestuffs are produced by combining the tetrazo derivative of a diamine with one molecular proportion of 1-1' amidonaphthol 3-3' disulphonic acid, and one molecular proportion of an amine, a phenol, naphthol, dihydroxynaphthalene or one of their sulphonic or carboxylic acids. The term amine would appear to include not only monamines of the benzene and naphthalene series, but also diamines and their sulphonic, carboxylic, or alkylated derivatives. The order in which the combinations are performed may be reversed, that is, the tetrazo compound may be first combined with the amine or phenol or a derivative thereof, and afterwards with the amidonaphthol disulphonic acid. It has now been discovered that these mixed tetrazo dyestuffs are capable of combining with another molecular proportion of a diazo or diazo-azo compound, or with half a molecular proportion of a tetrazo compound, or with one molecular proportion of an intermediate product resulting from the combination of equal molecular proportions of a tetrazo derivative of benzidine, its analogues and homologues and an amine, diamine, phenol, amidophenol, or amidophenol ether of the benzene or naphthalene series or a carboxylic or sulphonic acid thereof. A further extension of the original patent is that the amidonaphthol disulphonic acid can be replaced by other amidonaphthol sulphonic acids, such as amidonaphthol sulphonic acid G or B or the like, or by 1-3' or 1-2' naphthylamine sulphonic acid, amidonaphthol, amidonaphthol ethers, amidonaphthol ether sulphonic acids, amidonaphthoxy-acetic acids, amidonaphthoxy-acetic sulphonic acids, dihydroxynaphthalenes, or sulphonic derivatives of any of these components. The combinations obtained by any of the foregoing methods may, when insoluble, be sulphonated and thus rendered useful for dyeing purposes. Furthermore, those dyestuffs which contain diazotisable groups may be diazotised on the fibre and combined with amines, phenols, and their derivatives. The process for obtaining colouring matters described in Eng. Pat. 15,525 of 1891 (this Journal, 1892, 741), by combining one molecular proportion of a tetrazo compound with one molecular proportion of amidonaphthol disulphonic acid H, and one molecular proportion of *m*-tolylene diamine, chrysoidine, or Bismarck brown is disclaimed. The following example illustrates the general method employed in carrying out this invention. The intermediate product obtained from the tetrazo derivative from 24.4 kilos. of dianisidine and 26.2 kilos. of 1-1'-dihydroxynaphthalene sodium sulphamate is combined in an acid solution in presence of sodium acetate with 10.8 kilos. of *m*-phenylene diamine. The diazo compound from 30.3 kilos. of  $\beta$ -naphthylamine 3-3' disulphonic acid is then stirred into the liquid. After some time sodium carbonate is added and the mixture is heated, after which the dyestuff is salted out, filter-pressed, and dried. It has the following formula:—

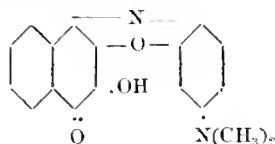


—T. A. I.



*The Manufacture or Production of Dyestuffs.* II. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 8093, April 21, 1893.

This is an extension of Eng. Pats. 4757 and 6035 of 1893 (this Journal, 1894, 245 and 383). The alkylated *p*-phenylenediamine thiosulphonic acids and their derivatives obtained by the action of thiosulphates on the nitroso derivatives of secondary or tertiary amines, yield thiazine colouring matters when oxidised together with amidonaphthols and amidonaphthol sulphonic acids (corresponding to the *o*-nitrosonaphthols and *o*-nitrosonaphthol sulphonic acids of Eng. Pat. 6035 of 1893), and the corresponding hydroquinones and their sulphonic acids are capable of interacting in the same manner. A mixture of 25 kilos. of dimethyl-*p*-phenylene diamine thiosulphonic acid and 28 kilos. of 1-2-3'-dihydroxynaphthalene potassium sulphate is suspended in about 800 litres of water and mixed with 200 kilos. of 25 per cent. sodium hydroxide. The brownish-coloured liquid produced is heated on the water-bath and air passed through for several hours, after which it is made acid with acetic acid, when the neutral sodium salt of the dyestuff is precipitated. The dyestuff is identical with that obtained from dimethyl-*p*-phenylene diamine thiosulphonic acid and potassium  $\beta$ -naphthoquinone sulphate, and the same product is also obtained if, in place of this product, the 1-amido-2-naphthol-3'-sulphonic acid be employed. When the products obtained are sparingly soluble they may be sulphonated by treatment with fuming sulphuric acid or other suitable sulphonating agents. The specification further describes certain colouring matters of the oxazine type, of which that obtained from nitrosodimethyl-*m*-amidophenol and  $\beta$ -naphthoquinone is a typical example. It has the following constitution:—

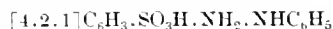


The product dissolves with difficulty in water, and is rendered more suitable for dyeing and printing purposes by sulphonation. A sulphonated product is obtained directly by adding a concentrated solution of 28 kilos. of potassium 1-2-3'-dihydroxynaphthalene sulphate and 25 kilos. of crystallised sodium acetate to a hot solution of 23 kilos. of nitrosodiethyl-*m*-amidophenol in 700 litres of water. The liquid is then boiled and stirred continuously until a sample on cooling shows no further increase in the amount of dyestuff deposited. The whole is then allowed to cool, when the colouring matter separates out in yellowish crystals with a greenish reflex. A further quantity can be precipitated from the mother-liquor by adding salt. The colouring matter dissolves easily in hot water, and gives bright blue shades on chrome-mordanted wool fast to light and fulling. In place of the dihydroxynaphthalene sulphonic acid, the corresponding amidonaphthol sulphonic acid may be employed, the resulting product being the same, as in this case ammonia is liberated during the condensation.—T. A. L.

*The Manufacture of New Sulpho Acids of Phenyl-rosinduline and Analogues and Homologues thereof.* J. Y. Johnson, London. From "The Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 8184, April 22, 1893.

Certain of the sulphonic acids of phenyl-rosinduline, as described in Eng. Pat. 15,259 of 1888 (this Journal, 1889, 877), are known in commerce under the name azo-carmines, and further discoveries with regard to these dyes are mentioned in Eng. Pats. 6875, 14,836, and 17,168 of 1890 (this Journal, 1891, 459, 830, and 831). The present invention describes the manufacture of isomers of azo-carmines which dye animal fibres red from an acid bath in shades different from that of azo-carmines. The process employed

is similar to that mentioned in Eng. Pat. 10,138 of 1892 (this Journal, 1893, 593) and more particularly referred to in Eng. Pat. 10,138n of 1892 (this Journal, 1893, 594). The methods there employed are the condensation of hydroxynaphthoquinone anil or its sulphonic acid with diamines and alkylated diamines. The new colouring matters described in the present specification are obtained by condensing sulphonated phenylene-*o*-diamines with hydroxynaphthoquinone anil or with its sulphonic acid. In the former case if phenyl-*o*-phenylene diamine sulphonic acid be employed, the product obtained can be converted into a soluble disulphonic acid by treatment with concentrated sulphuric acid, and the dyestuff so obtained is known as "iso-azo-carmines I." The colouring matter obtained by condensing phenyl-*o*-phenylene diamine sulphonic acid (*o*-amido-diphenylamine-*p*-sulphonic acid) with hydroxynaphthoquinone-anil-*p*-sulphonic acid is at once a disulphonic acid, and is known as "iso-azo-carmines II." Analogues and homologues of these substances may also be obtained by using, for instance, instead of the hydroxynaphthoquinone anil, the corresponding *o*- or *p*-toluides. In a similar manner the phenyl-*o*-phenylene diamine sulphonic acid may be replaced by the *o*- or *p*-tolyl-*o*-phenylene diamine sulphonic acid. The following proportions are given for preparing iso-azo-carmines I:—About 11 kilos. of *o*-amido-diphenylamine-*p*-sulphonic acid—



and 10 kilos. of  $\beta$ -hydroxynaphthoquinone anil are mixed with 200 kilos. of alcohol and 100 litres of water and boiled, when the new induline sulphonic acid separates out as a heavy precipitate. When a hot, faintly alkaline solution of the precipitate is completely precipitated by caustic soda and the mother-liquor is not coloured yellowish-red, the reaction is complete. The new phenyl-rosinduline sulphonic acid differs from its isomeride described in Eng. Pat. 15,259 of 1888 by giving alkaline salts soluble in hot and cold water. From the corresponding acids of Eng. Pat. 6875 of 1890 it can best be distinguished by its behaviour to sulphuric acid. About 10 kilos. of the phenyl-rosinduline sulphonic acid described above are stirred into 40 kilos. of concentrated sulphuric acid and heated to 100° C. until a test is soluble in 50 times its bulk of water. The melt is then poured on to 400 kilos. of ice and filter-pressed. After dissolving the press-cakes in boiling water an alkali is added and the dyestuff is precipitated by adding salt. Iso-azo-carmines I, thus obtained as an alkaline salt, is easily soluble in water, and dyes wool from an acid bath in bluer shades of red than the disulphonic acid of Eng. Pat. 15,259 of 1888. It differs from the disulphonic acids of Eng. Pat. 6875 of 1890, in that the colour of the solution of its sodium salt remains unchanged whether it is acid or alkaline. The new acid closely resembles or is practically identical with the sulphonic acid B of Eng. Pat. 17,168 of 1890. For the preparation of iso-azo-carmines II, about 10 kilos. of hydroxynaphthoquinone-anil sulphonic acid, and 8.5 kilos. of *o*-amido-diphenylamine-*p*-sulphonic acid are boiled with 100 litres of water. When the reaction is complete, caustic soda solution (containing about 35 per cent. NaOH) is added to the hot solution so long as a precipitate is produced. After cooling, the colouring matter is filtered off and purified by dissolving in water and reprecipitating with caustic soda. It very closely resembles in its properties iso-azo-carmines I, and the homologous colouring matters possess also very similar properties.—T. A. L.

*Improvements in the Manufacture or Production of Basic Compounds derived from Pyrazine.* H. E. Newton, London. From The Farbenfabriken vormals F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 8824, May 2, 1893.

The inventors find that the distillates obtained by heating glycerol with any ammonium salt, contain only homologues of pyrazine mixed with water, ammonia, and substances possessing an aldehyde-like odour, 2,5-dimethylpyrazine being formed as the chief product, with only a small proportion of dimethylethylpyrazine. To prepare and

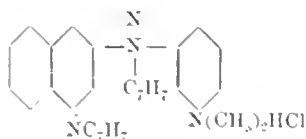
separate these two pyrazines the inventors acidulate the above distillate and distil with steam in order to separate the non-basic substances. The bases are removed by making alkaline and distilling, and solid caustic soda is then added to the distillate. The pyrazine bases are insoluble in the resulting concentrated alkaline solution and rise as an oily mass, from which the two bases are separated by distillation.

The 2,5-dimethylpyrazine boils at 155° C.

The second pyrazine obtained as above has the formula  $C_{11}H_8N_2$  and is 2:5-dimethyl-3-ethylpyrazine. It is a highly refractive liquid, boiling at 178–179° C.—J. G. W.

*The Manufacture or Production of Colouring Matters and Materials therefor.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer und Co.," Elberfeld, Germany. Eng. Pat. 8898, May 3, 1893.

THE first part of this specification refers to the preparation of substituted naphthalene derivatives by heating certain  $\alpha$ -naphthylamine sulphonic acids with a primary aromatic amine. In Eng. Pat. 7337A of 1892 (this Journal, 1893, 350) the method was described for obtaining alkylated naphthylamine sulphonic acids by heating certain naphthylamine sulphonic acids with aniline, *o*-, or *p*-toluidine with or without a salt thereof and in presence of benzoic acid. According to the present invention if the  $\alpha$ -naphthylamine sulphonic acid contains a sulphy group meta to the amido group, it also is replaced by the amine rest and a substituted *m*-naphthylendiamine results. For instance,  $\alpha$ -naphthylamine-*c*-disulphonic acid [1.3.1']  $C_{10}H_7NH_2(SO_3H)_2$  when heated with aniline and aniline hydrochloride is converted into diphenylnaphthylene diamine sulphonic acid [1.3.1']  $C_{10}H_7NHC_6H_5.NHC_6H_5.SO_3H$ . Similarly 1.3-naphthylamine sulphonic acid (Clève's  $\gamma$ -acid) when heated with *p*-toluidine and *p*-toluidine hydrochloride to 120–135° C. is converted into *p*-tolynaphthylamine sulphonic acid as described in Eng. Pat. 7337A of 1892, but if the temperature be raised to 140° C. sulphur dioxide is given off and 1.3-di-*p*-tolyl-naphthylene diamine is formed. By performing the operation in two stages and using different amines, mixed alkylated products can be obtained. All these products are to be employed for condensations with the nitroso derivatives of primary and secondary amines, forming rosinduline colouring matters and not isorosindulines as obtained according to Eng. Pat. 14,283 of 1886 and Eng. Pat. 3098 of 1890 (this Journal, 1891, 132). By condensing ditolyl-naphthylene diamine with nitro-*o*-dimethylaniline hydrochloride in an alcoholic solution a compound of the following constitution is obtained:—



This gives bluish-violet shades on cotton mordanted with tannic acid. On sulphonation with fuming sulphuric acid it yields a valuable sulphonic acid which dyes chromed wool a blue or greenish-blue shade, fast to soap and washing. Sulphonic acids of these dyestuffs can also be obtained by using sulphonic acids as original components for condensation, as, for example, a blue for wool is produced by condensing 1.3-di-*p*-tolyl-naphthylene diamine-1'-sulphonic acid with nitroso-ethyl-benzylaniline sulphonic acid in an alcoholic solution in presence of sodium acetate. The dyestuff obtained stands fulling and alkalis.—T. A. L.

*The Production of New Colouring Matters for Dyeing and Printing* Read Holliday and Sons, Ltd., and L. Holliday, Huddersfield. Eng. Pat. 9689, May 15, 1893.

By heating rosaniline with  $\beta$ -naphthylamine, Meldola (Chem. News, 1883, 1, 133) obtained some naphthylated rosanilines which on sulphonation dyed wool and silk redder shades of blue than the corresponding sulphonic acid of

triphenylrosaniline. By varying the conditions of the melt it is possible to obtain grimmer shades than from the phenylated rosanilines, and the proportions given are as follows:—100 lb. of rosaniline, 500 lb. of  $\beta$ -naphthylamine, and 5 lb. of benzene or glacial acetic acid are heated to 160° C. until a sample dissolved in alcohol and acetic acid, and spotted on paper, appears greenish blue by gas light. The time required is about 5 hours, when the melt is cooled and the excess of  $\beta$ -naphthylamine removed by hot hydrochloric acid and water. In order to sulphonate it, 100 lb. of the crude product are stirred into 300 lb. of sulphuric acid of 60° B. without the application of external heat until a sample washed with cold water dissolves in dilute ammonia. The melt is then poured into water, washed, and dried down with a slight excess of ammonia. By stopping the sulphonation at an earlier or carrying it on to a later stage other products can be obtained which can also be used in dyeing and printing.—T. A. L.

*The Manufacture or Production of Azo Colouring Matters.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer und Co.," Elberfeld, Germany. Eng. Pat. 9894, May 17, 1893.

THE colouring matters described are obtained by reacting with two molecular proportions of the same or of different diazo compounds on one molecular proportion of 1:1'-amido-naphthol 4-sulphonic acid obtained by the action of alkalis on  $\alpha$ -naphthylamine disulphonic acid 8. The products obtained dye blue to bluish-black shades, and are said to be fast to light.—T. A. L.

*Manufacture of Basic Colouring Matters.* C. D. Abel, London. From "The Actien Gesellschaft für Anilin Fabrikation," Berlin. Eng. Pat. 9069, May 18, 1893.

VIOLET-BLUE to blue colouring matters are produced by oxidising the unsymmetrically mono- or di-alkylated *p*-phenylene diamines or their salts, together with diphenyl-*m*-phenylene diamine or its homologues. The alcoholic solution of dimethyl-*p*-phenylene diamine hydrochloride obtained from 2.5 kilos. of nitrosodimethylaniline hydrochloride is mixed with 2.9 kilos. of di-*p*-tolyl-*m*-phenylene diamine. After heating to about 70° C. a strong solution of 3.75 kilos. of sodium bichromate is added. The mixture is boiled for 1 hour, and 100 litres of water and 30 litres of brine are then added, when the colouring matter precipitates on cooling. In order to purify it, it is dissolved in boiling water, filtered from the oxide of chromium, and precipitated with salt.—T. A. L.

*The Manufacture or Production of Colouring Matters and Processes for Dyeing and Printing therewith.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer und Co.," Elberfeld, Germany. Eng. Pat. 9,972, May 18, 1893.

THE specification refers to the production of mixed tetrazo-dyestuffs by combining one molecular proportion of tetrazo-diphenyl or one of its analogues with one molecular proportion of a naphthylamine sulphonic acid, naphthol sulphonic acid, amidonaphthol sulphonic acid, or di-hydroxy-naphthalene sulphonic acid, and subsequently with one molecular proportion of aniline, *o*-toluidine, *p*-xylylene, *o*- or *m*-amido-phenol ether, *o*- or *m*-amido-*p*-cresol ether, or the order of these operations may be reversed. The colouring matters dye unmordanted cotton red to reddish violet and violet, are capable of being diazotised on the fibre, and on development give shades varying from reddish-violet and violet to blue, fast to washing.—T. A. L.

*Improvements in Inks.* E. Nicolson, and L. Goldmark, New York, U.S.A. Eng. Pat. 3276, February 11, 1894.

ANY ordinary writing ink is evaporated down in the presence of a suitable non-porous material, such as granulated quartz,

so that the particles are about  $\frac{1}{30}$  inch in diameter. In this way the granules become coated with a firmly-adhering layer of dried ink, and may be handled and shipped, &c., as a dry powder. For use, they are placed in a bottle and just covered with water, the ink being at once dissolved. The patentees claim that they leave no sticky residues in the bottles, and that the granules serve to keep the nibs always clear and sharp.—F. H. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Fire-proofing Compounds.* P. Lochtin. Dingler's Polyt. J. 290, 230—235.

THE systematic testing of about 50 different substances for their capacity of rendering materials unflammable, showed that the compounds recommended for this purpose are of very unequal value. Thus, by holding in the flame of a candle, strips of filtering paper uniformly impregnated with solutions of the various bodies containing 20, 15, 10, 5, 3·5, 2, 1, and 0·5 per cent. of anhydrous substance, or charged with an insoluble body precipitated from such solutions, it was found that, while some were rendered practically unflammable, others did not appear to be much affected by the impregnation, or had become even more combustible than pure paper. In accordance with the results of a great number of such experiments, the substances employed are classified as follows:—

1. *Substances increasing Combustibility.*—Sodium sulphate, sodium sulphite, sodium thiosulphate, sodium silicate, sodium carbonate, sodium stannate, sodium tungstate, sodium chloride, potassium sulphate, potassium phosphate, potassium chloride, zinc carbonate, calcium carbonate, magnesium carbonate, calcium sulphate, ferrous sulphate, magnesium hydroxide.

2. *Indifferent Substances or Bodies, which are effective only when used in large quantities.*—Magnesium sulphate, aluminium borate, zinc borate, calcium phosphate, magnesium phosphate, aluminium phosphate, zinc phosphate, sodium acetate, potassium acetate, silicic acid, sodium phosphate, aluminium hydroxide precipitated from an acid solution, tungstic acid, ammonium tungstate, potassium carbonate.

3. *Substances which render Cellulose specifically unflammable.*—Ammonium sulphate, ammonium phosphate, ammonium chloride, calcium chloride, magnesium chloride, zinc chloride, zinc sulphate, stannous chloride, alum, borax, boric acid, aluminium hydroxide precipitated from sodium aluminate.

The lowest strengths of solution and the least quantity of substance (anhydrous) necessary for rendering 100 parts of cellulose unflammable are given in the following table. The figures to be taken as approximate only:—

Name of Substance.	Lowest Percentage of Substance in Solution necessary to render Cellulose Unflammable.	Least Quantity of Substance required for rendering Unflammable 100 parts of Cellulose.
Ammonium chloride.....	1·5	1·2
„ phosphate ..	1·5	1·5
„ sulphate ....	1·5	4·5
Zinc chloride.....	1·5	1·0
Calcium chloride .....	1·5	1·5
Magnesium chloride.....	1·5	1·5
Aluminium hydroxide ...	1·5	3·8
Alum.....	2·0	..

Name of Substance.	Lowest Percentage of Substance in Solution necessary to render Cellulose Unflammable.	Least Quantity of Substance required for rendering Unflammable 100 parts of Cellulose.
Zinc sulphate.....	1·5	4·5
Stannous chloride .....	2·5	..
Borax .....	1·5	8·5
Boric acid .....	2·5	10·0
Potassium carbonate.....	7·5	..
Magnesium sulphate.....	7·5	15·0
Sodium chloride .....	15·0	35·0
„ silicate.....	17·5	50·0
Silicic acid.....	12·5	30·0
Potassium chloride .....	20·0	45·0
Sodium phosphate .....	7·5	20·0
Potassium „ .....	20·0	..
Aluminium borate .....	12·5	24·0
„ phosphate ...	10·0	30·6
Calcium „ ...	12·5	30·0
Magnesium „ ...	12·5	30·6
Zinc borate.....	7·5	28·0
„ phosphate .....	Above 15	..
Tungstic acid.....	„ 10	Above 15
Sodium tungstate.....	„ 10	„ 15
Ammonium „ .....	7·5	„ 10
Clay (air-dried).....	..	75·0
Sodium and potassium acetates.	7·5—5·0	..

Of these substances the three first-mentioned ammonium salts and aluminium hydroxide may be considered to be the best adapted for practical purposes. The explanation of the fire-proofing properties of the ammonium salts is to be found in their becoming volatilised and dissociated by the influence of heat, the vapours formed producing an uncombustible mixture with the combustible gases. Calcium, magnesium, and zinc chlorides act in a similar manner through the separation of hydrochloric acid. Sodium and potassium chlorides being unalterable by heat are also ineffective as fire-proofing materials. Zinc sulphate and alum likewise owe their effectiveness to dissociation by heat. The action of aluminium hydroxide is a purely mechanical one, hence the striking difference in the behaviour of the granular modification left after drying of the voluminous precipitate from an aluminium salt and that of the exceedingly finely-divided product obtained by the action of carbonic acid upon a solution of sodium aluminate.

As regards the increased combustibility of paper after impregnation with the substances enumerated above, this appears likewise to be owing to a mechanical action, resulting in the prevention of loss of heat.

For practical use the following strengths of solutions are recommended:—

10 to 15 per cent. for textile fabrics, stage decorations, &c.

20 to 30 per cent. for pasteboard, thin boards, &c.

25 to 30 per cent., applied twice or three times, for heavy timber, thick boards, &c.

As an addition to the water used for extinguishing fires, calcium or magnesium chlorides might be useful.—F. M.

*English Jute-Fibre.* A. Pears, jun. Proc. Chem. Soc. 1894 [137], 85-89.

In continuation of previous observations (this Journal, 1893, 599), the seed saved from the plants grown in 1892 was sown at two periods, March and June 1893. Parallel with these, cultivations were made from Indian seed. The plants were cut down in the late autumn when the seed is mature, and the four specimens of fibre obtained were compared.

Slight differences were observed in the resistance to hydrolysis and in the percentages of cellulose, the specimens from the early sowings being the better.

It is evident from the study of these artificial cultivations, that they exhibit the essential features of jute-fibre, and differ chiefly in the degree of hydration, which is dependent on the conditions of growth. The specimen may, in fact, be regarded as a hydrated modification of the normal fibre, with which it corresponds in all the characteristic properties, differing merely in external features (colour, softness, &c.).

## VI.—DYEING, CALICO PRINTING, PAPER-STAINING, AND BLEACHING.

### PATENTS.

*An Improved Process of Dyeing for producing Genuine Turkey Red and Rose Colour in Yarn Hanks and Fabrics.* W. P. Thompson, Liverpool, Manchester, and London. From E. Haeffely, Barmen, Germany. Eng. Pat. 3378, February 16, 1891.

This patent is identical with Eng. Pat. 23,559 of 1893 (this Journal, 1894, 391), for which the patentees are E. Hölken and Co., Barmen.—R. B. B.

*Improvements in Printing on Cotton Fabrics.* E. Iantsch, Hilden, Prussia. Eng. Pat. 3550, February 19, 1894.

The fabric is dyed with a direct cotton colour and then padded with  $\beta$ -naphthol. A reducing discharge is printed on the cotton; this may consist of zinc dust or a stannous salt. After steaming, the fabric is printed with a diazo body, such as benzidine, which produces an insoluble coloring matter on those portions not covered by the resist. The goods are finally steamed, washed, and soaped.—R. B. B.

*Improvements in the Method of and Apparatus for Dyeing, Mordanting, and otherwise treating Woven or Felted Fabrics and Yarns.* J. Relfitt, Leeds. Eng. Pat. 9682, May 15, 1893.

The process described in this patent consists, in its simplest form, of four operations: (1) the fabric is passed through water and between rollers to ensure equal wetting throughout; (2) it is caused to travel in contact with a perforated roller which revolves in a vessel partially filled with dye-liquor, the fabric thus absorbing the dye-liquor from the roller; (3) it then enters a steam-box, and, passing therein in a zig-zag manner over a number of rollers, is subjected to the action of steam; after which it (4) passes between a pair of rollers revolving in water, whereby it is cooled and partially washed. It will often be found advantageous to work with three such machines connected together.

When dyeing thick or heavy goods, in order to ensure that the dye-liquor penetrates the fabric, it is necessary to

dye both sides of the fabric, and when this is the case two dye-vessels and dye rollers are provided in each machine.

The special advantages claimed for the apparatus are, (1) thorough and even dyeing, and (2) the small quantity of dye liquor required. The bulk of the waste liquors is greatly reduced, and the pollution of rivers, &c., minimised.

R. B. B.

*Improvements in or connected with Dyeing, Finishing, Sizing, and Bleaching, or similarly operating upon Textile Fabrics or Fibres and other Materials.* J. Smith Heywood. Eng. Pat. 10,327, May 25, 1893.

The improvement consists in the use of a vessel of U shape or semi-annular form. The warps, pieces, &c., are fastened together and passed in at one end of this vessel, through which they are carried by mechanical means, such as travelling brattice conveyors. The goods may be successively passed through two or more such vessels to undergo bleaching, mordanting, or dyeing operations.

The advantages claimed are (1) the amount of liquor required is small, (2) only a small space is taken up by the apparatus.—R. B. B.

## VII.—ACIDS, ALKALIS, AND SALTS.

*The Motion of Gases in Ducts and Towers.* K. W. Jurisch. Chem. Ind. 16, 425-435.

WHEN a gas passes through a tube, the velocity of motion at the axis is necessarily greater than that at the periphery, since delay is caused by friction against the sides of the pipe. It is usual to assume that the mean velocity in a round tube is the same as that at a spot situated at a distance from the side equal to one-third of the radius. The author has made a variety of measurements showing that this assumption is only approximately correct, the limits of error found lying between - 5 and + 30 per cent.; so that when a greater degree of accuracy is requisite, the velocities at various distances from the side must be measured, and the average value deduced therefrom. Thus, in the case of the gases passing to a Gay-Lussac tower through a circular leaden tube of 10.76 inches radius, the velocities at different distances from the side (observed by means of a Fletcher's anemometer) were as follows:—

Distance from Side,	Anemometer Reading,	Velocity in Feet per Second.
Inches.	Inch.	
1	0.020	4.968
2	0.025	4.514
3	0.030	4.195
4	0.035	5.731
5	0.040	5.716
7	0.045	6.556
8	0.045	6.556

From these values the velocity in each concentric annulus of cross section is calculated, the mean of the whole being 4.968 feet per second; whereas the velocity at one-third of the radius is 5.116 feet per second, or 103.25 per cent. of the true value.

In similar fashion, the exit gases from a hydrochloric acid condensing tower gave the following figures, the

diameter of the tube being 15 inches (Jones' saltake roasters employed):—

Distance from Sale.	Anemometer Reading.	Velocity in Feet per Second.
Inches.	Inch.	
0.5	0.005	8.500
1.5	0.115	9.682
2.5	0.115	9.682
3.5	0.140	10.650
4.5	0.155	11.235
5.5	0.160	11.420
6.5	0.165	11.600
7.5	0.175	11.945

These values lead to the figure 9.953 feet per second as the actual average velocity, whilst the velocity at one-third the radius is 9.682 feet per second, or 97.28 per cent. of the true value. A similar series of observations a month later gave 8.235 as true average velocity, and 8.075 as that at one-third the radius, = 98.05 per cent. of the true value. A third series made at a different period of working off the charges gave 6.321 as the true velocity and 6.384 as that at one-third the radius, = 101 per cent. Several other similar series of observations gave values at one-third the radius varying between 96.92 and 132.23 per cent. of the true average velocity.

Analogous results were obtained in the case of much wider tunnels conveying hot air and gases from saltake furnaces to the condensers; thus, with a tunnel of 52.5 inches radius, the following figures were obtained:—

Distance from Sale.	Anemometer Reading.	Velocity in Feet per Second.
Inches.	Inch.	
1	0.005	2.019
3	0.010	2.855
5	0.015	3.497
8	0.035	5.342
12	0.035	5.342
14	0.035	5.342
16	0.040	5.719
20	0.050	6.384
24	0.035	5.342
30	0.045	6.056
36	0.045	6.056
42	0.050	6.384

Whence the true average velocity is deduced as 3.434 feet per second, that at one-third the radius being 4.0 feet per second, = 116.5 per cent. In a second series of observations 1.206 was the true average velocity deduced, and 5.41 that at one-third the radius, = 128.62 per cent.—C. R. A. W.

*The Densities of Solutions of Soda and Potash.* S. U. Pickering. Phil. Mag. 37, 359—375.

Solutions of soda and potash were prepared from sodium and by crystallisation from alcohol respectively: the lumps were first washed in well-boiled water to remove any adhering carbonate, and were then dissolved to form nearly saturated solutions, which were filtered through glass-wool. The strength of the stock solutions was determined by titration, weaker fluids being prepared by addition of

known weights of water. The temperature throughout was  $15 \pm 0.01$ . Several different series of observations were made for the purpose of more completely eliminating errors; on plotting the results with densities and percentage compositions as variables the curves obtained show that throughout, the density increases with the strength at a decreasing rate with soda and at an increasing rate with potash. The following tables are ultimately deduced:—

#### DENSITY OF SODIUM HYDROXIDE SOLUTIONS AT 15° C.

Percentage of NaOH.	Density.	Percentage of NaOH.	Density.
0	0.999180	26	1.287390
1	1.010611	27	1.298877
2	1.021920	28	1.309708
3	1.033169	29	1.320496
4	1.044317	30	1.331213
5	1.055463	31	1.341879
6	1.066602	32	1.352472
7	1.077733	33	1.362991
8	1.088856	34	1.373453
9	1.099969	35	1.383815
10	1.111069	36	1.394092
11	1.122165	37	1.404279
12	1.133250	38	1.414363
13	1.144353	39	1.424353
14	1.155450	40	1.434299
15	1.166538	41	1.444161
16	1.177619	42	1.453929
17	1.188707	43	1.463623
18	1.199783	44	1.473249
19	1.210861	45	1.482850
20	1.221933	46	1.492406
21	1.233002	47	1.501927
22	1.244119	48	1.511412
23	1.255134	49	1.520863
24	1.266092	50	1.530282
25	1.277063		

#### DENSITY OF SOLUTIONS OF POTASSIUM HYDROXIDE AT 15° C.

Percentage of KOH.	Density.	Percentage of KOH.	Density.
0	0.99918	11	1.10127
1	1.00834	12	1.11076
2	1.01752	13	1.12031
3	1.02671	14	1.12991
4	1.03593	15	1.13955
5	1.04517	16	1.14925
6	1.05443	17	1.15898
7	1.06371	18	1.16875
8	1.07302	19	1.17855
9	1.08240	20	1.18839
10	1.09183	21	1.19857

DENSITY OF SOLUTIONS OF POTASSIUM HYDROXIDE AT 15° C.—*cont.*

Percentage of KOH.	Density.	Percentage of KOH.	Density.
22	1.20834	38	1.57686
23	1.21838	39	1.58793
24	1.22849	40	1.59906
25	1.23863	41	1.61025
26	1.24888	42	1.62150
27	1.25918	43	1.63289
28	1.26954	44	1.64429
29	1.27997	45	1.65577
30	1.29046	46	1.66733
31	1.30102	47	1.67896
32	1.31166	48	1.69067
33	1.32236	49	1.50245
34	1.33313	50	1.51434
35	1.34396	51	1.52622
36	1.35485	52	1.53822
37	1.36586		

The original plottings do not exhibit perfectly continuous curves, breaks being visible at various places; in the case of the soda curve these correspond with the following definite hydrates:—

Breaks in Density.	Hydrates Isolated and Breaks in Freezing Point Figure.
42.6 per cent. NaOH	42.55 per cent. NaOH = NaOH, 3H <sub>2</sub> O
38.77 " "	38.83 " " = NaOH, 3.5H <sub>2</sub> O
35.0 " "	35.71 " " = NaOH, 4H <sub>2</sub> O
29.4 " "	30.77 " " = NaOH, 5H <sub>2</sub> O
24.05 " "	24.10 " " = NaOH, 7H <sub>2</sub> O
20.0 " "	19.89 " " = NaOH, 9H <sub>2</sub> O
12.1 " "	13.3 " " = NaOH, 16H <sub>2</sub> O
5.4 " "	4.0 " " = NaOH, 33H <sub>2</sub> O

Six of these hydrates have been previously isolated in the crystalline condition, whilst in all cases the breaks indicated by the freezing method agree closely with those shown by the density method.

In the case of the potash curve the breaks indicated are less sharply marked; four are noticeable, situated respectively at—

43.2 per cent.	= KOH, 4.65 H <sub>2</sub> O
35.4 " "	= KOH, 5.68 H <sub>2</sub> O
19.0 " "	= KOH, 13 H <sub>2</sub> O
8.0 " "	= KOH, 36 H <sub>2</sub> O

—C. R. A. W.

**Preparation of Ammonium Nitrate from Sodium Nitrate and Ammonium Sulphate.** Fr. Benker, Clichy. Ger. Pat. 69,148, November 20, 1891. Chem. Ind. 16, 437.

On mixing solutions of sodium nitrate and ammonium sulphate and refrigerating, most of the sodium sulphate formed separates out; the remainder is treated with nitric acid, whereby sodium nitrate and sulphuric acid are formed, so that by repeated coolings ammonium nitrate separates. The mother-liquors are neutralised with soda and used over again to dissolve sodium nitrate and ammonium sulphate.

—C. R. A. W.

*The Action of Salts on Acids.* R. W. Wood. Amer. Chem. J. 15, 663—670.

DUGGAN has pointed out the value of diastase and starch as an indicator of neutrality, since the presence of a small trace of acid or alkali prevents the action of the ferment (Amer. Chem. J. 8, No. 3). In the case of weak acids, however, a small amount of their neutral sodium salts prevents this, and in consequence Duggan concludes that these salts are alkaline. The author regards the prevention of the inhibitory action of weak acids, in the manner described, as due to other causes depending upon the dissociation of the salts, and considers that the preventive action upon diastase is due to free hydrogen ions in the case of acids, and to free hydroxyl ions in that of alkalis. The preliminary experiments in support of this idea include the action of caustic soda and of several of the fatty acids and their salts upon the fermentation of malt by diastase. One part of sodium hydrate in 1,000,000 of starch paste has a sensible effect on the quantity of maltose formed in a given time; hence the test is most sensitive, for 5 parts to 1,000,000 of water are necessary for phenolphthalein to give a coloration. The effect of acids is less than that of alkalis, and in all cases a slight amount of acid helps the fermentation, a fact attributed to the presence of a small quantity of impurity of an alkaline nature in the starch. The action beyond this point is to prevent the fermentation, and its extent depends on the nature of and quantity of the acid and upon the temperature, the preventive power of the acid increasing with the temperature to a much greater extent than the power of the diastase. For instance, although diastase is most active at 55° C., yet in the presence of acid 45° C. is a far more favourable temperature. Neutral salts of the weaker acids have a slight retarding effect, and as a rule, the weaker the acid the greater is the retarding action. Salts of strong acids, such as sodium chloride, have little or no effect. The action of weak acids in presence of their neutral salts is to first increase the formation of maltose, owing to their neutralising action upon the impurity in the starch, and beyond this point it is necessary to add several equivalents of the acid (two in the case of acetic acid) before any retardation occurs.—C. A. K.

*Formation of Phosphorus from the Phosphates of the Alkalis and Alkaline Earths by Means of Aluminium as Reducing Agent; and the Action of Aluminium upon Sulphates and Chlorides.* A. Rossel and L. Frank. Ber. 27, 52—55.

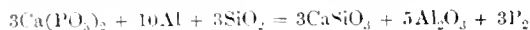
When microcosmic salt,  $\text{NH}_4\text{NaHPO}_4$ , is fused in a porcelain crucible until sodium metaphosphate,  $\text{NaPO}_3$ , is formed and small pieces of aluminium foil are then thrown into the fused salt, little flames are at once observable. If the operation be conducted in a glass tube in an atmosphere of dry hydrogen, phosphorus distils over. The formation of the phosphorus takes place slowly, but is much accelerated when the aluminium is used in the form of powder. The reaction is complex, and a residue is obtained that contains aluminium oxide, sodium aluminate, and a phosphorus-aluminium compound, which is stable at a high temperature, but which is decomposed by water into aluminium oxide, phosphoric acid, and phosphoretted hydrogen. Metaphosphoric acid and phosphorus pentoxide are also reduced at a red heat by aluminium.

The nature of the phosphorus-aluminium compound was investigated by leading phosphorus vapour, heated to a high temperature, over aluminium at a high temperature, and the product obtained was strongly heated until it ceased to evolve phosphorus. The compound formed was then found to be a grey crystalline powder, of uniform composition, which is expressed by the formula  $\text{Al}_2\text{P}_3$ . From the properties of this body, the authors conclude that it may be assumed that this substance is formed on the reduction of the phosphates by aluminium, although they are not able, with certainty, to deduce an equation. They

showed by quantitative determinations that from 28 to 31 per cent. of the phosphorus of the sodium metaphosphate distilled over.

Experiments were next made with the phosphates of calcium and magnesium, and it was found that at a high temperature all phosphates of these metals were decomposed by aluminium. When bone-meal, ground mineral phosphate, powder from fossil bones, magnesium pyrophosphate, calcium metaphosphate, are heated to redness with aluminium, more or less phosphorus distils, and a residue is obtained that behaves like the residue resulting from the reaction upon sodium metaphosphate.

It was further found that these residues, and also the phosphor-aluminium, were acted upon when heated with silica, a fresh liberation of phosphorus taking place. Calcium metaphosphate (or sodium metaphosphate) were then mixed with aluminium and silica in the proportions corresponding to the equations—



or



and heated to redness. The phosphorus, under these conditions, formed in a continuous steady manner, and was so completely expelled from the mixture that the residue scarcely retained a trace of it.

On repeating this experiment and using "superphosphate" (calcined bones were treated with two molecules of sulphuric acid, the mixture heated to redness, and afterwards ground) the reaction was so intense that a not inconsiderable explosion resulted. This unexpected result led to the trial of the action of aluminium upon sulphates.

When barium sulphate was heated with aluminium powder, an intense reaction set in, accompanied by a powerful explosion and by liberation of sulphur. The same reaction occurred with gypsum or calcium sulphate, which explains the intense reaction that took place when the mixture of aluminium, silica, and superphosphate was heated.

It was also found that chlorides were decomposed by aluminium, even sodium chloride, and some of them (chromium chloride) with liberation of chlorine. These reactions the authors will describe later; but they now make use of hydrochloric acid instead of sulphuric acid for converting the orthophosphates into metaphosphates, in which case the phosphorus is yielded by them without explosion, on heating with a mixture of aluminium and silica.—H. S. P.

#### *Decomposition of Sodium Peroxide by Aluminium.*

A. Rosel and L. Frank. Ber. 27, 55.

Sodium peroxide ( $\text{Na}_2\text{O}_2$ ) is for many purposes an admirable oxidising agent, which, however, must be used carefully, as Victor Meyer has already shown.

The authors use it in aqueous solution to oxidise the residues they have obtained when making phosphorus by the action of aluminium upon the phosphates (see preceding abstract). It acts on these residues vigorously, but not dangerously.

The reaction is quite different when aluminium powder and sodium peroxide are mixed together. When this mixture is exposed to the air for a short time the moisture that is absorbed by the sodium peroxide suffices to set up a spontaneous combustion. This may likewise be induced at once by the addition of a few drops of water, and is accompanied by the production of a very high temperature. This mixture must therefore be regarded as highly dangerous, and should be used with caution.—H. S. P.

#### *The Manufacture of Aluminium Sulphate.* K. Jurisch. Chem. Ind. 17, 89—93.

In the manufacture of sulphate of alumina the aim at present is to produce an article as free as possible from iron and containing 14—14.5 per cent.  $\text{Al}_2\text{O}_3$ , free from an excess of acid or of alumina, and thus yielding a clear aqueous solution.

In the usual way of treating bauxite with soda ash it is advantageous to decompose the red variety of bauxite, containing 2 per cent. of silica and yielding a ferric oxide almost wholly insoluble in the lye. The bauxite is calcined, ground, mixed with ash in the proportion of 1 of sesquioxide to 1.2 of soda ash, and subjected to a white heat (but not to fusion), say in Thomson's cryolite furnace. The melt should contain three-fourths of the total alumina in the soluble form. The lixiviation of the mass is best performed by means of weak caustic alkali (say 0.5 equivalent), since otherwise the lye readily deposits aluminium hydrate on contact with the insoluble residue. In any case the lixiviation should be effected with expedition, and hence the melt is ground up, introduced into the boiling solution, and after 5—10 minutes agitation is drawn off as a solution marking no less than 64° Tw. and containing 1 mol. of  $\text{Al}_2\text{O}_3$  to 1.8—2 mols.  $\text{Na}_2\text{O}$ , or 170—175 grms.  $\text{Al}_2\text{O}_3$  per litre. The filter-pressed solution is allowed to settle again and carbonated at 50°—90° C., when alumina is precipitated in a granular form and almost free from silica and iron. The filtered solution is available for the manufacture of soda crystals, whilst the slightly washed precipitate is introduced into sulphuric acid of 160° Tw., which is introduced into a steam-jacketed copper vessel and heated to 100° C. When all effervescence ceases the solution is run off into lead-lined crystallisers and allowed to cool, with occasional agitation. The colour when yellowish may be masked by the addition of copper sulphate to the hot solution. About 75 per cent. of the iron contained in the final product is introduced with the sulphuric acid. Aluminium sulphate thus manufactured has the following average composition:—

$\text{Al}_2\text{O}_3$ , 14.5;  $\text{SO}_3$ , 33.7;  $\text{Na}_2\text{SO}_4$ , 0.6;  $\text{Fe}_2\text{O}_3$ , 0.02; insoluble, 0.50; water, 50.68 per cent.—H. A.

#### *Fire-proofing Compounds.* P. Loebtin. Diagler's polyt. J. 290, 230.

See under V., page 512.

#### *The Action of Light on Oxalic Acid.* A. Richardson. Proc. Chem. Soc. 1894 [137], 88.

Downes and Blunt, in 1879, observed that solutions of oxalic acid evolve carbon dioxide when exposed to light. In the present paper the author shows that hydrogen peroxide is also formed. Experiments are described in which this was found to be the case with numerous specimens of oxalic acid obtained from different sources, and carefully purified. The presence of the peroxide was proved by the titanous acid and chromic acid tests.

The results of experiments in which the carbon dioxide evolved during the decomposition of a known weight of acid was estimated, showed that the oxidation of the carbon was complete.

Whilst the total amount of hydrogen peroxide formed in the solution increased with the concentration of the acid, the proportion of peroxide formed to acid decomposed, simultaneously decreased. The author draws the following conclusions:—

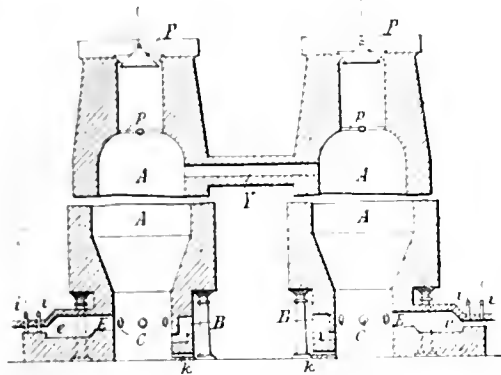
- (1) Hydrogen peroxide is stable in solutions of oxalic acid in the dark.
- (2) It is fairly stable in these solutions when exposed to light if excess of oxygen be present.
- (3) Rapid decomposition of hydrogen peroxide occurs in solution of oxalic acid in absence of oxygen when their solutions are exposed to light.



## PATENTS.

*Improvements in and Apparatus for obtaining Cyanogen, Cyanides, and other Products from Carbonaceous Matter or Substances containing Ammonia or Compounds thereof.* H. Aitken, Darroch, N.B. Eng. Pat. 4037, February 23, 1893.

The invention partly relates to utilising the gases produced in blast-furnaces for smelting iron and other ores, or oil- or gas-producers of the blast-furnace or Siemens type; and partly to a furnace specially devised for the production of cyanogen compounds. The latter apparatus consists, as shown in the drawing, of two producers connected by the flue Y, in all respects similar, which are charged through the bells P, provided with valves p, with carbonaceous matter, and lime or alkalis, into the chambers A;



k is a tap-hole for running out any molten metal when an ore is used with the charge. B are the slag-holes, C the tuyères, and E an aperture into the furnace from the chambers e, in which chambers may be placed an alkali. When both producers are fired the hot blast is supplied to No. 1, the dampers i i being closed, and the resulting gases pass through the conduit Y and have exit by E e in No. 2 into a dust chamber, whence they pass into a scrubber, containing, for instance, solution of an iron salt, and provided with suitable appliances for ensuring contact of the gases with the liquid. When the temperature in No. 2 falls too low, the current is reversed, the blast enters No. 2, and the gases have exit through No. 1, and thus the functions of the two producers are alternated indefinitely.

A single chamber may be used by taking off the gases at the top when combustion is well established, and then forcing in air at the top, with or without steam, and drawing off the products at or near the bottom. When it is desired to make an ordinary producer-gas, with cyanogen or its compounds, the tuyères are placed on one end of the furnace, and part of the gases pass off at the top in the usual manner, while the remainder are drawn off near the bottom, as shown in the duplex apparatus. Holes are provided in the chamber through which potash or other alkali, or nitrogenous substances, such as horns, hoofs, or the like, are introduced. Or gases rich in nitrogen, as ammoniacal gases, may be passed in regulated quantities into the furnace through these holes, for conversion into cyanogen compounds.

When the invention is applied to iron blast-furnaces, a valved pipe inclined upwards outwardly, and connected at its outer end by a vertical pipe to the dust-collecting and scrubbing apparatus, is inserted, preferably just above the level to which the slag is allowed to rise, so as to avoid any unnecessary mixture of free oxygen with the gases. Extra carbonaceous matter and lime may be added in the furnace, so as not to affect the quality of the iron made. In taking off gas from gas-producers of the Siemens type, the gases taken off are drawn as far as possible directly from the hot carbonaceous matter. In all cases the gases withdrawn may, after deprivation of cyanogen compounds, be returned

to the furnace or otherwise utilised. It is considered that atmospheric nitrogen takes part in the formation of cyanogen under the conditions described.

The dust collected in the dust chambers from the gases drawn off is washed for the cyanides it may contain. The salts used in the scrubber may be the sulphate, chloride, oxide, carbonate, or sulphide of iron, and these after use may be treated with sodium or potassium salts to obtain the corresponding ferrocyanide, which may be treated in any desired way to produce marketable cyanogen salts.

—E. S.

*Improvements in the Production of Alkali and in Apparatus therefor, the said Apparatus being applicable for Electrolysis generally.* J. Haigreaves, Farnworth-in-Wilms, and T. Bird, Cressington, Liverpool. Eng. Pat. 5197, March 10, 1893.

See under XI., page 528.

*Improvements in the Manufacture of Manganese Peroxide.* G. S. Albright, Oldbury, Worcester, and J. J. Hood, Shepherd's Bush Road, Middlesex. Eng. Pat. 9271, May 9, 1893.

Manganous sulphate is prepared, as a preliminary to obtaining manganic hydroxide, or a mixture of the peroxide with the monoxide and calcium oxide, known as Weldon mud, for application to such purposes as gas purification, &c. Manganese ores containing the peroxide are mixed with coal dust and sulphuric acid, and the mixture is heated on a furnace bed. Manganous sulphate is washed out of the product. Ores not containing the peroxide are simply heated with sulphuric acid until any iron present is rendered insoluble. Spent manganous oxide from gas-works may be roasted to obtain sulphuric acid and a residue of manganous sulphate; or it is simply heated with sulphuric acid, with or without hydrochloric acid, to obtain a residue of manganous sulphate and chloride, with calcium sulphate and free sulphur, which is lixiviated, and sulphur is distilled off the insoluble portion.

The manganous sulphate solution is treated with calcium chloride in excess; the calcium sulphate that forms is separated, and the washings are made with strong calcium chloride solution, the presence of which in the subsequent oxidation process is necessary. The manganous sulphate solution with calcium chloride and lime is then treated with air in a Weldon oxidiser to obtain the "mud." The calcium chloride remaining in solution is employed again for preparation, as before, of manganous chloride from the sulphate.

According to another process, the manganous sulphate solution is precipitated by ammonia, and air is blown in to precipitate the manganese as Weldon mud. The ammonia blown out is recovered, and the ammonium sulphate formed in the solution is drawn off and evaporated.

Weldon mud may be purified by treatment with dilute cold hydrochloric acid. Similar processes may be applied to recover manganic oxide from spent "mud" used in gas purification.—E. S.

*Improvements in Separating Salts of Soda and Potash, and particularly Common Salt, from Solutions of Magnesium Chloride.* T. H. Bell, Middlesbrough. From T. Schloosing, Paris. Eng. Pat. 10,341, May 25, 1893.

The inventor refers to a former patent, No. 11,469, 1891 (this Journal, 1892, 686), in which "chloride of magnesium in grains" is obtained by concentration of a solution, associated with sodium or potassium salts, which form small crystals and fall as the evaporation proceeds. He now finds that sodium chloride dissolved in magnesium chloride solution, is much more soluble hot than cold, so that much of the sodium salt may be obtained in large crystals by simply cooling the solution previously suitably concentrated. The solution of magnesium chloride obtained after having distilled upon magnesia the mother-liquor resulting from the manufacture of sodium carbonate by the

ammonia process, contains a large proportion of sodium chloride. These liquors, cleared from excess of magnesia, are concentrated until salting-out commences, and are then allowed to cool, or are cooled by an outer flow of cold water, when about half the sodium salt present separates in large crystals. The solution is run off these crystals, and the concentration and cooling are repeated; the solution is then again concentrated, and the fine crystals which form are fished out. These fine crystals are mixed with the larger crystals previously obtained, and are washed and dried.

In dealing with the magnesium chloride recovered in the process for obtaining chlorine from that salt, the solution being dilute and free from sodium salts, may be strongly concentrated in a boiler without danger of forming a deposit. Such a strong solution may then be added to the magnesium chloride solution containing common salt, obtained in the soda-ammonia process mentioned in the preceding paragraph, and the proportion of magnesium chloride present being thus greatly increased, a single concentration suffices to obtain about three-fourths of the whole sodium chloride in large crystals. After further concentration of the cleared solution, the remainder of the sodium chloride is salted out.—E. S.

*Improvements in connection with Electrolytic Apparatus.*  
H. Y. Castner, London. Eng. Pat. 10,584, May 30, 1893.

See under XI., page 529.

*Improvements in Calcining Apparatus for the Conversion of Bicarbonate of Soda into Carbonate of Soda and other Uses.* La Société Marcheville Daguin and Company, Paris. (Under Internat. Convention, date claimed December 3, 1892.) Eng. Pat. 12,997, July 3, 1893.

A CIRCULAR revolving hearth is supported by a set of conical rollers, and carries a vertical cylinder, perforated in places for outflow of gases, issuing into it from a series of superposed retort sections, which it traverses. The retort sections are so formed, with alternately inclined upper and lower sides, that the salt (such as sodium bicarbonate) fed into the upper part of the cylinder and directed outwards by a conical diaphragm, is carried first to the periphery, and then towards the centre of the section before it falls into the next section, an annular space being left for the purpose about the cylinder, which latter carries, so as to revolve with it, conical discs extending into each retort section. The upper internal face of each retort chamber has one or more scrapers, as has also each conical disc. In the example given, the retort sections are caused to revolve independently of, and in a contrary direction to, the central cylinder, the mechanism for effecting which is shown. The material or salt treated falls from the lowest retort section on to the hearth, over which is an arched space, channelled to admit heating gases. Adjustable rakes are provided for the hearth. The series of retorts is enclosed in a fixed casing of refractory material supported upon pillars, and supporting the fixed arch over the hearth. The casing is pierced on opposite sides of the space occupied by the retorts; on one side, to admit heating gases, and on the opposite side for discharging the products of combustion. The use of conical track rollers constitutes a claimed feature in the apparatus.—E. S.

## VIII.—GLASS, POTTERY, AND ENAMELS.

*The Preparation of Anhydrous Crystalline Silicates.*  
H. Traube. Ber. 26, 2735.

On adding a solution of zinc sulphate to one of sodium silicate an amorphous precipitate of zinc silicate is obtained.

The zinc silicate will be contaminated with zinc hydroxide in the event of the sodium silicate containing free alkali. Zinc silicate prepared in this manner as nearly free from zinc hydroxide as possible, was dried and exposed, together with eight times its weight of fused boric anhydride, in a platinum crucible to the highest temperature of a porcelain furnace in the Royal Porcelain Works of Charlottenburg, for a period of 10 days. At the end of this period the bulk of the boric anhydride had volatilised, and on dissolving the remainder in water a white crystalline product insoluble in acids was obtained. This body was proved to be zinc silicate  $ZnSiO_3$ ; it is a *pyroxene* isomorphous with the mineral *enstatite*  $MgSiO_3$ . It forms colourless prismatic crystals with rounded ends, belonging to the rhombic system. The same process was used by Ebelmen in 1851 for preparing magnesium silicate, but the product was partly monoclinic, partly rhombic in form. The method appears to be generally applicable to the preparation of similar, as well as complex silicates.—B. B.

*The Chemical Behaviour of Glass.* F. Foerster.  
Ber. 26, 2915—2922.

### A. The Action of Acids upon Glass.

EXPERIMENTS made with flasks of different kinds of lime-alkali glass proved that the loss of weight resulting after heating with diluted acids to  $100^\circ$  for six hours, was always the same with the same kind of glass, and was entirely independent of the strength of the acid or of its chemical composition. Sulphuric, hydrochloric, nitric, and acetic acids gave identical results. Only very strong acids had less effect than dilute ones, which again have less action than pure water. Similar results were obtained in working at temperatures of  $160^\circ$  and  $190^\circ$  with pieces of glass tubing enclosed in sealed tubes. The influence of the degree of concentration was, however, more pronounced than in the previous experiments, and was again in inverse ratio to the strength of the acid used. The dissolving action of the acids is therefore governed by the amount of water contained in them. Bearing in mind that the action of water upon glass consists in the liberation from it of alkali, which again further increases its vulnerability to water, the passive part played by the acid may be readily understood. In the case of glass containing an unusually large amount of alkali, the action of the acid, however, is more pronounced than that of pure water, the decomposition being analogous to that of many natural silicates. Lead glass exhibits the same peculiarities as lime-alkali glass, according to the proportion of lead contained in it. The nature of the base in combination with the silica likewise seems to influence the resisting capacity of the glass. Thus a zinc-lime-soda glass (Jena thermometer glass 16 III) was more attacked by concentrated acid than lime-soda glass of equivalent composition. The action of pure sulphuric acid is less strong than that of boiling water, but at very elevated temperatures its vapours produce a more marked effect.

### B. The Effect of the Atmosphere and of Water upon Various Kinds of Glass.

Dry carbonic acid does not affect glass, hence the action of the atmosphere primarily depends on the aqueous vapour contained in it. The liability of a glass to suffer changes by atmospheric influences can therefore be ascertained by estimating colorimetrically the amount of alkali separated on treatment with water. Glass, especially when rich in alkali, is capable of absorbing water which can only be completely expelled by heating to about  $500^\circ C$ . The water combines chemically, forming hydrates which represent the intermediate stage in the process of the decomposition of the glass by the action of water. An important part played by the alkali split off by water seems to consist in its facilitating the formation of such hydrates.

## C. Comparison of some Good Species of Glass with regard to Chemical Behaviour.

TABLE I.

SHOWING CHEMICAL COMPOSITION OF VARIOUS KINDS OF GLASS.

No.	K <sub>2</sub> O.	Na <sub>2</sub> O.	CaO.	MnO.	Al <sub>2</sub> O <sub>3</sub> (F.O.).	SiO <sub>2</sub>	I. R.O. II. RO:SiO <sub>2</sub>	Number of Alkali Molecules in 100 Molecules.
1	6.2	6.4	10.0	0.2	0.4	76.8	0.95:1: 7.16	10.4
2	7.0	8.3	8.1	..	0.3	76.3	1.44:1: 8.80	12.7
3	11.8	4.9	7.6	0.1	0.5	75.1	1.50:1: 9.24	12.8
4	4.3	10.0	7.8	..	0.3	77.6	1.48:1: 9.28	12.6
5	4.6	10.1	7.7	..	0.4	77.2	1.51:1: 9.36	13.0
6	0.6	14.3	11.2	0.4	2.9	70.6	1.18:1: 5.88	14.6
7	9.7	9.0	6.8	Trace	0.4	74.1	2.04:1: 10.17	15.4
8	6.7	13.7	7.2	0.3	3.2	68.9	2.27:1: 8.91	18.6

TABLE II.

SHOWING ACTION OF WATER, CAUSTIC SODA, AND SODIUM CARBONATE.

No.	Alkali taken up by Water, expressed in Thousandths of a Milligramme Na <sub>2</sub> O and referring to a Surface of 100 Sq. Cm.		Loss of Weight in Milligrammes over a Surface of 100 Sq. Cm., during 3 Hours heating to 100°	
	After 3 Days' previous Treatment with Water at 20°, after 8 Days' Treatment with Water at 20°	After subsequent Treatment, for 3 Hours, with Water of 80°	With Double Normal Caustic Soda.	With Double Normal Solution of Na <sub>2</sub> CO <sub>3</sub>
1	13	27	37	59
2	14	56	40	77
3	14.5	45	38	79
4	15	50	38	73
5	18	66	42	79
6	27	98	31	41
7	32	217	..	..
8	77	654	46	45

Nos. 7 and 8 are inferior soft varieties of glass commonly met with in commerce.

Of the good qualities Nos. 1—6, no single specimen shows an absolute superiority over the others. No. 1 closely resembles in its composition Stas' glass, which is still unsurpassed, and approaches the limit of that which is technically possible in the production of a glass capable of resisting the action of chemical agents.—F. M.

## PATENTS.

*Improvements in the Manufacture of Glazes, Enamels, and the like.* W. P. Thompson, Liverpool. From G. Leuchs and C. Leuchs, Nuremberg, Germany. Eng. Pat. 9611, May 13, 1893.

SILICA in glazes may be partly replaced by titanic acid without becoming coloured, provided the fusion be conducted in an oxidising atmosphere. The resulting glazes are said to be more fusible than those containing an equivalent of silica, and to be more resistant to corrosive agencies,

and also to possess a high specific gravity, good lustre, and a large coefficient of expansion. The latter quality allows of their use direct upon iron-ware, which has hitherto generally been coated in two operations, the underglaze having a suitable coefficient of expansion and the upper being resistant to acids. When a moderate amount of titanic acid is used, the glaze is transparent, and when a large quantity is employed an opaque glaze or enamel is produced. A typical mixture for the former is made from 15 to 30 parts of nitrate of soda, 116 of soda, 33 to 50 of carbonate of lime, 60 to 360 of quartz, 62—83 of boric acid, and 20 to 80 of titanic acid, while one for the latter is composed of 15 to 30 parts of nitrate of soda, 116 of soda, 100 of carbonate of lime, 60 to 180 of silicic acid, and 80 of titanic acid. Variations in the mixtures and the addition of coloring oxides are also claimed.—B. B.

*Waffer China Mosaic.* J. H. Bale, London. Eng. Pat. 22,362, November 22, 1893.

A LAYER of kaolin or clay is placed upon a suitable base or plate, such as a tile or sheet of metal, painted with coloring oxides, covered with glaze, and fired. The coloured glazed sheet thus produced is removed from the plate and used for mosaic work.—B. B.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*The Influence of Temperature on the Tensile Strength of Metals.* M. Rudeloff. Mitt. Königl. techn. Versuchs. 11 [6], 292.

See under X., page 520.

## PATENTS.

*Improvements in and connected with the Glazing of Bricks and like Articles.* W. Crookes, London. Eng. Pat. 9381, May 10, 1893.

Most commercial glazed bricks and tiles are made of fire-clay, and the present patent is concerned with the substitution therefor of ordinary non-refractory bricks appropriately glazed. Three types of brick are described, consisting of various mixtures of non-refractory clays, sand, and

glazeous materials. Five glazes or enamels are also mentioned containing red lead, silica, kaolin, carbonate of soda or other alkaline salts, arsenic, and tin oxide, which are used in various proportions, according to the nature of the required product. The glazes are fused, ground, and applied to the bricks in the usual manner.—B. B.

*Improved Method of Manufacturing Hydraulic Cement.*  
G. W. A. Stein, Wetzlar, Germany. Eng. Pat. 475,  
January 9, 1894.

IN order to render the usual preliminary crushing of cement clinker in stone-breakers unnecessary, the patentee adds 5 to 30 parts of granulated blast-furnace slag to 100 parts of the ordinary raw materials for the manufacture of hydraulic cement, and burns the mixture in the ordinary way, obtaining a product which is granular and brittle and can be fed direct to the grinding machinery without passage through a stone-breaker. By the use of a larger proportion of slag, e.g. 30 to 125 parts to 100 of cement raw materials and modifying the process of burning, a product can be obtained similar to hydraulic lime or Roman cement, or Portland cement.—B. B.

## X.—METALLURGY.

*The Influence of Temperature on the Tensile Strength of Metals.* M. Rudeloff. Mitt. Königl. techn. Versuchs.  
11 [6], 292—330.

THE tests were made on wrought iron, mild steel, copper, Delta metal (cast and rolled), and manganese bronze (4 per cent. rolled and 15 per cent. cast). No analyses of the samples are given. The temperatures adopted were the ordinary temperature of the room, 100° C. obtained by a steam jacket, 200° C. by naphthalene vapour, 300° C. by naphthylamine vapour, and 400° C. by a metal bath. In all cases the whole of the turned length of the specimen in which the fracture was to occur, and in addition a portion of the screwed end of the specimen by which it was held, were exposed to the hot bath to minimise lowering of temperature of the specimen by conduction through the screwed jaws of the testing machine. No tests of the actual temperature of the specimen as distinct from that of the bath are recorded. The gist of the results is as follows.

In the second table hereafter given, a comparison is made between the chief mechanical properties of the specimens at the ordinary temperature taken as a standard, and at the higher temperatures mentioned. In order to translate relative figures into absolute numbers, the following data are necessary for each of the metals tested at the ordinary temperature:—

TABLE I.

Material of Test Piece.	Elastic Limit.	Tensile Strength. (on 30 mm.)	Elongation (on 30 mm.)	Reduction of Area.
	Kilos. per Sq. Mm.		Per Cent.	
Wrought iron.....	26.3	35.3	35.3	55.0
Mild steel.....	27.3	41.8	34.4	50.6
Copper.....	26.9	20.1	17.9	54.6
Delta metal (rolled) ..	14.8	42.7	41.5	37.5
"    (cast) ....	9.7	32.9	21.9	24.8
Manganese bronze (4 per cent. rolled).	13.6	29.1	40.7	67.7
Manganese bronze (15 per cent. cast).	14.9	35.7	31.3	43.7

Calling each of these results 100, the figures obtained at the higher temperatures have the following values:—

TABLE II.

Material of Test Piece.	Temp.	Elastic Limit.	Tensile Strength.	Elongation (on 30 mm.)	Reduction of Area.
Wrought iron	100	100	110	54	87
	200	67	134	57	83
	300	64	138	94	90
	400	59	91	140	109
Mild steel	100	99	108	56	86
	200	88	125	56	69
	300	60	114	98	93
	400	48	92	92	52
Copper	100	95	92	66	103
	200	81	81	61	96
	300	56	70	75	78
	400	30	48	47	18
Delta metal (rolled)	100	112	92	110	109
	200	110	72	106	148
	300	75	50	158	151
	400	9	16	196	148
Delta metal (cast)	100	97	100	126	97
	200	119	82	134	118
	300	80	62	270	176
	400	17	20	146	100
Manganese bronze (4 per cent. rolled)	100	87	92	94	103
	200	85	89	93	97
	300	88	80	91	83
	400	70	71	39	30
Manganese bronze (15 per cent. cast)	100	100	92	79	80
	200	95	94	100	98
	300	102	93	91	79
	400	89	77	98	75

The following are the chief conclusions drawn from these results, regard being had to the fact that they apply primarily to the particular specimens tested, and are not necessarily to be accepted without modification for the varieties of metals examined generally.

Wrought iron and mild steel show a similar decrease in elongation up to 150° C., and a corresponding increase in tensile strength up to 250° C., indicating that the injurious influence of increase of temperature up to the latter point on the working quality of the metal is as true for wrought iron as it is generally known to be for mild steel. Above 300° C. wrought iron exhibits a decrease in tensile strength and increase in elongation, whereas mild steel, though also diminishing in tensile strength, suffers a decrease in elongation. It follows from this that the temperature below which wrought iron is liable to injury in working may be set at 300° C., whilst for mild steel a higher limit must be adopted.

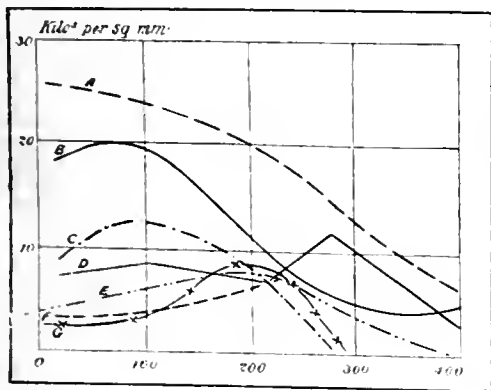
With cast Delta metal the elongation increased but slightly up to 200° C. and then considerably to 300° C., after which point a sudden fall took place, whereas rolled Delta metal at 200° C. gave values nearly 50 per cent. in excess of those obtained at the ordinary temperature, with a decrease from 200° C. to 300° C., and a further large increase as the temperature again rose. The deductions to be drawn from these relations are that in the case of the samples tested the most suitable temperature for working

the cast metal is about 300° C., while the rolled metal is not in a fit state for being subjected to mechanical work from 200°—350° C. Should this difference of the rolled from the cast metal be valid generally, the first heat used in rolling from the cast state should be of lower temperature than further heats for subsequent work on the metal.

**Manganese bronze** (4 per cent. rolled) is most fit to be worked at the ordinary temperature, its elongation being then greatest. The large diminution in elongation between 250° and 400° C. explains the fact (well recognised in practice) that this alloy is red-short. As far as the figures go, which were obtained for 15 per cent. cast manganese bronze, it appears that the mechanical properties of the alloy are not much influenced by rise of temperature, and any one of the temperatures used in the investigation is suitable for the working of the material.

The appearance of the surface of the broken test-pieces, pieces of Delta metal (cast and rolled) and manganese bronze (4 per cent.) indicated that the metal had drawn out more uniformly at the higher temperatures, a phenomenon which the author ascribes to an alteration of molecular state analogous to that exhibited by brass which has been softened by heating and quenching. A similar increase in softness and uniformity should therefore be produced by heating these alloys to 250°—300° C. and quenching. Microscopic examination is suggested for settling this question of change of state.

Passing from the consideration of the working properties of these metals and alloys to that of their qualities as structural materials, the chief point to be regarded is the limit up to which extension and the load producing it, are proportional. The variations in this value caused by alteration of temperature are not included in the figures quoted in the foregoing tables, but are given in the following diagram, in which A is the curve for mild steel, B for wrought iron, C for rolled Delta metal, D for manganese bronze (15 per cent.), E for east Delta metal, F for copper, and G for manganese bronze (4 per cent.).



It being conceded that the load on a structural material should not exceed this limit so that no permanent deformation may occur, the significance of these curves is at once apparent. In those cases where rise of temperature raises the limit, regard must also be had to the alteration of other properties, notably the elongation and ultimate strength by the same rise.—B. B.

#### Certain Ternary Alloys. Part VIII. C. R. Alder Wright. Proc. Roy. Soc. 55, 130—139.

THE author has previously shown (this Journal, 1892, 492, and 1893, 449) that the ordinary book statements respecting the miscibility of melted aluminium and cadmium are incorrect, and that so far from alloying together in all proportions, they are only miscible to a very limited extent. He now finds, as the mean of a number of observations, that at temperatures averaging near 725° C. a mixture of

aluminium and cadmium separates into two binary alloys of the following compositions:—

	Heavier Alloy.
Aluminium.....	67.22
Cadmium.....	32.78
	100.00
	Lighter Alloy.
Aluminium.....	96.61
Cadmium.....	3.39
	100.00

Addition of varying quantities of tin to mixtures of these two metals at the average temperature 725° C. gives rise to a series of pairs of "conjugate" ternary alloys, precisely comparable with those similarly produced with lead (or bismuth) and aluminium (or zinc) with tin as "solvent" metal. The critical curve obtained on plotting the results, lies well inside that obtained with aluminium-lead-tin at 800°, indicating that the replacement of lead by cadmium diminishes the area representing mixtures of the three metals incapable of forming a single homogeneous fluid. The same remark applies also to the substitution of cadmium for bismuth, the temperature being the same throughout. No irregularity of outline in the aluminium-cadmium-tin critical curve was noticed, nor any peculiar conformation of tie-lines, suggesting the existence of definite metallic compounds; unlike the tie-lines in the case of aluminium-lead-tin alloys, where the lower lines slope in one direction and the upper ones in the other, the change of slope being apparently due to the formation of the definite compound,  $\text{SnPb}_3$ . With cadmium or bismuth (in conjunction with aluminium and tin) the direction of slope of tie-lines is the same, viz., that shown by the upper tie-lines of the curve where lead is present, and opposite to that shown by the lower ones.

Mixtures of aluminium and lead (or bismuth) with antimony as "solvent" metal kept fused at 850°—900° separate into pairs of fluid conjugate alloys in a perfectly well-defined way as long as the amount of antimony present does not exceed a certain proportion of the total mass (some 30 or 40 per cent.). When, however, the antimony is present in sufficiently large quantity relatively to the aluminium, solid matter forms during the period of tranquil fusion necessary for the separation from one another by gravitation of the two ternary alloys produced; and this causes the topmost and lowermost portions of the total mass to become abnormal in composition. The effect of this is to produce an excrescence or horn-like projection at the upper part of the critical curve when the results are represented graphically. The solid matter thus separating appears to be the definite antimonide of aluminium previously described by the author (this Journal, 1892, 492), indicated by the formula,  $\text{AlSb}_3$ ; the existence of this compound (found by Roberts-Austen to fuse at nearly the same temperature as gold, about 1,045° C.) has since been corroborated by D. A. Roche (Moniteur Scientifique, 1893, 269), who appears to have been unacquainted with the previous work on the subject. So far as the lower normal portions of the critical curves are concerned, the usual rule holds, viz., that the curve where bismuth is a constituent lies inside of that where lead is present instead, *ceteris paribus*. In the case of the lead alloy curve, there is nothing irregular about the conformation of this lower normal portion; but with the bismuth alloy curve a strongly-marked notch or inward depression is noticeable, the maximum extent of which occurs nearly when the bismuth and antimony present are in the ratio indicated by the formula,  $\text{Sb}_2\text{Bi}_3$ ; precisely as in the parallel case of zinc-bismuth-antimony alloys. Doubtless, therefore, a definite antimonide of bismuth of this composition exists. As regards the direction of slope of tie-lines, it is noticeable that aluminium-lead-antimony and aluminium-bismuth-antimony alloys both furnish tie-lines sloping in the same direction as that exhibited by zinc-lead-antimony alloys; whereas the slope with zinc-bismuth-antimony alloys is in the opposite direction.—C. R. A. W.

*Plant for Granulating Slag.* R. H. Terhune. School of Mines Quarterly, 1894, 15 [2], 108-110.

THE slag for which the plant was designed was from a lead blast-furnace, and the chief object of the adoption of granulation was to facilitate the removal of the slag. The plant is of the type usual for iron blast-furnace slag in this country. The slag is run into a trough 2 ft. wide and 6 in. deep, lined with cast iron plates 1 in. thick, and received in flowing water, the trough having an inclination of  $\frac{1}{4}$  in. per foot. This trough fronts all the furnaces the slag of which is to be granulated, and thence extends to a settling pit 8 ft. deep, whence the slag is raised by an elevator with buckets of malleable cast-iron, and delivered into trucks, or a storage bin, as may be required. In running the slag into the trough the operator is warned of the need of cutting off the stream by sharp explosions due to small portions of matte accompanying the slag. The granulated slag is brittle, and will pass through a  $\frac{1}{4}$ -in. mesh. It makes fair ballast for railways, but would be better for this purpose if coarser and more coherent. An incidental advantage of granulation is that the sampling and weighing of the daily output of slag are more easily performed, than if that material were allowed to solidify *en masse*; thus also control of the working of the furnace is facilitated.—R. B.

*The Reduction of Aluminium as a Scientific Problem.*

Alfred H. Bucherer. Eng. and Mining J. 1894, 315-316.

As there is no agent capable of reducing alumina by a single reaction, we turn of course to methods which make possible its reduction by several steps. The only compounds which permit of such gradual reduction are the anhydrous halogen salts, viz.: fluorides, chlorides, bromides, iodides, and further the sulphide. One would then naturally reduce the oxide to one of these compounds. And we know that except with the fluoride we have to overcome the affinity of the oxygen for aluminium by calling into play two agencies, the one with a tendency to combine with the oxygen of the alumina, and the other with the aluminium. A typical reaction of this sort is the formation of the chloride—



The other halogen compounds are formed in a similar way. In 1890 the author demonstrated that a similar reaction can be effected at a white heat for the production of the sulphide—



In case of the halogen salts reduction can be effected by alkali metals. The reduction of aluminium chlorides, or rather the anhydrous double chlorides of sodium and aluminium, was for some time the exclusive method used for the production of aluminium. After a very carefully and long continued development of this method, it has been superseded, and we can confidently assert that neither the chloride, the bromide, nor the iodide will ever be used for the production of aluminium, for we can never expect to obtain them cheap enough to compete with the present process, independently of the present high cost of the sodium required for the reduction. It has appeared for some time as though the reduction of the fluoride by sodium offered commercial advantages; but in view of the cheapness of the present method this can only be possible if the cost of sodium could be very considerably lowered, and we must also take into consideration that the reduction of the fluoride, as well as its regeneration by the action of the sodium fluoride on aluminium sulphate, are based on reactions which offer great difficulties, for they are not complete. As to the chemical reduction of the sulphide, it is not possible to produce pure metal, as appears from a series of experiments which the author has made. Tin and iron seem to be the only agents capable of reacting on aluminium sulphide, but the product obtained is of no value. An experiment performed by the author for the reduction of aluminium sulphide by zinc, although its result was negative, is of some interest. After having tried the experiment at temperatures obtainable in furnaces, he resorted to electrical heat. A cylindrical rod of carbon  $\frac{3}{4}$  in. in diameter and

1 ft. in length was hollowed out, filled with a mixture of granulated sulphide and zinc, and the ends closely fitted into large carbon cylinders, 3 in. in diameter, which latter in their turn were connected with the terminals of a dynamo, and a slanting position given to the cylinder. In passing a strong current of 20 h.p., the rod was brought to a dazzling whiteness and was kept so for 20 minutes. It is interesting to note that the temperature was so high, that when, at the end of the experiment, the carbon was examined, it was found to be bent as a result of accidental strain. No aluminium had been produced, but the larger part of the zinc was found in the upper part of the tube, where it had condensed, the larger carbons acting as condensers. The zinc did not contain a trace of aluminium, although the conditions had been most favourable. It appears, then, that the aluminium cannot be economically produced chemically, since we have considered all methods, and the electrical methods only remain.

The reduction of aluminium compounds by the electrolysis of aqueous solution will never become an economical process for producing aluminium, as too much energy is wasted. This loss of energy is due to two causes: first, to the high voltage required, and secondly, to the small output, which latter, due to recombination, is not at all proportionate to the product of the number of amperes by the time. It goes without saying that with aqueous aluminium salts polarisation is excessive.

Before passing on to the discussion of the electrolysis of molten aluminium compounds, some general peculiarities of molten electrolytes observed in an extended series of experiments, are mentioned. The high temperature at which electrolysis is effected, the corrosive character of the molten substances, the different states of aggregation in which the metal is deposited, require different technical appliances, which in their turn influence the conditions of electrolysis. As it is necessary to make the electrolytising vessel the cathode, and as the carbon cylinders forming the anode have small surface, it follows that there is comparatively small current density at the negative electrode, which circumstance has a marked influence on the reactions going on, especially where there is danger of reversed action by which the metal re-enters the electrolyte as an ion. It is worthy of mention that, in experimenting, the electrolysis of molten compounds often fails for no other reason than that the density of the current has been too small.

It is an experimental fact that under certain conditions of temperature, and when there is a large excess of sodium fluoride, aluminium is capable of reducing sodium, or, what amounts to the same thing, sodium will not reduce aluminium fluoride in the presence of a large excess of sodium fluoride. Now in the present process we have actually aluminium in the presence of sodium fluoride contained in the cryolite, and as a matter of fact we find that the output of the metal is small if the relative amount of sodium fluoride be large, so that by adding aluminium fluoride to the cryolite we increase the output, and we effect the same result by increasing the current density, for it is evident that if more metal be deposited on the square inch the relative proportion of sodium fluoride which would be involved in the reaction, is reduced. It follows then that the output will be greatest when the current density at the cathode is great and when there is an excess of aluminium fluoride over that contained in the cryolite. This observation is entirely borne out by actual experiment.

A peculiarity of molten electrolytes, which at the beginning of electro-metallurgical art appeared to offer almost insurmountable difficulty, is their corrosive action on the electrolytising vessels. It is certainly very striking that, for instance, molten common salt passes easily through a cast-iron vessel of an inch thickness if the latter be at a dark red heat. The chemical activity of the dissociated ions of the common salt, which is enormously increased at the high temperature, accounts for the fact that no ordinary metallic vessel can withstand their aggressive action. This phenomenon confirms the modern hypothesis of electrolytes, for although the iron—in case of an iron vessel—does not ultimately combine with the chlorine ions of the sodium chloride, yet the chlorine ions in contact with the iron for an instant combine with it, and then immediately re-combine

with the stronger sodium ions. This continuous attack results in an entire disintegration of the iron. It is well known that it was only after applying internal electric heat, namely, by fusing and keeping in fusion the electrolyte through the heat generated by its own resistance to the passage of the current, that the electrolysis of molten compounds became an industrial possibility.

The electrolysis of molten aluminium compounds we can divide into three classes: first, the electrolysis of molten halogen compounds; second, the electrolysis of alumina dissolved in a molten solvent; and third, the electrolysis of the sulphide. Little need be said about the electrolysis of molten halogen compounds, or rather double compounds, of aluminium and an alkali metal, as there is no possibility of their ever competing with the method by which the alumina is directly reduced. This is self-evident. As to the second method of decomposing the oxide dissolved in a molten solvent, it is noteworthy that there are only three substances which in a state of fusion are capable of dissolving alumina. These are borax, cryolite, and lithium oxide. Borax is unsuitable, because in passing a current there is obtained an alloy of aluminium and boron, or boron alone—depending on current density. Cryolite is a solvent used in the present method. So much has been said about the present process that it will suffice to say that from a scientific and economic standpoint it is altogether the best method, and that the chief cost being that of the ore and of electrical energy, the development of the process will largely depend on the cheapening of the ore manufacture and the saving of electrical energy, which at present is about five times the amount theoretically required.

The fact that molten lithia dissolves alumina was discovered by the author, but although lithia is an excellent conductor in its molten condition, and though its affinity for oxygen, to judge at least from the heat of formation, and from the fact that lithium reduces silica at a temperature of 203° centigrade—neither the hydrate nor the oxide can be reduced by carbon—appears to be greater than that of any other element, yet the author has not hitherto succeeded in reducing aluminium by electrolysis in a molten bath of lithia containing alumina dissolved. The great cost of lithia appears at any rate to exclude its commercial use in the aluminium industry; nevertheless it would be interesting from a scientific standpoint if aluminium could be reduced in this way.

It remains to speak of the electrolysis of aluminium sulphide, patented by the author in Germany. As yet the electrolysis of the sulphide, although it offers some advantageous features, has not been able to show commercial superiority over the present method.

There do not exist any other compounds of aluminium that can be reduced electrolytically. And it appears that the development of the aluminium industry will depend rather on improving the present method than on any attempts at finding new processes, for we have seen that—perhaps with the exception of the fluoride—there are no compounds of aluminium existing that offer any probability of serving as a material for the industrial production of the metal. (See this Journal, 1883, 368.)

#### *The Separation of Metals from their Dilute Solutions.*

F. Mylius and O. Fromm. Ber. 27, 630.

See under XI., page 527.

#### *Results of some Trials made to Test the Extraction of Gold by Dilute Cyanide of Potassium with Different Samples of South Australian Gold Ores.* G. A. Goyder. Report to the South Australian School of Mines and Industries for 1893, 4—24.

**Method of Procedure.**—After digestion the solution of cyanide was displaced by water, and the ore washed till almost free from cyanide. The portion collected was evaporated to dryness and fluxed with a little litharge, and the resulting lead button cupelled, the gold and silver being parted, and weighed.

**Effects of Salts of Copper and Iron.**—Such tailings, as well as the one from which they were derived, involve a considerable loss of cyanide, which combines and forms double salts with the basic salts of iron and copper present, and thus prevents it from dissolving the gold.

**Effects of Washing with Water before Extracting.**—Nearly double the weight of the tailings in water was used for the washing before adding cyanide solution. Even then all the soluble salts of iron were not washed out. Though a stronger solution of cyanide was used, the extraction of 49 per cent. is very low.

**Beneficial Effect of Circulating Solution.**—Continuous circulation of the cyanide solution was found very beneficial, quite apart from time or extra time of contact. A continuous circulator was devised by the author for the laboratory, and is figured and described.

**Rapid Extraction of some Ores.**—Some, such as the Mount Torrens Central ores, though simply crushed and containing all the coarse gold and concentrates, were extracted to the extent of 85 per cent. of gold in only four hours, the cyanide liquor circulated amounting in weight to 27 per cent. of that of the ore treated.

**Effect of Uneven Packing of Wet Ore.**—Great stress is laid on the necessity for placing the ore evenly and so as to avoid channels in the percolators or leaching vats.

**Effect of Coarse Gold.**—Coarse gold is as a rule only dissolved by cyanide after long circulation.

**Observations of Rate of Solution of the Gold.**—A speck of gold only just visible to the naked eye in a good light was picked out of a sample. It measured 0.001 of an inch in its smallest breadth; taking the thickness at 0.001 of an inch, it would be 250 times as thick as gold leaf, and would take 3½ days (83 hours) to dissolve in dilute cyanide of potassium under favourable circumstances. This shows that coarse gold should be removed from the ore before it is subjected to the cyanide process, since only the finest gold is dissolved.

**Long Circulation.**—One lot was circulated for a week, and 91 per cent. of gold was extracted.

**Washing Out Cyanide Solution.**—A sample was saturated with 27 per cent. of its weight of 1 per cent. cyanide and allowed to stand 17 hours. Results of percolation were:—

First solution and first volume of wash yielded 6 dwt. 8 grs. of gold per ton.

Second volume of wash water yielded 4 grs. of gold per ton.

Third volume of wash water yielded ½ gr. of gold per ton.

It is thus evident that the cyanide solution of gold is quickly and thoroughly washed out of the ore by water. It is also found that in the case of ores not being packed evenly the washings must be prolonged in order to extract all the gold.

Twenty-four samples of South Australian ores were tried, and it was found that all except the "Eureka," could be advantageously treated by the MacArthur-Forrest process after having been passed through a battery fitted with the ordinary appliances for saving gold.

In working the cyanide process the following points are recommended for attention:—

1. The ores should be crushed so as to produce a minimum of slimes.
2. The ores should be packed evenly in the vats, and lightly.
3. The solutions should be allowed to percolate continuously, as the gold is dissolved much more rapidly when the solution is moving than when at rest.
4. Solutions containing about ½ per cent. of potassium cyanide are strong enough for most ores.
5. Ores containing copper or other metal forming double cyanides, require a stronger solution or a greater volume of the weak solution.
6. Ores containing double salts, which form double cyanides with KCy, should be treated with water to dissolve out the soluble salts, before cyanide treatment. If not, loss of cyanide results.—W. S.



*The Separation of Blende from Pyrites.* W. P. Blake. Trans. of the Amer. Inst. of Mining Eng., Aug. 1893; and Proc. Inst. Civil Eng. **115** [2], 61—63.

IN south-west Wisconsin large deposits of zinc ore occur in the form of sheets of blende and pyrites (marcasite), sometimes well separated, but often as a close mixture, which cannot be brought up to the proper standard for reduction by mechanical methods alone, the specific gravity of the two minerals being so nearly the same that a marketable concentrate cannot be obtained by jigging. Experiments in this direction were made by the Wisconsin Lead and Zinc Company, who erected two mills and turned out hundreds of tons of concentrates of different sizes, which were unsaleable owing to the high percentage of pyrites retained. Galena could be easily separated from such mixed ore, but blende and pyrites were inseparable, and neither was obtained in a marketable form, as the smelters of zinc ores in the Mississippi valley will not take those containing more than 7 per cent. of iron. The method devised by the author is similar to that adopted in tin dressing, the metallic minerals being first separated from the bulk of the earthy waste as a mixed concentrate, which is subjected to an oxidising heat in a Branton calciner, in which the revolving bed, instead of being a smooth cone, is stepped, forming a series of annular shelves 18 ins. broad, with a maximum diameter of 16 ft. at the bottom. The ore is fed through a funnel in the apex of the roof, and is discharged at the circumference of the bottom shelf. This treatment entirely or partially decomposes the pyrites, while blende and galena are scarcely, if at all, changed, the former mineral scarcely losing its brilliant lustre, whilst the latter is hardly tarnished. Beyond a slight breaking up of the fragments of blende by decrepitation, there is no change of form in the concentrate except as to the pyrites, which suffers a change of bulk with the loss of its sulphur. The pieces of marcasite swell, expand, crack, and exfoliate, becoming more bulky than before roasting; and the blende fragments, being reduced in size by decrepitation, a condition favourable to separation by jigging is obtained. The products of the latter operation are clean blende, or "dressed jack," and tailings, consisting chiefly of red oxide of iron, which serve as the basis of an excellent metallic paint, their value being enhanced by containing a small proportion of oxide of zinc. Any lead ore present is removed in the first compartment of the jigging machine, the furnace operation having split off the blende, leaving the galena free. This is another important advantage, as blende containing more than 2 per cent. of lead is unfit for the production of the best spelter. From a first raw concentrate, containing blende, 25 per cent.; pyrites, 25 per cent.; and galena, 5 to 10 per cent.; and the remainder dolomite and flint, after roasting and second jigging, marketable blende is obtained of a very good quality, assaying in the best samples over 62 per cent. of zinc, less than 3 per cent. of iron, and 1 per cent., or in some cases below  $\frac{1}{2}$  per cent., of lead.

Complete and even roasting is essential for the success of the process, as every particle of bisulphide of iron must be decomposed. Even a remnant or kernel of unchanged pyrites will cause the fragment to remain with the blende. The outer oxidised coating may be broken off and pass into the tailings but the unchanged part will not go over, and such imperfectly roasted ore will have to go back to the furnace. It has been found that by proper manipulation the pyrites, when reduced to the magnetic form by the removal of the second atom of sulphur, may be left in a condition to be attracted and removed by the magnet. Experiments have been made upon this subject by Mr. J. W. Meier, who finds that mixed ores, with 33 to 36 per cent. of zinc and above 10 per cent. of iron, may, by roasting in a muffle and magnetic separation, be easily converted into concentrates with 50 to 54 per cent. of zinc and only about 4 per cent. of iron. The chief difficulty is the conversion of the whole of the pyrites to the magnetic state, as fragments fully converted into ferric oxide cannot be removed by the magnet, and a partial reduction of these from ferric to magnetic oxide by the action of solid or gaseous carbon seems to be necessary after roasting. This method might probably be applied with advantage to the

ferruginous calamines of Wisconsin, it having been proved to be perfectly successful with similar ores at Monteponi, in Sardinia.

*Sulphur in Cast Iron.* W. J. Keep. Trans. Amer. Inst. of Mining Eng., Aug. 1893; and Proc. Inst. Civil Eng. **115** [2], 69—70.

THE conclusion finally reached is that the proportion of sulphur retained by grey cast iron cannot materially injure the iron except by increase in shrinkage, which in the extreme ends seems to be from 0.168 to 0.194 in. per ft. The general testimony is that most of the sulphur present in pig iron is lost in re-melting, and that it is impossible that it can be re-absorbed to any damaging extent from the fuel. The influence of sulphur is diminished by increase of carbon or silicon. In wrought iron, which is practically free from these elements, a small amount of sulphur is said to do great harm, and such iron will take up sulphur in considerable quantity. In steel containing less than 1 per cent. of carbon and practically no silicon, a few hundredths per cent. of sulphur cause decided injury. When carbon increases to saturation as in white cast iron, not more than 0.50 per cent. of sulphur can be retained, and as silicon increases, producing graphitic carbon, it is still more difficult to find sulphur in the metal, as it will not absorb it from the outside.

The influence of sulphur on all cast iron is to drive out carbon and silicon, to increase shrinkage, and in general to reduce strength, but in practice sulphur will not enter the iron in the foundry to a sufficient extent to realise these defects. When, as happens sometimes, iron which was grey when put into the cupola comes out white with increased shrinkage and ebull, and often with decreased strength, the change is due to loss of silicon, and can be remedied by re-melting with an appropriate addition of ferro-silicon or other highly-silicified metal.

That highly sulphurised iron cannot be produced in practice is evident from the fact that ores are used (in America) containing over 3 per cent. of sulphur with fuel containing nearly 1 per cent. of the same element; but all smelters are agreed that not more than 0.75 per cent. of sulphur can be found in white iron, 0.25 per cent. in mottled, and not more than a few hundredths in grey foundry iron. The experiments show that 0.50 per cent. of sulphur will not exert any appreciable deleterious influence, and even that may be at once corrected by a slight increase in silicon.

Remembering that the only noticeable effects would be to slightly increase the shrinkage, the above conclusion seems to be proved by the fact that, in a foundry working with a substantially uniform mixture, returning from 25 to 40 per cent. of its metal to the cupola to be re-melted over and over again each day, the castings will almost invariably have a less shrinkage than the average of the pig iron used, and yet they will probably contain more sulphur and less silicon than the pig iron originally did.

*Experiments on the Behaviour of Phosphoric Acid in the Blast-Furnace, and the Influence of Phosphorus on the Composition of Pig Iron.* N. Kjellberg. Jernkontorets Annaler, 1892, 180; and Proc. Inst. Civil Eng. **115** [2], 71—72.

UNTIL recently it has been generally supposed that the whole of the phosphorus present in a blast-furnace charge passed into the iron, and that no appreciable quantity was to be found in the slag. There must, however, be a point at which the iron becomes saturated, and the phosphorus must then combine with the slag. With basic slags, and at low temperatures, much of the phosphoric acid passes into the slag; but it is not easy to determine in advance how much of this impurity will be ultimately found in the slag, and how much in the iron.

In Sweden there are large deposits of iron ore rich in phosphoric acid, especially at Grängesberg and Gellivara, and the author's experiments were undertaken with the view of finding the best method of utilising these ores.

There are three main causes which determine the proportion of phosphorus in slag or metal, namely, the amount of phosphorus in the charge, the temperature in the blast-furnace, and the basicity or acidity of the charge. The experiments were made in a small blast-furnace with quantities of 1 tons of ore in each case. The ores were smelted without previous calcination, and their percentage of phosphorus varied from 0.2 to 3.6. Each sample of ore was run with basic and acid slags, and the difference in the composition of metal and slag determined in each instance. The author deduces the following conclusions:—

(1.) When the phosphorus in the ore does not exceed 1½ per cent., neither the low temperature in the blast-furnace nor the higher or lower proportion of silica in the slag has any appreciable influence on the reduction of the phosphoric acid. The greater part of the phosphorus of the ore combines with the iron, so that ultimately 90 to 95 per cent. is found in the pig iron and 5 to 10 per cent. in the slag.

(2.) When there is more than 1½ per cent. of phosphorus in the ore there appears to be a more definite division of the phosphorus between slag and iron. Up to a percentage of 3.5 the greater proportion of the phosphorus unites with the iron; but with a basic charge from 40 to 50 per cent. may be driven into the slag. With a high temperature and acid slag, about 95 per cent. of the phosphorus may be brought into combination with the iron.

(3.) There appears to be no volatilisation of phosphorus in the blast-furnace in the case of ores containing up to 3.6 per cent.

(4.) The carbon in pig iron diminishes as the phosphorus increases, especially when the latter is above 3 per cent.

(5.) The proportion of silicon also decreases as the phosphorus rises, and a pig iron containing 4 per cent. of phosphorus may not contain more silicon than steel. The specimens of iron containing most phosphorus were so brittle that the ingots could be broken by a light blow from a hammer.

In the case of ores containing 50 per cent. of iron, the pig iron from the blast-furnace will contain about double the percentage of phosphorus existing in the ore. When the percentage of iron in the ore amounts to about 60, it is possible to produce an iron with a smaller proportion of phosphorus when the basic process is employed. For foundry pig iron, with an amount of phosphorus not exceeding 1 per cent., 60 per cent. ores should be chosen with not more than 0.6 per cent. phosphorus. For pig iron to be treated by the Thomas basic process, and containing at least 2 per cent. of phosphorus, the percentage of that impurity in the ore should not be less than 1.6.

*The Action of Concentrated Acids on Certain Metals when in contact with each other.* George J. Burch and J. W. Dodgson. *Proc. Chem. Soc.* 1894. 137. 84—87.

IN 1790, James Keir (*Phil. Trans.* 1790, p. 367) announced that a mixture of strong nitric and sulphuric acids could be used for stripping the silver from old-plated copper goods. The authors have investigated the chemical and electrical phenomena of this and other reactions of the same type, and now present a preliminary account of their work.

*Experiments with a Silver-Copper Couple.*—A sheet of "pure" silver was cut into strips, which were scraped bright, bent into a U-shape, and weighed. To each was fitted a similar strip of copper, carefully cleaned and weighed, the whole forming an elliptical ring, held together by the elasticity of the metals. A number of test-tubes arranged in a water-bath contained the mixed acids diluted with various proportions of water. Each ring remained under the surface for 17 minutes, and was then rapidly removed, wiped, washed, and weighed. The liquid was not stirred during the experiment.

**SILVER-COPPER COUPLE, in a Mixture of 1 part NITRIC ACID of rel. dens. 1.436 with 5 parts H<sub>2</sub>SO<sub>4</sub> of rel. dens. 1.84 with various PROPORTIONS of WATER ranging from 1 to 10 molecules per molecule of H<sub>2</sub>SO<sub>4</sub>. Temperature 20° C.**

Molecular Proportions of Water.	Silver.			Copper.		
	Taken.	Loss.	Loss.	Per Unit Area.	Loss.	Taken.
None added.	0.7710	0.0425	0.01075	0.00027	0.0013	1.3265
1	0.7000	0.0358	0.00994	0.00061	0.0027	1.2082
2	0.7114	0.0234	0.00616	0.00038	0.0018	1.1320
3	0.7284	0.0251	0.00633	0.00075	0.0036	1.3252
4	0.7395	0.0170	0.00523	0.00250	0.0160	1.1351
5	0.7052	0.0052	0.00111	0.00288	0.0127	1.2209
6	0.7505	0.0110	0.00399	0.01792	0.0717	1.2671
7	0.7290	0.0176	0.00463	0.03475	0.1526	0.9775
8	0.7033	0.0343	0.00866	0.05116	0.2456	1.3216
9	0.6957	0.0268	0.00827	0.05302	0.2129	1.1854
10	0.6342	0.0300	0.00833	0.05835	0.2594	1.2055

In the stronger solutions, a yellowish-white powder separates from the silver, and a clear, heavy liquid of strong refracting power streams from it. Little or no gas is evolved.

During a few seconds after immersion the copper is covered with a white froth. This clears away, and the surface remains bright. Probably, nearly all the loss of copper occurs at this stage. When the dilution is represented by 8H<sub>2</sub>O + 11H<sub>2</sub>SO<sub>4</sub>, nitrous fumes first begin to appear. It will be observed that the amount of silver dissolved is least with 5H<sub>2</sub>O, after which it increases again, and that the copper, which is practically untouched at 3H<sub>2</sub>O, is now rapidly attacked. At temperatures above 50° C., the results are somewhat different.

*Magnesium-Silver Couple.*—In a mixture of 10 parts sulphuric acid of rel. dens. 1.84 with 1 part nitric acid of sp. gr. 1.436, the magnesium retained its brilliant appearance during an hour and 20 minutes, while the silver dissolved steadily. No gas was evolved. The magnesium lost 0.0015 grm., and the silver lost 0.1167 grm., the ratio of the loss per unit area being—

Ag : Mg :: 61.5 : 1.

*Electrical Phenomena.*—The influence of each metal on the rate of solution of the other is in most cases strongly marked. With the copper-silver couple the resistance increases rapidly on short circuit, apparently at the surface of the copper. The quantity of current is not proportional to the weight of silver dissolved.

**Sodium and Sulphuric Acid** (rel. dens. 1.84).—Sodium alone is less rapidly attacked by strong sulphuric acid at 16° than by water. The action was moderate but steady. Bubbles of gas were freely given off, and very little heat was evolved. The sodium was completely dissolved in about 20 minutes, a small quantity of a whitish substance being formed near it towards the end of the process.

The contact of another metal with sodium exerts an influence on the rate and manner of its solution in strong sulphuric acid. In most cases the action of the solvent is much retarded. The gas evolved comes from the *entire surface* of the platinum, carbon, or iron, when these substances are employed, and is given off freely from first to last. No bubbles appear on the sodium, which is clean and bright. The iron-sodium couple lasted 40 minutes, the platinum-sodium about two hours, and the carbon-sodium was still acting slowly after eight hours. In this case gas came from the sodium, and none from the carbon for the first 10 minutes after the initial effervescence was over. Then the sodium became quiescent, and bubbles rose from the carbon. Sulphur was separated after a while.

With lead and tin the action was similar, but much slower. The sodium was not so clean and the liquor became very turbid.

The tin acquired a brownish-yellow coating and the lead assumed a grey colour. It was several days before the sodium disappeared.

Zinc exerts comparatively little restraining action, but makes a very turbid solution.

An aluminium-sodium couple, weighted with a glass rod to sink it, evolved torrents of gas, and after about five seconds the sodium caught fire.

A magnesium-sodium couple similarly weighted gave a flash of light as it touched the liquid. Then, for about three seconds, there was a copious evolution of gas, and the sodium burst into flame while completely submerged.

With the cadmium-sodium couple the action was extremely slow. Fewer bubbles came from the sodium than from the cadmium, which became covered with a brown film. The liquor was turbid.

Coming now to those metals which produce a markedly different effect.

Nickel scarcely evolves any gas, but is soon coated *near the sodium* with a brownish film, while a few bubbles rise from the sodium. Antimony exerts the most powerful restraining influence of any substance yet tried. The initial action is very small. Bismuth behaves in much the same way. Both these metals become coated with a film near the sodium. Very little gas is evolved, and what there is comes from the sodium, which remains for days undissolved.

Silver in contact with sodium turns dark. Very few bubbles appear on either metal, but the sodium, which is quite clean, is visibly smaller after two hours.

Thus a magnesium-silver couple is almost inert in the acid, and a sodium-silver couple acts very slowly, but a magnesium-sodium couple bursts into flame, whereas either metal separately would be a long time in dissolving.

Copper evolves no gas, but becomes coated with the gas given off from the sodium, and presents a reddish fawn-coloured film. The sodium is brilliantly clean, and during the first stage of the reaction evolves gas slowly.

*The Oxidation of the Alkali Metals.* Wilmot Holt and W. Edgar Sims. Proc. Chem. Soc. 1894 [137], 95.

POTASSIUM and sodium (and probably lithium) are not attacked by perfectly dry oxygen, and may be distilled in it, without undergoing oxidation.

## PATENTS.

*Improvements in Extracting Gold and Silver from Ores and the like.* J. S. MacArthur, C. J. Ellis, and the Cassel Gold Extracting Company, Limited, all of Glasgow. Eng. Pat. 5218, March 10, 1893.

IN the MacArthur-Forrest process as heretofore used there is formed a sulphide soluble in the cyanide which retards and objectionably affects the action of the cyanide on the precious metals. This sulphide is now rendered inert by adding to the solution or to the ore, or to the mixture of ore and solution, a salt or compound of a metal which can form a sulphide practically insoluble in the cyanide. The following salts may be used, preferably in the order named, viz., salts or compounds of lead, such as plumbates; carbonate, acetate, or sulphate of lead; salts or compounds of other metals, such as sulphate or chloride of manganese; zincates; oxide or chloride of mercury; and ferrie hydrate or oxide. The proportion to be used in any case will depend on the proportion of soluble sulphide present, which is ascertained by a practical test on the laboratory scale.—A. W.

*Improvements relating to the Pickling of Metals and Alloys to remove Oxide and other Impurities from the Surface thereof.* A. J. Smith, Kingston-on-Thames. Eng. Pat. 6137, March 22, 1893.

INSTEAD of cleaning the surface of metals by the ordinary method of dipping in acids, the author uses the articles to be cleaned as the positive pole of a battery, the negative pole being either carbon or wire gauze electro-plated with silver and preferably covered with amorphous platinum black. The bath consists of dilute sulphuric acid with sometimes a depolariser such as a nitrate or chromic acid. By this means it is stated that the plates are better cleaned, with little, if any, "pitting" of the metal, and also that the offensive odours usually evolved are prevented when chromic acid is used. The electric current produced by this means is utilised by being passed into storage batteries for lighting purposes.—A. W.

*Improved Alloys for Use in the Manufacture of Wire.* A. Macdougall, Glasgow. Eng. Pat. 7301, April 10, 1893.

THE alloy consists of nickel, 2 to 3 parts by weight, zinc 10 to 14, and copper 83 to 88. The copper and nickel are melted together and the zinc afterwards added. The alloy is manufactured into wire in the usual manner; and the object of the above composition is to diminish the liability to corrode in making endless wires for paper-making machines and other purposes.—A. W.

*Improvements in the Manufacture of Steel, and in the Refining of Fluid Iron.* B. H. Thwaite, London. Eng. Pat. 8456, April 27, 1893.

THE ladle and a small converter are fixed at the ends of a bell crank lever with the open ends at right angles to one another, so that the swinging of the lever through a quarter of a circle backwards and forwards will alternately bring the mouth of one above the other. The whole is mounted on a carriage, a quantity of metal from the cupola is run into the ladle, the crank lever is turned, and the metal poured into the converter; herein it is blown with air or oxygen, and the ferro-manganese added if necessary. When the metal is ready the crank lever is turned back again and the metal poured once more into the ladle, which is then taken to the mould. The apparatus is more particularly applicable to and useful in the rapid treatment of comparatively small quantities of metal.—A. W.

**Improvements in the Treatment of Zinc Plates to be used as Substitutes for Lithographic Stones.** E. Forrest, New Southgate, and F. L. H. Bucholz, London. Eng. Pat. 8800, May 2, 1893.

The zinc plates, as pure as can be obtained commercially, are passed through highly-polished cold rollers to remove any surface marks. They are then placed in dilute nitric acid (1 part acid to 100 parts water). This brings to the surface of the zinc a blackish mass containing impurities, which is removed by washing with water and scouring with pumice powder. This is repeated until the nitric acid and water no longer produces any appreciable amount of the blackish mass. Such treatment renders the plates absorptive and removes all matters deleterious to lithography. They are then dried and subjected to the sand-blast process to produce a grained surface. When this has been done they are placed in a bath composed of 4 parts of nitric acid, 1 part of phosphoric acid, and 500 parts of water, after which they are washed with water. A second and sometimes third treatment with this solution and washing with water is resorted to. In some cases, when fine-grained plates are required, a bath containing 1 part of sulphuric acid in 100 parts of water is used instead of the mixture of nitric acid, phosphoric acid, and water, as the former in no way interferes with the grain. Finally, the plates are washed with a weak solution of alum (1 part of alum to 50 parts of water), after which they are dried and are then ready for use. The plates produced by this process are absorptive and sensitive to grease, and of a similar nature to lithographic stones. Moreover, they retain their light colour after being submitted to the aforesaid baths.—A. W.

**Improvements in Treating Malleable or Wrought Iron.** J. H. Ladd, London. Eng. Pat. 9406, May 10, 1893.

"CRYSTALLISED wrought-iron" is changed to a fibrous condition by subjecting it to the action of a gaseous composition consisting of air, hydrocarbon vapour, and water vapour in a state of combustion. The mixture should contain 3.395 grains of water vapour and 45 grains of hydrocarbon vapour (benzene) to the cubic foot. By subjecting the iron to this mixture during combustion, it is said to become carburised before it reaches its welding point, the amount of carburisation depending upon the length of treatment.—A. W.

**Method and Apparatus for Extracting Gold and Silver from their Ores by the combined Action of Amalgamation and Centrifugal Force.** A. Trug, Verespatak, Hungary. Eng. Pat. 25,071, December 30, 1893.

THE apparatus consists of an inverted cone-shaped vessel open at the top and fixed to a vertical shaft rotated by a pulley. Within the vessel is a distributing disc fixed to the shaft and provided with radial ribs and a circular rim. The ore-sludge is distributed by this and thrown with an upward velocity on to the inner surface of the cone-shaped vessel. This surface has annular ribs formed on it, which prevent the mercury falling down to the bottom of the vessel. By the rotation of the vessel the ore is carried upwards over these ribs to the top of the vessel, whence it is thrown off into an annular trough. The mercury containing the gold is washed down into the bottom of the vessel and through perforated plugs. Electricity may be used to maintain the mercury surface active, and this is done by connecting the tubes feeding the distributing disc with the positive pole of a current generator, the negative pole being connected with the surface carrying the mercury.—A. W.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

*The Separation of Metals from their Dilute Solutions.*  
F. Mylius and O. Fromm. Ber. 27, 1894, 630—651.

The authors have made elaborate researches on the phenomena occurring in the precipitation of one metal by another in dilute solution, and in the electrolysis of solutions of similar tenuity. Their conclusions are summarised under the following heads:—

### 1.—Precipitation of one Metal by another.

- (1) Metals are capable of union with each other at the ordinary temperature, at the moment of deposition.
- (2) Alloys are formed by the action of a metal upon the dilute solution of one electro-negative to it, and their formation can only be explained on electrolytic grounds.
- (3) The composition of the alloy corresponds with the solvent capacity of the negative for the positive metal, under the conditions of the experiment.
- (4) The resulting alloys are generally porous and may be either black and apparently amorphous, or crystalline; in the latter case their composition usually corresponds with some simple atomic relationship.
- (5)  $\text{Cu}_2\text{Cl}_2$ ,  $\text{AuCl}_3$ ,  $\text{Cu}_2\text{Sn}$ , and an alloy of platinum and lead can be obtained crystallised in the wet way.
- (6) Alloys obtained in this manner are in no way different from other substances insoluble in water in respect of their mode of formation and decomposition.
- (7) The alloys are decomposed and new alloys are formed by treatment with solutions of their negative metals.
- (8) The alloys are decomposed by acids with the evolution of hydrogen and separation of their negative constituent, behaving in this reaction like couples of low E.M.F.; the decomposition is generally incomplete.
- (9) The action of oxidising agents is usually confined at first to the positive constituent of the alloy, sharp separations being sometimes obtained in this manner.

### II.—Precipitation by an Electric Current.

- (1) The heavy metals may be separated more or less easily from their dilute solutions in the form of black, porous, apparently amorphous precipitates.
- (2) Silver and copper precipitated in this manner may occlude hydrogen during the passage of the current, but the greater part of the gas escapes on opening the circuit.
- (3) Silver and copper precipitates exhibit spontaneous change into the crystalline state, frequently accompanied by the evolution of hydrogen.
- (4) The conversion of the black into the crystalline modification is hastened by the action of metallic salts, acids, and oxidising agents, the metals being in this respect analogous to the alloys.
- (5) The black variety of copper containing hydrogen is essentially different from Wurtz's copper hydride.
- (6) An electrolytic method of formation of copper hydride no more exists than does a process of preparing the same body by means of zinc.
- (7) The black precipitate frequently observed on the zinc of a Daniel's cell consists of a copper-zinc alloy. The same body separates on the copper plate when the cell is exhausted.—B. B.

*The Action of Concentrated Acids on Certain Metals when in Contact with each other.* George J. Burch and J. W. Dodgson. Proc. Chem. Soc. 1894 [137], 84—87.

See under X., page 525.

*Quantitative Analysis by Electrolysis. The Electrolytical Determination of Lead.* A. Classen. Ber. 27, 163.

See under XXIII., page 547.

*Quantitative Electrolytic Estimation of Lead.* A. Kreichgauer. Ber. 27, 315—318.

See under XXIII., page 547.

### PATENTS.

*Improvements in and connected with Galvanic Batteries.* C. A. J. H. and H. E. R. Schroeder, London. Eng. Pat. 23,101, December 15, 1892.

The patentees claim an exciting fluid—to be used without a porous diaphragm—consisting of 0.5 to 1.5 parts of nitric acid, 2.5 to 4 of sulphuric acid with 10 to 18 of water, all by volume. They also claim the construction of "comb-like" carbon electrodes, by clamping a number of carbon plates together with suitable carbon distance-pieces, by a single bolt with nuts and washers protected by gilding or platinising. They claim further the use generally of gilded or platinised connections; the application of slate screws and nuts to galvanic batteries; and porous pots indented with deep hollows to reduce their resistance.

In certain cases it is proposed to have in one of the end cells of the battery, as many zinc or carbon plates as there are lamps to be lighted, and to run a separate wire between each lamp and its plate. The other terminals of the lamps to be joined by a single conductor to a single electrode at the other end of the battery.

It may be mentioned that in a single fluid cell working at 60° C., the inventors state that an electrolyte containing 1 volume of nitric acid, 3 of sulphuric acid, and 2 of water, is suitable for current densities of 5 amperes per sq. inch.

—E. T.

*Improvements in the Manufacture of White Lead.* S. Z. de Ferranti, London, and J. H. Noad, East Ham, Essex. Eng. Pat. 23,572, December 21, 1892.

See under XIII., page 532.

*Improvements in the Production of Alkali, and in Apparatus therefor, the said Apparatus being Applicable for Electrolysis generally.* J. Hargreaves, Farnworth-in-Widnes, and T. Bird, Cressington, Liverpool. Eng. Pat. 5197, March 10, 1893.

In carrying out this invention a solution of sodium chloride on one side of a porous diaphragm is subjected to the action of an electric current, while a gas, such as air and moisture, acts on the opposite or cathode side of the diaphragm; or where the production of a carbonate is required carbonic acid gas and moisture may be admitted to the cathode side, and if a combustion engine be employed for driving a dynamo for supplying the electric current, the carbonic acid gas for acting on the alkali may be derived from the products of combustion given off by the engine, any moisture required being evaporated and conveyed to the cell by the aid of heated air or other gas. To prevent polarisation metallic oxides or catalytic material, such as platinum, together with oxygen or an oxidising substance, may be applied to the cathode. A containing electrolytic cell is shown divided by a horizontal porous partition in contact with the cathode, above which the solution to be decomposed is retained, and below which is a space for the gas to be applied to the cathode as above described. The apparatus is of the type shown in Eng. Pat. 18,871, 1892 (this Journal, 1894, 250).—J. C. R.

*Improvements in and in Making Electrolytic Apparatus.* J. Hargreaves, Farnworth-in-Widnes, and T. Bird, Cressington. Eng. Pat. 5198, March 10, 1893.

This refers to the combined cathode and diaphragm described in Eng. Pat. 18,039 of 1892 (this Journal, 1894, 255). They propose to form such diaphragm by first depositing on the wire gauze a material such as lime mixed with a fibrous substance like asbestos, and then drying.

The whole is then dipped in silicate of soda or potash, whereby the lime is converted into an insoluble silicate binding the fibrous material to the gauze.—E. T.

*Improved Methods for Effecting the Electro Fusion of Steel and its Alloys.* B. H. Thwaite, London. Eng. Pat. 6000, March 21, 1893.

This invention relates to a method of effecting the fusion of ferro-carbon of steel and its alloys, by which fusion is effected economically and rapidly, and in such a way as to produce a continuous flow, without producing deterioration of the metal produced, which is described as follows:—"I superpose one crucible over another, the latter however may be an ordinary open hearth steel furnace hearth, or it may be a chamber having a calcined basic or acid lined hearth or bottom similar in method of formation to that of an open hearth furnace. At the bottom of the upper of the two crucibles there is an opening provided by and through which any fused metal in this crucible can flow into the lower crucible or chamber described as rapidly as fusion takes place; through the sides of the crucible I pass positive and negative carbon electrodes, which are so arranged that the scrap or other steel can be easily fed continuously. The voltaic arc, as well as the resistance to the current set up by the metal to be fused, soon provides the fusion temperature, and the metal flows into the second chamber, across which I may fix two or more electrodes, so that in this second chamber there is also established a fusion of steel melting temperature, by which the steel can accumulate in a molten condition. When sufficient metal has accumulated the tap hole is opened and the metal runs into a ladle to be cast into ingots in the ordinary way." There are drawings illustrating this invention, and methods of preventing oxidation, of effecting reductions of other metals, producing alloys, are also dealt with.—J. C. R.

*Improvements relating to the Pickling of Metals and Alloys to Remove Oxide and other Impurities from the Surface thereof.* A. J. Smith, Kingston-on-Thames. Eng. Pat. 6137, March 22, 1893.

See under X., page 526.

*A Process and Appliance for Electrically Treating Gaseous and other Bodies, a New Compound Gas made by such Process, and Apparatus for Producing and Consuming said Gaseous Product.* T. G. Hall, Chicago, County of Cook, U.S.A. Eng. Pat. 6551, March 28, 1893.

This invention, embracing 19 claims, is said to depend upon gaseous electrolysis produced in compound gases by certain electric couples, such as copper and iron, insulated from each other by a porous material, and built up as a stack in a chamber or chambers, which are heated, and through which the gases to be operated upon are passed, resulting in decomposition and recombination, producing a different order of things, said to be useful, for details of which the specifications must be consulted. Eng. Pat. 6551A 1893, is referred to.—J. C. R.

*Improvements in Secondary Batteries.* V. C. Doubleday, London. From M. Sussmann, Neuruppin, Germany. Eng. Pat. 6780, March 30, 1893.

The active material consists of a finely-divided metal—such as lead—or metallic oxide, mixed into a paste with india-rubber or gutta-percha solution, 5 to 15 per cent. of powdered pumice or other porous material being added.

The conductors used are flat plates from which numerous tongues are stamped out so as to stand up at an angle to the surface. The active material being spread over the whole plate and compressed, is held in position by these tongues, and at the same time a large conductive area of lead plate is provided.—E. T.

*Apparatus for the Artificial Production of Chalybeate Water.* O. March, London. Eng. Pat. 2362, May 10, 1893.

This invention relates to apparatus for the production of chalybeate water by means of electrolysis, water being employed as the electrolyte, with a pair of electrodes of iron, the anode of which supplies iron to the solution, similar to that found in many natural chalybeate waters, and has valuable medicinal properties.—J. C. R.

*Improvements in Connection with Electrolytic Apparatus.* H. Y. Castner, London. Eng. Pat. 10,584, May 30, 1893.

This patent refers especially to Eng. Pat. 16,046 of 1892 (this Journal, 1893, 768) where a sodium amalgam is formed by electrolysis of sodium chloride, and afterwards removed to another vessel to allow of the extraction of the sodium as caustic soda.

By the present invention the process is made automatic by using an electrolytic cell provided with compartments communicating with each other. This is caused to rock slightly by machinery at a suitable rate, so that at one time the mercury is in one compartment and amalgamating with the sodium formed by electrolysis, but a short time later is in another where the sodium is being extracted. A depth of mercury of about  $\frac{1}{2}$  inch is mentioned.—E. T.

*Improvements in Primary Voltaic Batteries.* W. Walker, jun., Birmingham, and F. R. Wilkins, Handsworth. Eng. Pat. 10,942, June 5, 1893.

A porous pot containing a zinc electrode suspended by lugs from its top edge, is surrounded by a wire gauze tube. Between this and the porous pot is packed gas carbon, powdered where in contact with the porous pot, in coarser granules next the gauze. The whole is supported in a tray so that any liquid percolating through the porous pot may keep the carbon moist, but if in excess it may collect in this tray. The carbon having in this way an enormous surface exposed to the air, takes up oxygen which acts as a depolarising agent to combine with the hydrogen produced during action in the cell.—E. T.

*Improvements in Apparatus for Electrolysis of Water.* Siemens Bros. and Co., London, and F. A. Obach, Old Charlton. Eng. Pat. 11,973, June 17, 1893.

From the top of a vessel constructed preferably of cast-iron, a cylinder projects downwards below the surface of the electrolyte, and is continued by an iron wire gauze tube to the bottom of the cell. The vessel is thus divided by the cylinder and gauze tube—which acts as diaphragm—into two co-axial compartments in which the two gases collect separately. The electrodes are tubes or shells of iron inside and outside the gauze diaphragm and suitably protected against the possibility of contact with it.

The electrolyte is a solution of caustic soda or potash, or a mixture of the two.—E. T.

*Improvements relating to the Electro-deposition of Metals.* J. Rudholzner, Munich, Germany. Eng. Pat. 2900, February 9, 1894.

“ARTICLES which are to be coated with metal by electrolysis and some parts of which are subject to greater wear than other parts necessitate care in the distribution of the metallic coating.” This invention sets forth improvements in obtaining varying thicknesses of deposits on articles to be coated with metal by electrolysis, by arranging in the electrolytic bath between the article to be coated and the anode freely suspended plates of insulating material, provided with larger or smaller apertures, so that the parts of the article which are directly opposite the apertures receive a thicker coating of metal than those parts which are protected (screened) more or less from the direct action of the current by the plates suspended in front.—J. C. R.

*An Improved Electric Insulating Compound.* L. Bærenhielm and A. Jernander, Stockholm, Sweden. Eng. Pat. 3045, February 12, 1894.

See under XII., page 531.

*Improvements in the Heating of Gases by Electricity.* W. Donaldson, London. Eng. Pat. 3223, February 14, 1894.

This invention relates to methods of heating gases by electricity by passing them through an elongated chamber or tube, in contact with an internal coil of wire or other partial conductor carrying an electric current, the resistance and resulting temperature being arranged so that it is of least intensity at the point where the gas enters and of greatest intensity where the heated gas issues.—J. C. R.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*Crude Wool Grease and Lanolin.* Chem. Ind. 17, 149—155.

ORDINARY sheepswool (unwashed) contains 12—23 per cent. of moisture, 31—43 of actual wool fibre, 3—23 of dirt, and the remainder, some 28—42 per cent., of a mixture of potash soaps and fatty matters the majority of which consists of cholesterol, isocholesterol, and analogues and ethers thereof (such as cholesterol benzoate, stearate, &c.) and some waxy matters, more particularly ceryl cerotate. The isolation of the fatty matters in a very crude form was described by Dioscorides, the product being designated *oxyppus* by Pliny in the early years of the Christian era; but the separation in a more complete way of the cholesterol and waxy constituents is of entirely recent origin.

The use of solvents for extracting the fatty matters, e.g., carbon disulphide, petroleum spirit, “benzine,” amyl alcohol, &c.), although promising enough *a priori*, has in practice not been found to work satisfactorily, the wool fibre being generally damaged during the operation of freeing it from the adherent solvent after the grease has been washed out, or otherwise deteriorated as regards its fitness for spinning; accordingly, the extraction methods and apparatus of Richter, O. Braun, Moisan, and others have now only historical interest. Similar remarks apply to some of the earlier methods adopted for utilising to some extent the waste liquors of wool washing, e.g., those of Kurrer, v. Westraumb and others, where the grease was precipitated by milk of lime, and the lime soaps, &c. thus obtained subjected to dry distillation for illuminating gas-making; although this process is still applicable to the residues left on filter pressing the grease recovered from the liquors by addition of acid. Bräsin first employed the fatty acids obtainable from the liquors for soapmaking, the lime precipitate being prepared by treating under 8 atmospheres pressure, and distilling off the unaltered cholesterol by superheated steam, the residual lime-soaps being decomposed by hydrochloric acid. Viallette and Vinchon endeavoured to convert wool-grease entirely into saponifiable matter by heating the dry grease with solid alkali to 250°—300°, air being excluded by means of an indifferent gas or superheated steam; in this way not only are all free fatty acids and glycerides converted into soaps, but further the cholesterol ethers are saponified and the fatty alcohols formed more or less completely converted into acids under the influence of the alkali with disengagement of hydrogen; so that on decomposition of the mass with acids, a mixture of various fatty acids results with but little of any other organic constituents. Thus ceryl alcohol forms a cerotate; and so on.

At the present time, the wash waters of woollen factories are usually simply treated in tanks with sulphuric acid, the quantity of which is best regulated by alkalimetrically



examination of an average sample of liquor; usually about 0.4 per cent. of acid of 66° B. is requisite. The liquors are generally of two kinds mixed together, viz., the liquors from the "leviatans" or washing machines for raw wool, consisting essentially of watery soap solution with emulsified wool-grease suspended therein; and the purer liquors obtained from the further washing of spun and woven goods where soap is employed as detergent, whilst oily matter applied to the wool to facilitate spinning, &c. (olive oil, &c.) is washed out. After acidulation, waste steam may conveniently be blown in to facilitate the separation of the fatty acids, &c. ("magma") from the watery fluid. After draining and filter-pressing (first cold, then hot) about 50 per cent. of the crude magma remains as a dirty press-cake of which the following may be taken as the average composition (Deite). Water, 10.66. Fat, 34.74. Organic matters, 22.37. Sand and dirt, 30.32. Soluble silica, sulphuric acid, phosphoric acid, oxide of iron, alumina, lime, magnesia, alkalis—1.91. Total, 100.00.

The other half that passes through the filter is a watery fat requiring refining. As a preliminary operation the grease is boiled for an hour with its volume of water containing 2 or 3 per cent. of sulphuric acid, for which purpose wooden vats provided with steam coils are best suited. After standing at rest for two hours the watery liquor is run off, and the grease boiled up with plain water to wash out entangled acid. After standing at rest for 12 or 14 hours the grease is carefully drawn off from above and dehydrated by blowing superheated steam through the mass. The dark product requires bleaching, for which purpose potassium bichromate and sulphuric acid give good results according to some, whilst this is not confirmed by others. The bleaching liquor consists of 1 part  $K_2Cr_2O_7$  of acid at 66° B. and 10 to 12 parts water; sodium bichromate has lately come into use instead of the potassium salt, being cheaper and more soluble. Instead of sulphuric acid, the equivalent quantity of hydrochloric acid may be employed. In any case the temperature should not exceed the fusing point of the grease by more than a few degrees, otherwise the emulsifying of the grease is more difficult, and the bleaching action correspondingly hindered, larger visible globules and droplets being formed instead of minute or microscopic ones. The grease and bleach liquor are well agitated together for 20 minutes by hand or machinery; the green aqueous fluid is run off, and the purified grease washed by agitation with hot water; it then consists of a mixture of free fatty acids, glycerides, and cholesteroid bodies.

Vohl's process for extracting grease from wool-washing liquors consists in treating the liquors with calcium chloride solution, so as to form insoluble lime soaps which carry down with them the cholesteroid bodies, &c. The precipitate is collected and decomposed by hydrochloric acid, and the fatty matters that separate again boiled up with water containing a little acid; on standing, three layers form, viz., watery solution of hydrochloric acid and calcium chloride at the bottom, clean grease at the top, and an emulsion in between, the working up of which presents difficulties. Solution of fatty matter therefrom by light petroleum spirit ("benzine"), and distillation to recover the grease and spirit, may be employed. Magnesium sulphate may be used instead of calcium chloride. Neumann employs milk of lime as a precipitant, followed up by addition of magnesium sulphate and ferrous sulphate; a perfectly clear effluent is thus obtained on filter-pressing, the press cake being then treated with acid as before.

The grease resulting from one or other of these processes was formerly employed as a lubricating material or for the manufacture of coarse soaps; and more recently was frequently worked up by distillation, and separation of so-called "oleine" and "stearine" (respectively liquid and crystalline constituents). The increasing demand for lanolin, however, renders it advantageous to extract the cholesteroid bodies by separation from the free fatty acids, &c. This may be effected in various ways, usually depending on the conversion of the fatty acids into soaps insoluble in solvents (e.g., acetone), which readily dissolve the cholesteroids. O. Braun's process, however, is a mechanical one, the wool-fat emulsion in soap solution being

agitated in a centrifugal machine so as to separate the suspended grease from the soap-water, somewhat as in creams separation from milk. The wool liquors, for example, may be partially precipitated by alkaline-earth salts (especially calcium chloride), the precipitated lime-soap carrying down the cholesteroids, which may then be separated by means of acetone, which leaves the soaps undissolved. Or the grease may be previously separated, purified by solution in benzene, carbon disulphide, &c., so as to separate dirt, and then treated with acetone, &c., to dissolve out cholesteroids. Details as to the exact methods employed in the somewhat difficult and troublesome process of refining wool-fat are not obtainable from extant publications; the descriptions given in various patent specifications form a chaos, out of which it is difficult to decide accurately which points are, and which are not, of actual practical utility.

In commerce two preparations occur, substantially consisting of the purified cholesteroids, viz., "anhydrous lanolin" (*Lanolinum anhydricum* or *Adeps lanae*); and "lanolin," a product containing some 30 per cent. of water, obtained by kneading the anhydrous substance with water. The former should be perfectly neutral, not becoming rancid by exposure to air, yellowish-white in colour, and of the consistence of a stiff salve. It should be free from all traces of chlorine, metals, glycerol or glycerides, soaps, and saline matters; no mechanically intermixed impurities or colouring matters should be present, nor should it have any disagreeable odour. On rubbing on blue litmus paper no reddening should occur; no residue should be left when incinerated on platinum foil and it should be perfectly soluble in ether. The hydrated product should possess corresponding properties. Pure lanolin for medicinal use when melted in a steam bath with 5 parts of water for half an hour should be free from froth; the aqueous liquor should not give more than 0.02 per cent. of residue on evaporation, and the fat should melt at 38–40° C. 0.05 gm. lanolin, 5 cc. chloroform, and 5 cc. sulphuric acid should produce a deep brownish-red colour at the junction of the two fluids.

The peculiar absorptive action of the human skin as regards lanolin renders this substance specially useful as a medicinal unguent; on rubbing on to the top of the head corrosive sublimate lanolin salve (1 in 1,000), a metallic taste becomes perceptible on the tongue after a few minutes. An increasing demand exists for lanolin as a constituent of toilet and medicinal soaps, and for the introduction of certain sulphur compounds into the body it appears to be well fitted.—C. R. A. W.

#### Manufacture of Oil and Food from Peanuts. United States Consular Reports 1894, 44, 683.

The report deals with the trade in peanuts, as a source of oil and as a food, in Germany, where there is a considerable consumption of this material. The import of peanuts into Germany for 1893 was 20,973 tons, 7,040 tons coming from India; 8,471 tons from West Africa; and 5,431 tons from East Africa. The best peanuts brought into the market are from Senegal, and the worst from Madras. The following are recognised commercial grades. Senegal, 51 per cent. oil; Congo, 49 per cent.; East African, 49 per cent.; American, 42 per cent.; Bombay 44 per cent.; and Madras 43 per cent. The nuts are generally imported in the shell from West Africa and from America, but the kernel only is often shipped from East Africa and India. The saving of freight by this practice is counterbalanced to some extent by the greater tendency of the unprotected kernels to become rancid on the voyage. The shells, which amount to about 23 per cent. of the total weight of the dried nuts, are used in Germany for making paper and for cattle food. The oil is obtained by three crushings, the first yielding 40 per cent. of oil, the second 4 per cent., and the third 2–3 per cent. Cold-pressed oil from good peanuts is used for eating instead of olive oil, and fetches 14.75 to 26 dollars per 100 kilos. The last pressings go for soap-making and "other technical purposes, among which is included the 'fattening' of oleomargarine." The American peanut is larger, sweeter, and, when roasted, of better flavour than the others, but the oil is only of medium quality.



The oil-cake sells for 30—33 dollars per ton and is used for cattle-food, but is said to be too rich and oily for working animals, e.g., horses.

Peanut meal contains so large a percentage of nitrogenous matter that experiments have been made to determine its suitability for army and navy rations and as a cheap food for the labouring classes. Analysis of "peanut grits" made from the residue obtained after the expression of the bulk of the oil, showed it contained: nitrogenous matter, 47.26 per cent.; oily matter, 19.57 per cent.; non-nitrogenous matter (other than oil), 19.06 per cent. This, therefore is richer in nitrogenous matter than are soyabean which have been hitherto considered as a typically rich material of this class. As the wholesale price per pound of peanut meal is 4 cents, this material forms certainly a cheap nitrogenous food.—B. B.

### PATENTS.

*Laundry Glaze.* John Conroy, Bebington. Eng. Pat. 9433, May 11, 1893.

The use of the following mixture is claimed for the manufacture of a laundry glaze, viz.:—

	Lbs.
French chalk.....	35
Barilla ash soup.....	2
Borax.....	$\frac{1}{2}$
Resin.....	$\frac{1}{2}$
Water.....	15

This mixture is dried and powdered, or made into a paste, if desired.—J. J. K.

*Improved Process of Producing Consistent and Semi-consistent Fats and Oils without applying heat.* G. Pommerhanz, Vienna. Eng. Pat. 9882, May 17, 1893.

This is based on the property possessed by wool-fat or grease of incorporating with most salts, and of solubility in mineral oils, which are solidified by it. The degree of solidification is increased by the addition of caustic soda. Seventy lbs. of mineral oil are thoroughly mixed in an agitator with 20 lbs. of melted wool-fat, and 3 lbs. of caustic soda lye of 35° B. with 6 lbs. of common salt (dissolved in as little water as possible) are then added. A stiff yellow mass is formed, suitable for lubricating machinery, and greasing yarn or leather.—J. J. K.

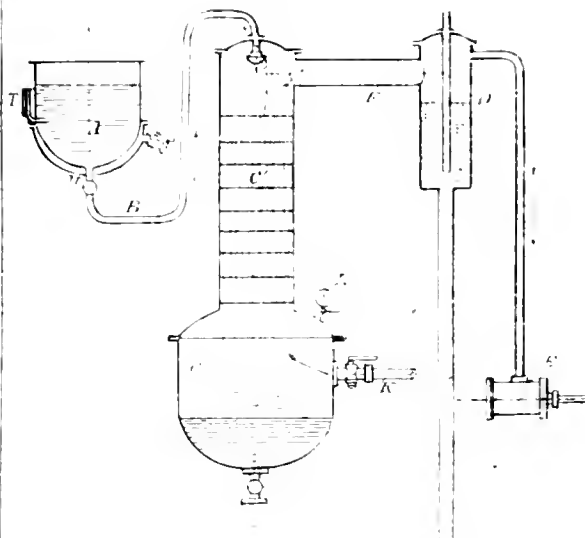
*A Process of Improving or Preserving Oils and Fats.* E. Watel, Paris. Eng. Pat. 12,847, June 30, 1893.

This invention consists in the improvement of the quality and better preservation of oils, by eliminating from them volatile matters which impart odours or flavours, and traces

of water left in, when the oils are extracted by pressure or fusion. Two methods are described, intermittent and continuous. Fig. 1 shows the apparatus in which the oil is purified intermittently; the fatty material is heated in the steam jacketed boiler A to 140° F., when it is run through the pipe B into the closed apparatus C, in which a vacuum is maintained by the condenser D and the vacuum pump E through a pipe F. Dry steam is then introduced into C through the pipe H and the injector I, when the volatile matters and water are driven off into the condenser D.

The continuous method is shown in Fig. 2, where the melted fat or oil runs down a column C', and is met by the

Fig. 2.



steam entering the chamber C, through the pipe K. Vacuum indicators L, thermometer T, and glass plates G, enable the operation to be watched.—J. J. K.

*An Improved Electric Insulating Compound.* L. Baatnhielm and A. Jernander, Stockholm, Sweden. Eng. Pat. 3045, February 12, 1894.

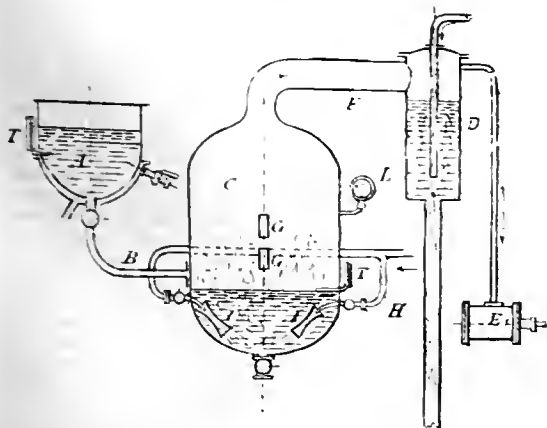
This invention relates to the production of a compound, consisting chiefly of vulcanised stearin pitch, from the pitchy residuum obtained in the distillation of fatty acids, which is a good insulating material, but at the ordinary temperature it becomes soft and sticky, whilst in the cold it is brittle and cracks. To obtain a more suitable product, 74 parts of stearin pitch are mixed with 25 of sulphur, for a hard material, or 70 parts of stearin pitch, with 20 of linseed oil and 10 of sulphur, for a soft material, and respectively heated to a temperature of from 120° to 175° C.—J. J. K.

*A Superfatted Biniodide of Mercury Soap.* C. R. Ungworth, West Kensington, London. Eng. Pat. 12,884, July 1, 1893.

This is an improved medicated soap, for use in cases of infectious fevers, skin disorders, and disinfection generally. Any superfatted soap is dehydrated and thoroughly incorporated with about 1 per cent. of a solution of mercury biniodide in potassium or sodium iodide, and the mass compressed for the purpose of stamping into tablets.

—J. J. K.

Fig. 1.



### XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

#### (4.)—PIGMENTS, PAINTS.

##### PATENTS.

*Improvements in the Manufacture of White Lead.* S. Z. de Ferranti, London, and J. H. Noad, East Ham, Essex. Eng. Pat. 23,572, December 21, 1892.

This invention relates more especially to improvements on the process described in the specification of a former Eng. Pat. 6049, 1892 (this Journal 1893, 363). In this process acetate of ammonia is decomposed by electrolysis with lead electrodes, yielding a lead solution and caustic alkali, which is afterwards carbonated, when on mixing the two solutions white lead is precipitated. As the process was formerly conducted the whole of the lead could not advantageously be precipitated by the carbonated alkali, but a certain amount had to be left in solution, to be afterwards removed by the addition of chromic acid, by which the cost of production was increased. By means of the present invention the inventors claim the immediate precipitation of the whole of the lead in solution as white lead. This is effected by heating the lead solution and precipitating vessel lead from the hot solution, employing in the mixing vessel an excess of ammonia over and above that which results from electrolysis. The excess of ammonia is afterwards separated by distillation, the filtrate being sprinkled over a steam-heated coil, after the precipitated white lead has been previously separated by a filter press. Volatilised ammonia and any ammonium carbonate is thus condensed, whilst the acetate of ammonia solution is stored up for use again in the electrolyser, which is constructed of lead plates locked up together in a frame, as in a filter press, the plates being separated by porous diaphragms, which are made preferably of stout Willesden paper, insulated at the bottom to prevent irregular electrolytic action. In the electrolyser, with a series of cells with exit openings at the bottom, elastic bags distended by fluid within, are so arranged as to close these openings.—J. C. R.

*Improvements in Apparatus for the Manufacture of Metallic Pigments or Compounds, and in the Production of Lead and Zinc Pigments or Compounds and Combinations of these by Means of such Apparatus.* A. C. J. Charlier, Glasgow. Eng. Pat. 7119, April 6, 1893.

This patent is a continuation and enlargement of Eng. Pat. 7117, 1893 (this Journal, 1894, 407), air being introduced into the oxidising chamber of the apparatus under pressure, and the producer furnace being built in an elliptical instead of rectangular form. The plant is also used for the manufacture of "marine white," a mixture of lead sulphate and zinc oxide, by introducing into the furnace, together with the lead ore, some material containing zinc, so that the two may be volatilised and condensed together. It is, moreover, available for the preparation of zinc oxide or carbonate alone by heating the furnace up to the temperature of about 1,300° C., throwing in the zinc ore, and drawing the metallic fumes and furnace gases by means of an injector up a vertical tower and through a water condenser. In the water pumped off from the precipitated carbonate are found soluble zinc salts, which are precipitated by alkaline carbonates. The zinc carbonate may be used as a pigment or converted into oxide in the usual manner.

—F. H. L.

*Improvements in the Manufacture or Production of Lamp-black.* R. Irvine, Royston, Granton, near Edinburgh. Eng. Pat. 68, April 25, 1893.

TARS or oils rich in carbon are sprayed over incandescent coke, and the permanent gases so produced mixed with air and steam, or steam only, are burnt in the presence of a limited supply of air to produce lampblack.—F. H. L.

*Improvements in the Manufacture of Pigments.* O. Imray, London. From J. Sachs, C. E. Meier, and N. Gerstendörfer, New York, U.S.A. Eng. Pat. 166, January 3, 1891.

In order to make cotton and linen absorb certain dyes it is necessary to treat them first with albuminoid substances. Such a process is known as "animalising," and the inventors have discovered that a similar process may be employed in the case of mica and other minerals, which renders them available for use as the base of various pigments. The finely-divided mica is washed and introduced into a solution of egg albumen, dried, and then treated with steam at a temperature slightly above 100° C. to coagulate the albumen. So treated it will readily take up any aniline colour, no mordant being required, but for wood extracts aluminium or tin salts may be used. The albumen may also be coagulated by acid vapours or it may be replaced by a faintly ammoniacal solution of caseine. The dyed mica has a beautiful lustre, which may be increased and rendered more metallic by incorporating with it some well-polished metallic powder, such as bronze powder or bisulphide of tin, &c.—F. H. L.

### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

##### PATENTS.

*Improvement in the Art or Process of Waterproofing Leather, and in the Compounds employed in the same.* E. H. Lewis, St. Louis, Missouri, U.S.A. Eng. Pat. 3615, February 20, 1894.

THE finished leather is made pliable and waterproof by coating it with a compound of rubber-paste or caoutchouc, 3 parts; spirits of turpentine, or its equivalent, 2½ parts; and naphtha or other volatile solvent, 2½ parts. It may be found desirable to sprinkle soap-stone dust or other absorbent material on the prepared side of the leather just before using it.—A. G. B.

*Improvement in the Art or Process of Waterproofing Leather.* E. H. Lewis, St. Louis, Missouri, U.S.A. Eng. Pat. 3616, February 20, 1894.

THE leather is immersed in a bath of equal parts of wax (beeswax, paraffin wax, or spermaceti) and a volatile solvent vehicle, such as naphtha, heated to 150°–170° F. for 5–10 minutes, and allowed to dry. When the leather has been tanned "under the chemical or hot liquor process," and therefore has a very dry grain, it is well to add turpentine to the bath; this will make the chemically-tanned leather pliable, so that it can be successfully operated upon by the channeling machine.—A. G. B.

*Process and Compound for Waterproofing the Soles of Shoes.* E. H. Lewis, St. Louis, Missouri, U.S.A. Eng. Pat. 3617, February 20, 1894.

THE following compound is applied over the welt and insole, or over the seams, joints, peg-holes, &c.:—2½ lb. of wax are melted and 3 lb. of powdered talc, stearite, or soapstone are mixed therewith; 4 pints of rubber-paste or caoutchouc (Brazilian gum) are then incorporated with the mass.

—A. G. B.

## XV.—MANURES, Etc.

## PATENT.

*Improvements in the Manufacture of Fertilisers.* J. J. Seldner and John Watson, jun., Baltimore, Md., U.S.A. Eng. Pat. 2624, February 6, 1894.

This relates to the utilisation of refuse hair, leather, and similar material in the manufacture of high-class nitrogenous manures. One method is to put the hair, &c., with a weak solution of sulphuric acid into a closed steam-jacketed digester, and heated by means of the steam jacket until there is a pressure of 30 lb. in the digester, when the water in the acid solution is vaporised; the fibre is destroyed, and a pulpy mass produced. Alkaline matter, phosphate rock, or bone is added to neutralise the free acid left in the mass, then the mixture is dried, and forms a nitrogenous manure containing about 3 per cent. of ammonia.

A preferable method is to mix the hair, &c., with dissolved bones or superphosphate of lime (which contains enough free acid to disintegrate the hair), with the addition of water, and stirring the mixture by an agitator in the digester during the heating.—J. J. K.

## XVI.—SUGAR, STARCH, GUM, Etc.

*Crystalline Levulin.* E. Schulze and S. Frankfort. Ber. 27, 1894, 65–66.

THE authors have isolated a carbohydrate by the process described in the preceding abstract from the stalks of rye gathered before the commencement of seed formation. It resembles levulin in most of its properties, but is crystalline—a difference which may perhaps be due to its superior purity. It is optically inactive, and does not reduce Fehling's solution; it gave numbers on analysis indicating the formula  $C_6H_{12}O_{11}$ . When treated with acid it yields levulose. Until the identity or otherwise of this carbohydrate with ordinary levulose is definitely settled, the authors propose provisionally to denote it  $\beta$ -levulin.

—A. R. L.

*Estimation of Calcium Sulphide in Animal Charcoal by means of Bromine.* P. Herrmann. Die Deutsche Zuckerind. 1893, 18, 225.

See under XXIII., page 546.

## PATENTS.

*Process for obtaining Para-phenetolcarbamide ("Dulcin").* J. Wetter, London, from J. D. Riedel, Berlin. Eng. Pat. 10,850, June 2nd, 1893.

See under XX., page 542.

*Improvements in Refining Sugar.* E. Langen, Cologne, Germany. Eng. Pat. 3782, February 21, 1894.

THIS is a process for obtaining an artificial massecuite by the admixture of syrup with raw sugar, and the subsequent subjection of the mass to crystallisation in motion. The syrup may be concentrated either before or after the introduction of the raw sugar.—A. R. L.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*Dissemination of Saccharose in Seeds of Plants.* E. Schulze and S. Frankfort. Ber. 27, 1894, 62–64.

THE authors employ the method already described by one of them (Schulze, this Journal, 1893, 347) for the isolation of saccharose from seeds. The seeds are digested with 90 per cent. alcohol, and strontium hydroxide added to the alcoholic extract the precipitate being decomposed by carbonic anhydride. The saccharose was generally obtained in the crystalline condition, being purified by repeated crystallisation from dilute alcohol. It was identified by its form, taste, rotatory power, reaction with resorcinol after treatment with hydrochloric acid (levulose reaction), and its behaviour towards invertase and Fehling's solution. In this way saccharose has been recognised in the seeds of wheat, rye, oats, buckwheat, hemp, sunflower, pea, soja-bean, and coffee. Stirling and Morawski (Monatsh. Chem., 7, 76), have proved the presence of this carbohydrate in soja-beans, whilst Stenhouse, Graham and Campbell, and Ewell, showed that it occurs in coffee. Saccharose has likewise been recognised by numerous observers in the seeds of field-bean, scarlet-runner, barley, maize, pig-nut, hazel, wallnut, and almond; the author confirms its presence in these as well as Richardson and Crampton, to whom the observation is due that saccharose occurs in wheat germs.

The green pods of the pea contain saccharose, a fact of some interest, inasmuch as it is probable that the sugar passes thence into the ripe seeds where it is converted into starch.—A. R. L.

*The Preservation of Wines by means of Sodium Silicofluoride.* L. Surre. Monit. Scient. 1894, 3, 258.

THE author has detected in a wine, which he had occasion to examine for antiseptics, the presence of fluorides. A sample of the preservative employed was obtained, and found to consist of sodium silicofluoride.—H. T. P.

*Note on the Fermentation of Molasses.* J. Effront. Monit. Scient. 1894, 3, 161–165.

IN distilleries employing molasses as raw material serious losses frequently occur owing to the marked resistance to complete fermentation exhibited by particular samples. Fermentation in such cases proceeds very irregularly, and, when apparently complete, the lyes still contain considerable quantities of unaltered sugar. The potential loss of alcohol thus incurred must not be confounded with the losses arising from actual destruction of alcohol or sugar by the undue development of acid ferments, the use of inferior yeast, &c. The peculiarity in question is inherent to the molasses, and is generally ascribed to the presence of a free volatile acid (which retards fermentation), and, in a smaller degree, to an excess of salts (nitrates, &c.). The various means, based on this theory, suggested to render such molasses easily fermentable, all aim at removing the free acid, either by neutralisation or by boiling, with or without the addition of sulphuric acid, and subsequent neutralisation of the latter. According to the author, the benefit accruing from such treatment, although occasionally well marked, is very often nil. Experiments made with good and bad samples of molasses are quoted, which show that:—

1. There is practically no connexion between the percentage of volatile acid and the fermentability of molasses.
2. The removal of the acid is advantageous only when its amount is unusually large.

The cause of the non-fermentability of certain molasses must be sought elsewhere, and is shown by the author to be due to the presence of a particular micro-organism. This view is based on the following observations:—

1. A trace of difficultly fermentable molasses added to a solution of normal molasses renders the whole difficultly fermentable.
2. This effect is not destroyed by boiling the mixture.
3. The effect is greatest when inoculation is made before fermentation.

The author has succeeded in isolating the organism and finds it to be essentially anaerobic in character. Grown in a solution of molasses it forms small viscous masses, which under the microscope are seen to consist of aggregations of short rods embedded in a gelatinous medium. Cultures of the organism added to sterilised solutions of normal molasses rendered them in every case difficultly fermentable. The organism possesses in an eminent degree the power of resisting the influence of heat and antiseptics. Its suppression in worts by the liberal addition of antiseptics is, therefore, not practicable, since the large amount necessary would deleteriously affect the yeast. Its removal is best effected by some method of clarification. For this purpose, the solution of molasses is diluted to a strength of 18 Balling, slightly acidified, and treated with tannin at the rate of 25–35 grms. per 100 kilos. of molasses. The solution is then, preferably, raised almost to the boiling point and allowed to cool. The precipitate which settles carries with it all the bacterial cells, and the supernatant liquid when decanted off will be found to ferment quite normally. Pieric acid may also be used as precipitant with excellent results, but its presence is inconvenient in the subsequent production of "vinasses." Other clarifying agents, such as white of egg, alumina, &c., may also be successfully employed.—H. T. P.

*The Preservation of Wines by means of  $\beta$ -Naphthol Sulphonic Acid.* Sinibaldi. Monit. Scient. 1893, 7, 842–844.

The calcium salt of this acid ( $C_{10}H_7OSO_3$ )<sub>2</sub>Ca + 2H<sub>2</sub>O, which has been previously described under the names of *asiprol* (this Journal, 1892, 772) and *abristol*, has been found to exert a remarkable preservative influence on wine, entirely preventing as it does, even under the most trying circumstances, the development of acidity, &c. Moreover, Dujardin-Beaumetz and Staekler have shown that the new antiseptic is perfectly harmless to the consumer, even when used in much larger quantities than necessary for the preservation of wine (10 grms. per hectolitre). As regards the legal aspect of the question, it seems probable that the addition of *abristol* to wine must be prohibited if the letter of the law be adhered to. Possibly a limit of toleration may be permitted, as in the case of salt and potassium sulphate (see next abstract).—H. T. P.

*Note on Abristol.* E. Noelling. Monit. Scient. 1894, 2, 257–258.

ABRISTOL, the calcium salt of  $\beta$ -naphthol sulphonic acid,  $Ca(C_{10}H_7OSO_3)_2 + 3H_2O$ , has been latterly employed with excellent results for the preservation of wines. In spite of the complete harmlessness of the product, considerable objection has been raised by the French press, &c. to its use, opposition being offered to the addition of all chemicals, however harmless, in the case of alimentary substances. As the subject is one of those under discussion before the "Comité d'Hygiène de France," the author takes the opportunity to bring forward the evidence in favour of the harmlessness of *abristol* from a hygienic standpoint. The maximum quantity of *abristol* necessary for the preservation of wine is 10 grms. per hectolitre. Dujardin-Beaumetz and Staekler have, however, shown that 6 grms. of *abristol* may be taken internally per diem without any ill-effects. As regards the possibility of noxious decomposition products being formed from *abristol*, the only reaction that need be considered is the possible formation of free naphthol and potassium sulphate. The quantities of these substances obtainable by the decomposition of 10 grms. of *abristol* are 6 and 8 grms. respectively. Potassium sulphate is tolerated by law in wines up to 2 grms. per litre; whilst naphthol is even more harmless than *abristol* itself, and may be taken in doses up to 26 grms. without risk. From these facts it appears that the addition of *abristol* to wines might with advantage replace the usual practice of "plastering," since by the latter operation considerable quantities of potassium sulphate are formed, a substance which has been shown to exert a distinctly unfavourable influence on the human organism.—H. T. P.

*Aeration: Its Effect in Brewing.* L. Briant. Trans. North of Eng. Inst. Tech. Brewing, 1893–94, 3 [2], 23–32.

The value of aeration is now universally admitted, and there are few breweries in which it has not been adopted. Dealing with the contamination side of the question, the author quotes some figures showing the number of micro-organisms contained in the air at various seasons and at different altitudes from the ground. As these micro-organisms include bacteria, wild yeasts, &c., the desirability of preventing infection is obvious; but the means at one time adopted for this purpose—of discarding the coolers entirely, and using only internal refrigerators—are not commendable (and quickly fell out of use) because aeration is reduced to a minimum, and the resulting beers "drop bright" with difficulty.

Aeration is of two kinds, hot and cold. The former is a chemical process, i.e., the oxygen of the air is absorbed and combines with the wort constituents. During the latter, oxygen is mechanically dissolved by the wort. The precise effect of hot aeration, and the manner in which the oxygen is fixed, are not known. But the author disposes of the theory—accepted by many—that certain nitrogenous constituents of the wort are precipitated thereby. Experiments are quoted which show that the nitrogen percentage is practically unaltered by aeration. Aerated wort, however, is always slightly darker in colour, and settles bright readily as compared with non-aerated wort. The latter, in fact, retains a persistent opalescence (due to finely-divided hop-resins) which extends to the resulting beer, causing a turbidity which is difficultly removable, even by finings. Taking into account these facts, as well as Hayduck's researches on the hop-resins, it is probable that the oxygen absorbed combines with the carbohydrate constituents of the wort, and partly with the hop-resins, causing the latter to agglomerate and deposit readily. Hot aeration takes place best at temperatures ranging from 180°–200° F., i.e., when the wort is on the hop-back and early on the coolers. The author regards as practically useless all processes involving the blowing of air through the boiling wort in copper. Although moderate aeration is desirable, over-aeration is probably objectionable, as it appears to result in a loss of hop flavour (Pasteur), and possibly also in loss of antiseptic power of the hop-resins. Moreover, the wort is unnecessarily exposed to the risk of infection.

Cold aeration occurs chiefly on the refrigerators. At this stage the wort becomes almost saturated with dissolved oxygen, the presence of which is necessary, as Pasteur has shown, in order that a vigorous fermentation may be obtained. The oxygen thus dissolved disappears during the early stages of fermentation (8–10 hours after pitching), and is undoubtedly utilised by the yeast.

In the discussion which ensued after the reading of the paper, no points of importance were raised.—H. T. P.

*Hops.* L. Briant and C. S. Meacham. Trans. Inst. Brew. 7, 117–140.

EXPERIMENTS conducted by Hayduck's method show that the deterioration which takes place in hops on keeping is due to the transformation of soft oleo-resin into hard resin, but this change would seem not to be the result of an oxidising action of the atmospheric oxygen.

The amount of resin contained in the same kind of hops at different stages of development and ripening increases with the age of the hop until the hop has attained a degree of brown ripeness, which would militate very greatly against its market value on the ground of appearance. The authors have confirmed their former opinion (this Journal, 1893, 703) that foreign hops have a greater preservative action than English hops, and show that this is due, not to foreign hops yielding a greater proportion of acid to beer than English ones, nor to their containing any materially greater amount of tannin, but to their being richer in total resins and containing a larger proportion of oleo-resin than English hops.

The authors are of opinion that the amount of albuminoids precipitated by the hop tannin during the boiling of wort is very small and has been much over-estimated.

—J. G. W.

*Studies on Secondary Fermentation and "Frets."* H. Van Laer. Trans. Inst. Brew. 7, 55—113.

By cultivating the deposits of English and Belgian beers, which were undergoing secondary fermentation, in wort-gelatin or gelatin and beer, taking, however, the necessary precaution of continuing the cultivation for a sufficient length of time, the author has isolated, in addition to *Saccharomyces cerevisiae*, *Mycoderma cerevisiae* and torulas, ten growths presenting marked differences in their physiological characters which he has made the objects of investigation. The same yeasts were also found in the yeast used for pitching these beers, but to isolate them directly from such a yeast it was first necessary to induce their appearance by fermenting products of starch conversion, previously fermented and freed from alcohol, with this yeast.

A deposit is obtained in this way, after several weeks' forcing, consisting of the secondary forms pre-existent, but microscopically indistinguishable in the pitching yeast. Secondary forms were also obtained direct from ordinary yeast by placing it for eight days on sterilised filter-paper under a desiccator and afterwards cultivating in a very attenuated beer which had been previously fermented and then pasteurised, after the addition of a little maltose. By this method of culture the *S. cerevisiae* forms existent in the yeast reproduce themselves less rapidly than do the secondary forms.

The species of secondary forms isolated in this way were distinguished by (1) their macroscopical aspect, which varied from heavy and clotty to light and flocculent; (2) the limit-attenuation to which they gave rise in boiled worts, which showed considerable variation; and (3) their fermentative energy or the relative rate of destruction of carbohydrates which they produced in the same wort under identical conditions.

With regard to their microscopical aspect, the *S. cerevisiae* forms retained their original round form during all the phases of fermentation, while the *S. ellipsoideus* and *S. Pastorianus* forms showed considerable polymorphism dependent on the stage of fermentation and the medium in which it was conducted.

Experiments were conducted on the fermentation of wort by means of isolated species, both singly and in mixture, and the author arrives at the conclusion that when dry hops are added to a beer at racking (1) the after-fermentation will show itself after a more or less extended period, irrespective altogether of the nature of the yeast used for pitching; (2) if the primary yeast is mixed with even minute quantities of secondary yeasts, the after-fermentation always shows itself and with greater intensity than when the beer has only been pitched with the primary yeast; and (3) that the intensity of the fermentation depends upon the nature of the yeasts mixed with the principal yeast. The author explains the turbidity produced in beer by "frets" as being due to the coefficient of reproduction, i.e. the ratio of the number of cells produced to the matter fermented, of yeasts of the *ellipsoideus* and *Pastorianus* types being greater than that of *S. cerevisiae*.

Fermentations conducted without the addition of dry hops at racking have established the following conclusions:—

(1.) The exclusive use of certain types of yeast may give rise to an after-fermentation, which must be regarded as a continuation of the primary one. This after-fermentation is, however, too vigorous and is accompanied by a too great reproduction of yeast to be applicable commercially.

(2.) Every system of yeast formed by one or more races of a similar type and of very small quantities of a yeast having a lower limit of attenuation may give rise to after-fermentation, in which instance the attenuation obtained at racking is practically that which would have been obtained by fermenting with the preponderating yeast only, whilst the limit-attenuation will be that of the lowest type of the system of yeasts.

(3.) An after-fermentation can also be obtained by introducing into a yeast or mixture of yeasts, the pace of fermentation of which is rapid, a large quantity of a slower yeast, by which means the attenuation at racking will be prevented from attaining the limit corresponding to the rapid yeast. The after-fermentation is then produced by virtue of the difference existing between the attenuation at

racking and that corresponding to the limit-attenuation of the system of yeasts used.

Beers fermented with certain mixtures of *Saccharomyces* possess a greater immunity as regards wild yeasts and bacteria than do beers which are the result of single-race fermentation.

*Pure Cultivation Yeast and the Use of Antiseptics, especially of Fluorine Compounds, in the Distilling.* A. Claus. Zeits. für Spiritus-ind. 16, Ergänzungsheft, 91.

AFTER a careful discussion of the results of himself and others, the author draws the following conclusions:—(1.) The introduction of pure yeast in the manufacture of spirit is a decided advance, as antiseptics are not able to insure a pure culture nor the destruction of disease ferments. (2.) True pure culture is only attainable by Hansen's method of starting from a single cell. (3.) Effront's proposal to purify the yeast by hydrofluoric acid is not certain to effect its purpose of enabling the culture yeast to destroy the disease ferments. (4.) The advantages of the Hansen method, which have been proved in brewing, are equally valid in spirit manufacture. (5.) The introduction of the pure culture system does not exclude the use of antiseptics, which are still necessary in certain directions. (6.) With the employment of pure cultures, the use of disinfectants in cleansing of vessels, &c. is even more necessary than hitherto. The author considers fluorine compounds the best disinfectants for these purposes. (7.) The use of disinfectants with the yeast, mash, &c. is still necessary, as in spirit manufacture the mash is more susceptible to bacterial infection than is the case with the hopped mash of the brewery. Diastase especially needs the protection of antiseptics. (8.) The mode of working will be modified where pure culture is introduced, as less impurities are introduced into the mash and the latter becomes more resistant to outside influences. (9.) Hydrofluoric acid is of great value in helping the pure yeast to resist the harmful influence of acidifying ferments, &c., both during fermentation and during the continuous yeast culture. This beneficial influence is not, however, as far-reaching as Effront states it to be. (10.) Hydrofluoric acid exercises a specific physiological action on yeast and if employed with care, may be a useful stimulant for the yeast. It must be remembered, however, that under certain circumstances this acid may also act as a stimulant to the disease ferments; and an exact knowledge of the culture and disease yeasts, and of the other organisms met with in spirit manufacture, is necessary before a really rational and complete use of the hydrofluoric method is possible.

—L. T. T.

## PATENTS.

*An Improvement in the Manufacture of Beer.* J. Pikhart. Mahrisch-Schoenberg, Moravia. Eng. Pat. 10,132, May 20, 1893.

THIS invention relates to the application of a preparation of the fruit of the carob tree (*ceratonia siliqua*) in brewing, with the object of imparting a pleasant aroma and giving greater body to beer; also to mask the rapid bitter flavour and render the beer better fitted for keeping and more wholesome. The fruits are treated with warm water, and are washed and dried at about 35° C. until they present a brownish colour, and the juice of the fruit when the latter is broken is dark red. The dried fruits while still warm are cut into pieces and may be stored or added directly to the mash in the proportion of about 2.5 kilos. to a hectolitre of ordinary beer.—A. R. L.

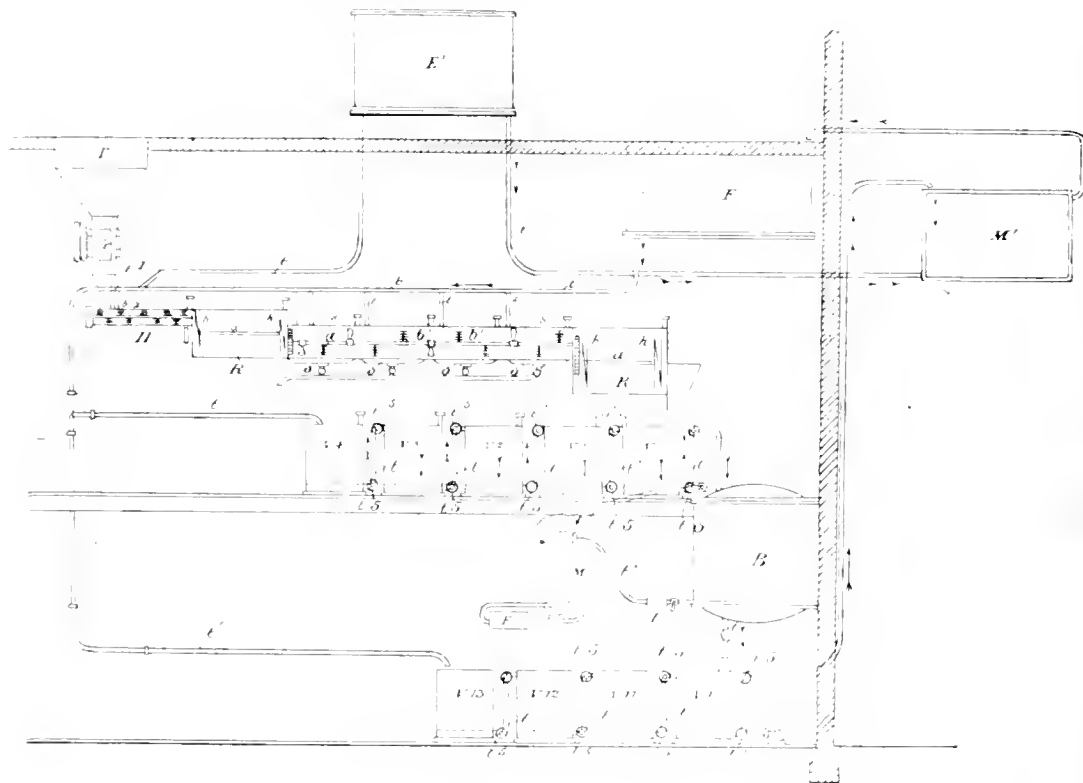
*An Improved Still or Apparatus for Distilling Spirits and all kinds of other Liquids.* G. Morris and W. Edney, both of Bristol. Eng. Pat. 16,063, August 25, 1893.

See under I., page 504.

*Improvements relating to the Saccharification of Wort or the Malt, and to the Extraction and Subsequent Treatment thereof in Brewing, Distilling, and in the Manufacture of Maltose, Syrups, and other Extracts, and Apparatus therefor.* G. de Geyter, Monseron, Belgium. Int. Pat. 6168, March 22, 1894.

THE process claimed in this specification comprises the following clauses:—(1.) The continuous diastatic saccharification of the amylaceous materials obtained by uninterrupted circulation within appropriate vessels (see figure). (2.)

The exhaustion or extraction and continuous washing of the malt and hops. (3.) The coagulation of the nitrogenous materials of the wort by oxidation in a tubular apparatus, followed by the purifying of the wort by separation of the nitrogenous coagulated materials by cold filtration and subsequent reheating during the boiling process, and without interruption. The invention of the apparatus (see figure) for carrying out the above process is also claimed. It consists of three troughs, namely, a "resting trough" R, a "saccharifier" S, and a "resting trough" R' for the



saccharifier. Each of these is provided with "moistening," "working up," and "propelling" gear so that the diastatic action may proceed continuously. There are also washing and exhausting batteries for the returns and hops, V to V<sup>1</sup> and V<sup>2</sup> to V<sup>3</sup>, having false bottoms, in which the water arriving over the most exhausted returns or hops passes successively over the gradually richer returns or hops so as to produce a complete exhaustion. Lastly, the combination of an aerator tube t<sup>(1)</sup>, serving to bring about the coagulation of the nitrogenous matters of the wort, with a heat interchanger M and a filter F for separating the coagulated nitrogenous matter during the boiling of the wort.—A. R. L.

Skimmed milk, exposed in a eudiometer to the action of oxygen ozonised by the passage of an electric spark, became acid and coagulated. He now finds that other oxidising agents, such as hydrogen peroxide, bromine water, potassium permanganate and bichromate, have a like effect. The effect of rennet is not, on this account, necessarily to be considered one of oxidation, being more probably due to precipitation of the casein by lactic acid arising from milk-sugar acted on by the rennet. He suggests the use of hydrogen peroxide in place of rennet for cheese-making.

—B. B.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY AND WATER PURIFICATION, DISINFECTANTS.

### (A).—CHEMISTRY OF FOODS.

*The Curdling of Milk.* M. W. Hes. School of Mines Quarterly, 15 [2], 1894, 105—108.

In 1877 the author published the following observation in the Boston Journal of Chemistry, 12 [6]:—

*The Carbohydrates of the Fruit of the Kentucky Coffee-Nut Tree (Gymnocladus Canadensis).* W. E. Stone and W. H. Test. Amer. Chem. J. 15, 660—663.

THE Kentucky coffee-nut tree occurs in fertile soils in the west and south-west of the United States, and is closely related both to the common thorny or honey locust (*Gleditsia*) and to *Ceratonia siliqua*. The fruit consists of a leathery pod 3 to 6 ins. in length, and containing from two to six brown, oval and very hard seeds, which are embedded in a greenish waxy gum. The dry matter of the gum contains about 15 per cent. of saccharose, and almost as much glucose; it is free both from galactose and from starch. The insoluble gum of the fruit which remains after extracting the fresh gum with alcohol is partially hydrolysed when treated with dilute sulphuric acid, the products of hydrolysis being dextrose and a pentose, probably arabinose.

It is questionable whether these are derived from separate mother-substances in the gum or from an individual substance.—C. A. K.

*Manufacture of Oil and Food from Pea Nuts.* United States Consular Reports, 1894, **44**, 683.

See under XII., page 530.

*Natural Oxycelluloses. I. Celluloses of the Gramineæ.* C. Smith. Proc. Chem. Soc. 1894 [137], 89.

THE "celluloses" of esparto and the cereal straws, as isolated by the well-known processes of the paper-maker, were further purified, and (1) their ultimate composition, (2) the amount of furfural obtained on boiling with aqueous hydrochloric acid (1.06 rel. dens.) were determined.

These "celluloses," were found to be oxycelluloses, and, since they do not give the reactions characteristic of the pentoses, they are of the type of those obtained by Cross and Bevan by regulated oxidation of the normal celluloses (Ber. 1893).

Such oxycelluloses being widely distributed in nature, it becomes important to study their physiology. A course of systematic observations has therefore been undertaken on the germination and growth of the barley plant in relation to the composition and constitution of its permanent tissue ("cellulose").

It has already been observed that, by germination in the dark and growth of the sprouts (etiolated) until the endosperm is nearly exhausted, there is a considerable increase in the furfural-yielding constituents (60–80 per cent.), the early material of the tissue giving 5 per cent. furfural, and at the same time no pentose reaction, which proves it to be an oxycellulose. The results are still more marked in the case of the tissues of plants grown in the light. In the discussion following, Mr. Warington hoped that the authors would continue this investigation, as the subject was of much importance in relation to the digestibility of animal food. They had already established the important fact that the production of furfural can no longer be utilised for the estimation of the pentoses, since the oxycelluloses also furnish this compound.

*The Use of Lead Pipes for Conveying Water.* M. T. Lecco. Chem. Zeit. **17**, 1431.

See under XXIII., page 547.

## PATENTS.

*A New or Improved Method of Improving the Quality of Damaged or other Coffee.* A. Brongier, Munich, Germany. Eng. Pat. 8270, April 24, 1893.

THE inventor's claim is one of improvement of the coffee by roasting the beans with an extract containing caffeine, which extract is obtained by boiling new unroasted shells or the pulp of the coffee berries, or coffee leaves, or leaves of the cocon, kola, mathé, or tea. The extract is only added at the moment when the outer layers of the beans begin to give off volatile oil and develop a bluish smoke. Sea-damaged or other coffee of inferior quality is soaked in lime-water previous to the roasting process. Any excess of lime is removed by washing.

Whatever roasting apparatus is used, it must be constructed so as to allow of a periodical insertion of a tube into its roasting drum, for instance, through the hollow axis, to that point where the beans begin to swell and get mellow. The extract containing the caffeine is then sprayed over the beans by means of a distributing apparatus.—L. de K.

*Apparatus for the Artificial Production of Chalybeate Water.* O. March, London. Eng. Pat. 9362, May 10, 1893.

See under XI., page 529.

*Process for Preserving Milk and Cream for a Length of Time, rendering it suitable for Lengthy Transit.* W. F. E. Case, Copenhagen, Denmark. Eng. Pat. 15,161, August 8, 1893.

MILK, which should be quite fresh, is put into suitable containers and mixed with pieces of already frozen milk from the same or a previous milking. The vessel is then generally closed, but in a pure atmosphere it may be left open. The pieces of frozen milk remain floating on the surface, keeping the milk for a time at 32° F. During the gradual melting the liquid is thus kept in a state of continual motion, which prevents the rising of the cream. If the container is large enough to hold 80 or 100 gallons of milk and a sufficiency of frozen milk, and if it be packed in a layer of sawdust 1 or 5 ins. thick, the milk may be, as a rule, kept quite fresh for two or three weeks.—L. de K.

*Improvements in the Treatment of Pepper.* M. Kirchberger, London. Eng. Pat. 16,941, September 8, 1893.

THE inventor claims to have made an improvement in the bleaching of pepper, chiefly by using alum.

After any dust has been separated from the pepper by winnowing, it is placed in water and any grains that float are rejected. The grains which sink are removed and watered at intervals until the outer coverings are rotten. The pepper is then placed in an iron pan and sprinkled with alum or a solution of the same, and trodden under foot until the husks and the black oily matter have been removed. After thoroughly washing with water, the grains are placed in a solution of chloride of lime mixed with alum or a little sulphuric acid. To remove excess of chloride of lime, the pepper is once more rinsed in alum water. In the treatment according to this invention of commercial white pepper coloured with lime, the latter must be first removed by treatment with dilute acetic or some other weak acid.

—L. de K.

*A New or Improved Meat Extract.* Communicated by J. H. Niemann, Warracknabeal, Australia; R. F. Macloskey, Manchester. Eng. Pat. 1170, January 19, 1894.

THE inventor claims to have prepared a superior meat extract by expressing the juices of raw lean meat and evaporating them to a thick consistency or to dryness, after first adding about one eighth part of culinary salt. Before packing the extract for sale and use, it is mixed with about an equal bulk of Liebig's extract of meat or any other similar preparation; also with pepper sauce if desired.

The beef, or other suitable meat, should be freed from fat as far as practicable before being pressed out.—L. de K.

## (B.)—SANITARY CHEMISTRY AND WATER PURIFICATION.

*The Use of Lead Pipes for Conveying Water.* M. T. Lecco. Chem. Zeit. **17**, 1431.

See under XXIII., page 547.

*Experiments on the Disinfection of Town Sewage with Sulphuric Acid.* M. Ivanoff. Proc. Inst. Civil Eng. **115** [1], 42–43; and Zeits. für Hygiene, 1893, 86.

THE fact that the cholera bacillus displays an intense susceptibility to the action of acids was known to its discoverer, for Dr. Koch pointed out that in the acid secretions of the stomach these bacilli speedily lose their vitality. The



subsequent experiments of Kitasato have shown that very minute additions of sulphuric and hydrochloric acids to bouillon cultures destroy cholera germs in the course of a few hours; and, lastly, Messrs. Stutzer and Burri have studied exhaustively the effect upon these organisms of very dilute solutions of sulphuric acid. At the suggestion of Professor Pfuhl, the author undertook to investigate the action of dilute sulphuric acid upon cholera bacteria when present in sewage water. From the inception of his experiments he surmised that under these conditions the acid must be used in a more concentrated form than in above cases, because the sewage water invariably contains substances which would combine with the acid, and would thus, to some extent, neutralise its effects. It was, however, ascertained that the additional amount of acid rendered necessary on this account was but trifling. The samples of sewage water were derived both from the Berlin and the Potsdam sewers, and were infected alternately with pure cultures of the cholera bacillus and with the fresh dejections of a cholera patient. The acid in the case of the Berlin sewage water was used in three degrees of strength:—(1) as a 0.02 per cent. solution; (2) as a 0.04 per cent. solution; and (3) as a 0.1 per cent. solution. The conditions under which the analyses were made are fully described, and the author availed himself of microscopic observations, as well as bacteriological tests. Four parallel series of experiments were carried out, and the results were in every case identical. The Potsdam sewage, which is three times as concentrated as that of Berlin, was treated with stronger acid, viz., with 0.04, 0.06, 0.08, and 0.12 per cent. solutions. It was found that in the case of the Berlin sewage, the 0.04 per cent. solution of acid was fatal to the cholera bacilli, but that with the stronger sewage water of Potsdam, the amount of acid needed was that present in the 0.08 per cent. solution. It is pointed out that whereas, previous to treatment, the sewage water was faintly alkaline, the sample to which 0.08 per cent. of acid had been added had a strongly acid reaction to litmus paper, and that such reaction may be regarded as an indication that the necessary dose of acid has been employed. This treatment is, with the exception of the use of lime, the cheapest system that can be adopted.

*Experiments concerning the Action of Peat Dust upon the Bacteria of Cholera and Typhoid Fever.* C. Fränkel and E. Klipstein. *Zeits. für Hygiene*, 1893, 333; and *Proc. Inst. Civil Eng.* 115 [1], 44–45.

From certain researches by Schröder, it has become evident that, in addition to its properties as a disinfectant, peat possesses the power of destroying the cholera vibrios, though certain other species of bacilli prove themselves more capable of resisting its influence. The authors have conducted a series of tests with two descriptions of peat, one of which had a strongly acid reaction, the other was more feebly acid. Samples of each kind of peat, both sterilised and non-sterilised, were added to cultures of cholera, typhoid fever, and other germs, and the results are set forth in tables. It is deduced that in from 2½ to 5 hours the comma bacillus is destroyed by the addition of peat dust; but exposure to this substance for only one half or one hour will sensibly diminish its vitality. The cholera vibrios may retain their vitality for as long as 14 days in a mixture of peat dust with urine; but, as a rule, they are destroyed at the end of a week, or, in very acid urine, even after only one day. Mixtures of peat with various substances valuable in agriculture (kainit and superphosphates) were also tested by the authors; kainit was found to be wholly devoid of influence upon the cholera bacteria, while both kinds of superphosphate examined proved themselves to be possessed of marked effect in augmenting the germicide powers of peat. Investigations specially undertaken to determine upon what properties in the peat this action depended, demonstrated that its influence was due to the acids therein contained, and led to the conclusion that, in order to increase the efficiency of the peat to the utmost, care must be taken to intensify, as far as possible, this acid reaction by artificial means. Under all circumstances, where a dry system is unavoidable, as, for instance,

in the case of isolated buildings, hospitals, barracks, &c., the use of dried peat may be confidently recommended as furnishing a safe, cheap, and reliable system of dealing with excreta. (See this Journal, 1893, 994.)

## PATENTS.

*A New and Improved Method of Treating Sewage by Means of Ferric Compounds.* R. C. Tanner, Chester. Eng. Pat. 9063, May 6, 1893.

SEWAGE, when not already alkaline, is treated with milk of lime, chloride of lime (calcium hypochlorite), and with solution of ferrous sulphate crystals. But if the sewage is already alkaline, the lime may be omitted.—E. S.

*Improvements in Apparatus for Softening and Purifying Water.* A. Smith, Camberwell. Eng. Pat. 14,195, July 22, 1893.

THE water to be softened is caused to flow through the channels of a mixing cistern, where it meets with the reagent, such as lime or the like, and becomes thoroughly mixed before passing out into the softening cistern, formed with a central entrance funnel, which discharges the water into the cistern where the softening process is completed; the water finally escapes by rising and flowing over the edge of a discharge gutter placed round the inside of the cistern. The process of softening in the mixing cistern may be accelerated by heating the water as it flows through the mixing channels.—E. G. C.

## (C).—DISINFECTANTS.

### PATENT.

*A Superfatted Biniodide of Mercury Soap.* C. R. Illingworth, West Kensington, London. Eng. Pat. 12,884, July 1, 1893.

See under XII., page 531.

## XIX.—PAPER, PASTEBOARD, Etc.

*Discoloration of Papers made from Sulphite "Cellulose."* E. Muth. *Dingler's polyt. J.* 291, 235.

THE author has made careful observations upon the discoloration of papers made from bleached sulphite pulp (with addition of the usual rosin size), and after eliminating all other possible causes, concludes that the discoloration is the result of chemical changes in the "cellulose" itself. Having concluded from superficial observations on the liquors obtained as by-products of the pulp manufacture, that these contain "tannins," the author made experiments upon solutions of pyrogallol acid and other phenolic compounds applied to papers, which he finds produce a rapid discoloration. He concludes that such compounds are present in the bleached pulp and are the immediate cause of the discoloration of the papers in question.—C. F. C.

*Spots in Paper.* *Mitth. Königl. tech. Versuchs.* 11 [5], 260.

A YELLOWISH paper which had been attached by means of starch paste to ordinary cardboard made from mechanical wood pulp showed a number of sharply defined and unsightly spots, and a sample, together with some of the pieces of the paper, was submitted to the Institute. After an experiment had been made showing that the starch paste was not responsible for the spots, a piece of the paper was treated with an acid solution of potassium ferrocyanide, when a number of blue spots made their appearance over

the whole surface. When the paper and cardboard were submitted to the same test, the paper side showed similar spots, whilst the cardboard remained unaltered. A slight difference, however, was noticeable in the spots, those on the original paper being sharply defined, whilst those on the paper attached to the cardboard were in part surrounded by a pale blue halo.

The paper was then separated from the cardboard by soaking in water, and the two faces which had been stuck together were treated with the ferrocyanide solution. Both the paper and the cardboard showed spots, but the cardboard only in the places where it had been in contact with the spots of the paper, whilst the back of the cardboard showed no iron reaction. It was therefore fair to conclude that the spots were due to the presence of iron in the water used in the manufacture of the paper. These spots coming into contact with the starch, a small quantity of the iron passes into solution, and as a result of subsequent pressure, the spots appear surrounded by a halo. At the same time the softening of the spots under the influence of the starch paste explains their partial transference to the inner side of the cardboard.—E. G. P. T.

*Natural Oxycelluloses. I. Celluloses of the Gramineæ.* C. Smith. Proc. Chem. Soc. 1894 [137], 89.

See under XVIII. A., page 537.

#### PATENTS.

*An Improved Paper or Textile Fabric and Methods of and Apparatus for producing said Paper or Fabric.* S. Wheeler, Albany, U.S.A. Eng. Pat. 9827, May 16, 1893.

THE object of the invention is to produce a paper or other fibrous material permanently wrinkled, and having on its surface distinct figures or ornamentations, such as diamonds or stars. Such wrinkled material is very advantageous in the production of ordinary "sanitary" paper, as it presents a corrugated or uneven instead of the usual surface. The improved product may be obtained either by operating upon material already manufactured or upon material in the process of manufacture. In the first instance the fabric is damped at suitably situated spots by touching it with a damping device. It is then dried, preferably by artificial heat. The damped spots, during the drying, cause the adjacent portions to form into permanent wrinkles, which, unlike the indents formed by folding, crimping, &c., remain absolutely permanent.

In the second instance, the wet paper, after it leaves the forming machinery, is dried in varying spots or lines and at different points, by passing the wet web over a series of heated surfaces so formed as to dry the web at certain points only, leaving the intervening spaces to be dried afterwards. Suitable machinery for producing these results is described and claimed.—E. G. P. T.

*Improvements in the Manufacture of Cellulose.* C. Kellner, Vienna, Austria. Eng. Pat. 24,287, December 16, 1893.

THE process consists in treating wood or other plant substances with hydrolysing or oxidising agents, such as nitric oxide, nitrous, hyponitrous, hydrochloric, and chlorous acids in the gaseous state. The plant substances, previously damped, having been exposed for some time to the action of the acid vapours in a suitably-constructed vessel, are treated with hot or cold water, then subjected to a mechanical treatment in a breaking engine, and subsequently treated with an alkaline solution in order to dissolve the by-products. Finally, they are subjected to violent agitation in a beater, so as to cause the cells separated by the previous treatment to become thoroughly and uniformly intermingled.—E. G. P. T.

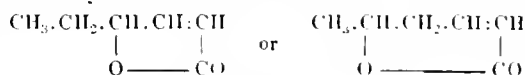
*Improvements in the Manufacture of Paper for Bank Notes, Securities, and the like.* C. Battner, Eppendorf, and F. Will, Königsfeld, Germany. Eng. Pat. 3453, February 17, 1894.

FOR the purpose of this invention a fine, transparent, open-mesh fabric made of slender but strong threads is passed into paper pulp, made from the best raw materials, while such pulp is moving over the wire in its passage from the stuff-vat to the couch rolls and before it reaches the latter. The fabric may be plain or with designs in it. In this state the fabric, lying flat upon the wire, is encompassed on all sides with pulp and passes through the couch rolls, whereupon an intimate combination takes place between and through the meshes of the fabric. It is claimed for the paper thus produced that it offers an absolute bar to imitation and falsification, and that it possesses a high degree of tenacity and durability.—E. G. P. T.

#### XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*The Volatile Oil of Rowan-berries, Parasorbic Acid, and its Isomerism with Sorbic Acid.* O. Doehner. Ber. 27, 344—351.

THE fruit of the rowan tree (*Sorbus aucuparia*), or mountain ash, contains, besides malic acid, a volatile weak acid boiling at 221°, and converted into an i-omeric strongly marked acid by treatment with caustic alkalis or concentrated sulphuric acid (A. W. Hofmann). The former has been usually known as *parasorbic acid*, and the latter as *sorbic acid*. The author finds as the result of his experiments that parasorbic acid is not a true acid at all, but a lactone derived from either  $\gamma$ - or  $\delta$ -oxyhydro sorbic acid, and indicated by one of the two formulæ—



whilst sorbic acid is represented by the formula—



Parasorbic acid takes up only two atoms of bromine, whilst sorbic acid unites with four; the latter neutralises the appropriate quantity of alkalis on titration, whilst the former only neutralises about a tenth part of that amount. Parasorbic acid when dissolved in alcohol and treated with hydrochloric acid gas, furnishes the ether of sorbic acid, boiling at 195°; this affords a better way of producing sorbic acid (by saponification of the ether) than the action of alkalis. One gram. of sorbic acid administered to a small dog produced no notable effect, whilst the same quantity of parasorbic acid produced emesis and salivation, but no other marked poisonous action. The quantity of parasorbic acid contained in mountain ash berries is about the same whether they are half-ripe or wholly so, but the green berries do not yield it at all; these, on the other hand, contain malic acid, which disappears as ripening progresses, yielding sorbite,  $\text{C}_6\text{H}_{14}\text{O}_6$ , and sorbinose,  $\text{C}_6\text{H}_{12}\text{O}_6$ , as alteration products.—C. R. A. W.

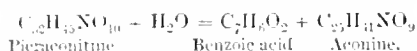
*Aconitine.* M. Freund and P. Beck. Ber. 27, 433—436.

THE authors have examined the crystallised alkaloid manufactured by E. Merck, of Darmstadt, with results differing somewhat from those of other observers who have previously operated with *A. napellus* roots. They regard it as identical with the alkaloid examined by Alder Wright, and subsequently by Dunstan, but obtained numbers leading to the formula,  $\text{C}_{34}\text{H}_{45}\text{NO}_{11}$ , or more probably,  $\text{C}_{34}\text{H}_{47}\text{NO}_{11}$ , instead of  $\text{C}_{33}\text{H}_{43}\text{NO}_{12}$ . As stated by Ehrenberg and Purfürst it hydrolyses into acetic acid and a new base; this yields crystallisable salts; the hydrobromide melts at 282°, and the hydrochloride at 217° or 270°, according

as it is crystallised from water or from hydrochloric acid. The authors regard this base as indicated by the formula  $C_{22}H_{41}NO_{11}$ ; and as identical on the one hand with the "pieraconitine" isolated by Groves and found by Wright to be indicated by the formula  $C_{31}H_{45}NO_{10}$ ; and on the other hand, with the "isaeconitine" of Dunstan, regarded by him as  $C_{33}H_{47}NO_{12}$ ; accordingly they designate it *pieraconitine*, the relationship of pieraconitine to aconitine being represented by the equation—



i.e., aconitine being acetylpieraconitine. By further hydrolysis benzoic acid is formed, and a new base, apparently identical with the "aconine" of Alder Wright, this being developed from pieraconitine in virtue of the reaction—



Alder Wright, however, found the formula,  $C_{26}H_{39}NO_{11}$ , whilst Dunstan found  $C_{27}H_{41}NO_{11}$ . Hence aconitine is acetyl-benzoyl aconine,  $C_{25}H_{39}NO_9(C_7H_5O)(C_7H_5O)$ .

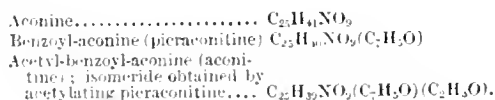
—C. R. A. W.

*Aconitine.* W. R. Dunstan. Ber. 27, 664.

THE author and his collaborateurs have previously shown that aconitine furnishes acetic acid on heating, and that consequently it is to be regarded as acetyl-benzoyl-aconine, as recently shown by Freund and Beck.—C. R. A. W.

*Aconitine.* M. Freund and P. Beck. Ber. 27, 720—733.

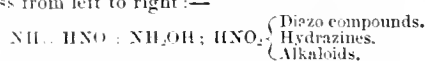
IN continuation of their experiments on crystallised aconitine prepared from *A. napellus* roots by E. Merck, of Darmstadt (this Journal, *supra.*), the authors have found that the alkaloid examined by them gives the same crystallographic measurements as were found by Dunstan; but instead of yielding numbers on analysis agreeing with his formula  $C_{33}H_{45}NO_{12}$ , they obtained values leading to the formula  $C_{34}H_{47}NO_{12}$ ; these values are close to those obtained by Alder Wright for "apo-aconitine" (derived from the alkaloid  $C_{25}H_{43}NO_{12}$ , examined by him, by removal of the elements of water) and for "japaconitine" (from Japanese aconite roots). On the other hand, Jürgens deduced from his analyses the formula  $C_{33}H_{45}NO_{12}$ , and Ehrenberg and Purfürst the formula  $C_{27}H_{43}NO_{11}$ . These latter found that four methoxyl groups were present, as methyl iodide to the corresponding extent was formed on treatment with hydriodic acid. The gold salt from Merck's aconitine melted at  $134-135^\circ$  when prepared by treatment in contact with alcohol, but at  $152^\circ$  after heating so as to drive off alcohol and render anhydrous, in these respects corresponding with different modifications of aconitine aurochloride described by Dunstan. The hydrobromide dried at  $100^\circ$  melted at  $163^\circ$ . On boiling with water aconitine hydrolyses in two ways, partly forming acetic acid and pieraconitine,  $C_{22}H_{41}NO_{11}$ , and partly forming acetic and benzoic acids and aconine,  $C_{15}H_{35}NO_9$ . Pieraconitine, when heated with alcoholic potash, similarly forms benzoic acid and aconine; and on treatment with acetic anhydride furnishes an acetyl derivative, apparently only isomeric and not identical with aconitine, since it melts at  $255-256^\circ$ . The relationships of the different alkaloids examined are expressed by the formulae:—



—C. R. A. W.

*Recent Developments in Pharmaceutical Chemistry with Special Reference to Medicaments obtained by Chemical Synthesis.* Hugo Erdmann. Zeits. ang. Chem. 1893 [13], 379—382.

THE suitability of a chemical substance for pharmaceutical purposes depends upon a variety of circumstances. It should be capable of exerting physiological action, which should be limited definitely to certain parts or functions of the entire organism. It should, as a medicament, be capable of healing unsound organs with the least possible collateral effect upon those which are sound. It should act promptly and energetically and yet not endanger life. The condition is also important, that after long use its efficacy remain unimpaired, and that no toxic phenomena develop themselves. The question naturally follows: "On what does the poisonous quality of a chemical substance depend?" for it is found that the bodies suited as medicaments as a rule occupy a transition position, or one midway between the actually poisonous and the inert elements and compounds. Of the different compounds of one and the same element, the most unstable are usually the most poisonous, as may be observed in the following series of nitrogen compounds in which poisonous quality regularly increases as we pass from left to right:—



Nevertheless both valency and mode of linking of the elements play an important part:—

Harmless.....  $CO_2$   $H_3PO_4$  .....  $KCl$  .....  $MnCl_2$   
Poisonous.....  $CO$   $H_3PO_3$   $As_2O_3$   $KClO_3$   $CrCl_3$   $KMnO_4$   
Very poisonous.  $C_2H_2$   $P$   $AsH_3$  .....  $K_2CrO_4$  .....

Those compounds are especially suspicious, which exert oxidising or reducing action, and thus disturb the functions of the organism. Hence all elements act as strong poisons which furnish several series of stable compounds, easily passing one into the other. Thus arsenic, mercury, thallium, tin, and copper. In one group of similar elements the toxicity steadily increases with increasing atomic weight (see Type I.), in another, however, it decreases steadily (see Type II.).

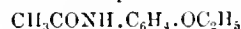
Type I. . . . .	Group VIa.	O	S	Se	T	
Type II. . . . .	Group VIb.	Cr	M	W	U	
Type III. . . . .	Group V.	N	P	As	Sb	Bi

In the second type, the maximum of toxicity in this series, is attained only in certain nitrogen compounds (alkaloids, &c.), whilst e.g. in the case of the hydrogen compounds  $AsH_3$ ,  $PH_3$ ,  $NH_3$  the toxicity from arsenic onwards, again diminishes. Thus we seem to encounter a kind of contrary motions of certain laws, and this fact would appear to be accounted for in the expression of the following law: *that the toxicity of a chemical element is approximately inversely proportional to its frequency of occurrence in the earth's crust.*

The frequently occurring elements are all more or less suited for medical uses, but the strongest action can only be conferred upon them by union with certain of those elements of rarer occurrence, among which only few are to be found which are non-poisonous.

A table is given exhibiting the periodic functions of the elements and their relative physiological activities.

Ethyl, in opposition to methyl and the higher homologues, exerts a favourable nerve-quieting action. This, it is suggested, may be merely attributable to the fact of the habit acquired by mankind of partaking of alcoholic beverages. It is only possible to register it as a singular fact, that whilst the entrance of  $NH_3$  or  $OH$  converts aromatic compounds into poisonous substances, the simultaneous presence of these two salt-forming groups in the para-position to each other, means the mutual paralysis of their physiological properties, that a relatively harmless compound (para-amidophenol) is obtained, in which, moreover, the aggressive properties of both salt-forming groups by further union of the amido group with an acid residue and of the hydroxyl with alkyl (especially with the active ethyl group) may be further diminished, as, for example, in the formation of phenacetin



Strongly acid groups deprive organic compounds of almost all physiological effect; thus, the acid dyestuffs are, almost without exception, non-poisonous, and the sulphonated medicaments become for the most part inactive.

The presence of a carboxyl group sensibly diminishes physiological properties, but is not able to remove them in the case of stronger poisons.

## Poisonous Mother-substance.

Phenol,  
Pyrogallol,  
Guaiacol.

## Non-poisonous Carboxylated Substances.

Salicylic acid,  
Gallic acid,  
Guaiacol-carbolic acid.

TABLE OF THE MOST IMPORTANT MEDICAMENTS.

Trade Name.	By whom introduced.	Scientific Name.	Formula.	Active Elements or Groups.
<i>A.—Hydrocarbons, Alcohols, Phenol Derivatives.</i>				
Pental.	v. Merz.	Trimethylethylene.	$\begin{array}{c} \text{CH}_3 \\ \text{CH}_3 \end{array} \text{C} = \text{C} \begin{array}{c} \text{CH}_3 \\ \text{H} \end{array}$	Two tertiary carbon atoms.
Anylene hydrate.	v. Merz.	Tertiary amyl alcohol.	$\begin{array}{c} \text{CH}_3 \\ \text{CH}_3 \end{array} \text{C} \begin{array}{c} \text{C}_2\text{H}_5 \\ \text{OH} \end{array}$	A tertiary carbon atom with an ethyl group.
Salol.	Sabli.	Salicylic phenyl ester.	$\begin{array}{c} \text{C}_6\text{H}_5 \\ \text{C}_6\text{H}_5 \end{array} \text{C} \begin{array}{c} \text{CO}_2\text{C}_6\text{H}_5 \\ \text{OH} \end{array}$	Salicylic acid, phenol.

*B.—Substances containing Sulphur.*

Sulphonal.	Kast.	Diethylsulphone dimethyl-methane.	$\begin{array}{c} \text{CH}_3 \\ \text{CH}_3 \end{array} \text{C} \begin{array}{c} \text{SO}_2\text{C}_2\text{H}_5 \\ \text{SO}_2\text{C}_2\text{H}_5 \end{array}$	A tertiary carbon with two ethyls.
Trional.	Baumann and Kast.	Di-ethylsulphone-methyl-ethyl-methane.	$\begin{array}{c} \text{CH}_3 \\ \text{C}_2\text{H}_5 \end{array} \text{C} \begin{array}{c} \text{SO}_2\text{C}_2\text{H}_5 \\ \text{SO}_2\text{C}_2\text{H}_5 \end{array}$	A tertiary carbon with three ethyls.
Saccharine.	Adamo and Mosso, Salkowski, Leyden.	<i>o</i> -sulphobenzoic imide.	$\begin{array}{c} \text{C}_6\text{H}_4 \\ \text{CO} \end{array} \text{SO}_2 \text{NH}$	Imide group? Sulphur?

*C.—Bases.*

Methylene Blue (Medicinal).	Ghillany, Ehrlich, and Lippmann.	Tetramethylthionine.	$\text{C}_{19}\text{H}_{15}\text{N}_3\text{S}^+\text{Cl}^-$	Quinone group, sulphur.
Proctanin coeruleum crystallisatum.	Stillinz.	Crystal Violet.	$\begin{array}{c} (\text{CH}_3)_2\text{NC}_6\text{H}_4 \\ (\text{CH}_3)_2\text{NC}_6\text{H}_4 \end{array} \text{C}_6\text{H}_4\text{N} \begin{array}{c} \text{CH}_3 \\ \text{Cl} \\ \text{CH}_3 \end{array}$	Quinone group.
Ethylproctanin.	Stillinz.	Hexa-ethyl- <i>p</i> -rosaniline Hydrochloride.	$\begin{array}{c} (\text{C}_2\text{H}_5)_2\text{NC}_6\text{H}_4 \\ (\text{C}_2\text{H}_5)_2\text{NC}_6\text{H}_4 \end{array} \text{C}_6\text{H}_4\text{N} \begin{array}{c} \text{C}_2\text{H}_5 \\ \text{Cl} \\ \text{C}_2\text{H}_5 \end{array}$	Quinone group, six ethyls.
Proctanin aureum.	Stillinz.	Auramine.	$\begin{array}{c} (\text{CH}_3)_2\text{NC}_6\text{H}_4 \\ \text{NB}_2 \end{array} \text{C}_6\text{H}_4\text{N} \begin{array}{c} \text{CH}_3 \\ \text{Cl} \\ \text{CH}_3 \end{array}$	Quinone group.
Piperazine.	Scheering.	Diethylene diamine.	$\text{N} \begin{array}{c} \text{CH}_2 - \text{CH}_2 \\ \text{CH}_2 - \text{CH}_2 \end{array} \text{NH}$	—
Diuretin.	Gram and von Schroeder.	Theobromine sodium salicylate.	$\text{C}_7\text{H}_7\text{N}_4\text{O}_2\text{Na} + \text{C}_6\text{H}_5\text{OH} \cdot \text{COONa}$	Theobromine, salicylic acid.

*D.—Iodine Preparations.*

Iodol.	Carmichael and Silber.	Tetra-iodol pyrrol.	$\begin{array}{c} \text{Cl} = \text{Cl} \\   \quad   \\ \text{Cl} = \text{Cl} \end{array} \text{NH}$	Iodine.
Soluble with difficulty.	Ostermayer.	Di-iod- <i>p</i> -phenol potassium sulphate.	$\begin{array}{c} \text{Cl} = \text{CH} \\   \quad   \\ \text{HO.C} \quad \text{C.SO}_2\text{K} \\   \quad   \\ \text{Cl} = \text{CH} \end{array}$	Iodine?
Soluble easily.	"	Di-iod- <i>p</i> -phenol sodium sulphate.	$\begin{array}{c} \text{Cl} = \text{CH} \\   \quad   \\ \text{HO.C} \quad \text{C.SO}_3\text{Na} + 2\text{H}_2\text{O} \\   \quad   \\ \text{Cl} = \text{CH} \end{array}$	Phenol hydroxyl.
Aristol.	Messinger and Vertmann.	Dithymoldi-iodol.	$\begin{array}{c} \text{CH}_3 \\   \\ \text{IO} \\   \\ \text{C}_6\text{H}_5 \end{array} \text{C}_6\text{H}_2 - \text{C}_6\text{H}_2 \begin{array}{c} \text{CH}_3 \\   \\ \text{OI} \\   \\ \text{C}_6\text{H}_5 \end{array}$	Iodine.
Rubidium Iodidum.	Erhmann.	Rubidium iodide.	Rb I.	Iodine, Rubidium.

*E.—Medicaments containing Heavy Metals.*

Hamel.	Kobert.	Reduced blood-colouring matter.	47.5 per cent. carbon, 12 per cent. nitrogen, and about 1 per cent. iron.	Iron.
Hydrazgrum amido-succinum.	v. Mering.	Mercury succinimide.	$\begin{array}{c} \text{CO} \\ \diagup \quad \diagdown \\ \text{C}_2\text{H}_5 \quad \text{N.H}_2.\text{N} \quad \begin{array}{c} \text{CO} \\ \diagdown \quad \diagup \\ \text{C}_2\text{H}_5 \end{array} \end{array}$	Mercury.
Dermatol.	Heinz and Liebrecht.	Basic bismuth gallate.	$\text{H}^+ \text{C}_6\text{H}_4\text{COO, Bi(OH)}_2$	Bismuth.

—W. S.

## PATENTS.

*Manufacture of a New Chemical Substance from Orris-root.* J. C. W. F. Tiemann, Berlin. Eng. Pat. 8735, May 1, 1893.

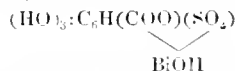
In the alcoholic extract of orris-root the inventor has discovered a new substance, which is the aromatic principle of the root, and to which he gives the name "*irone*." It is a ketone, having the formula  $\text{C}_{15}\text{H}_{10}\text{O}$ . This body has the characteristic odour and flavour of the orris-root, and may be preferentially employed in perfumery, &c. Its preparation is carried out thus:—The alcoholic or ethereal extract of the root is distilled in a current of steam. Organic acids, ethers, alcohols, and irone pass over into the distillate, which is then treated with ether and the ethereal solution agitated with a dilute alkali solution in order to separate the free acids. The mixture is evaporated down and the residue dissolved in alcohol, which solution is mixed at the ordinary temperature with a weak solution of an alkaline hydrate in order to saponify the ethers of the organic acids. After some minutes it is poured into water, the neutral oils are dissolved in ether, the ether is evaporated, and the residue distilled in a current of steam. Irone is one of the bodies distilling over first, and by repeating this operation several times it may be obtained fairly pure, but still containing small quantities of aldehydes, which are eliminated by treatment with weak oxidising agents. The irone is then converted into its phenylhydrazone or condensed with another substituted ammonia to a ketone, from which bodies it is obtained by decomposing with dilute acids and distilling.

Irone boils at 111° C. under a pressure of 16 mm. and has a specific gravity of 0.939.—J. G. W.

*The Manufacture or Production of New Sulpho-Acids and Derivatives thereof.* H. E. Newton, London. From the Farbenfabriken vormals F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 9895, May 17, 1893.

DRIED gallic acid, or its alkaline salts, is gradually stirred into either sulphuric acid mono-hydrate, fuming sulphuric acid, or sulphuric chlorhydrin, taking care that the temperature does not rise too high. The product is cooled, when sulpho-gallic acid crystallises out. The salts of this acid

are obtained by saturating it with the hydroxides or carbonates of the metals or by acting on certain of its salts with metallic salts. The bismuth salt obtained by either of these methods is a yellow, apparently amorphous powder, having the following formula:—



Sulpho-gallic acid and its bismuth salt are more powerful antiseptics than gallic acid and its bismuth salt. Bismuth gallo-sulphonate is also less readily soluble in alkalis than bismuth gallate, and at the same time produces no stimulating effect on wounds, so that it possesses advantages as an antiseptic over that salt.—J. G. W.

*Process for obtaining Para-phenetolcarbamide ("Dulcin").* J. Wetter, London. From J. D. Riedel, Berlin. Eng. Pat. 10,850, June 2, 1893.

THE methods hitherto known for the production of *p*-phenetol-carbamide ("Dulcin") do not yield the theoretical amount owing to secondary reactions. The inventors claim that the theoretical yield is obtained by heating *p*-phenetidine to the boiling-point for several hours with an excess of an aqueous solution of urea nitrate, in a flask provided with an inverted condenser. An alternative process consists in heating *p*-phenetidine hydrochloride and free urea to 120° C. in an open vessel.—J. G. W.

*A New or Improved Anti-pyretic and Anti-neuralgic Preparation.* S. Radlauer, Berlin. Eng. Pat. 3179, Feb. 14, 1894.

WHEN acetanilide and salicylic acid are heated together in molecular proportions to a temperature of 100° C., an oily additive product is formed, which on further heating to 120° C. evolves acetic acid, leaving a body having the formula  $\text{C}_9\text{H}_9\text{N}.\text{CO}.\text{C}_6\text{H}_4.\text{OH}$  which is the subject of the patent. It melts at 100° C., is soluble in alcohol, but not so in water.

—J. G. W.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

## PATENT.

*An Improved Finely Granulated Paper, and Process for Producing the Same.* W. P. Thompson, Liverpool. From C. Schäuffelen, Heilbronn, Germany. Eng. Pat. 23,502, December 6, 1893.

IN patent 14,756, 1891, the inventor described the production of a granulated paper with a uniform grain of pyramidal form in relief. He now claims the production of a pyramidal granulated paper with recessed or depressed grains characterised by the grains, which are placed at a uniform distance apart, being depressed (*in intaglio*) instead of being raised or in relief, but which are produced in an exactly similar manner to the raised grains by one of the two calender rollers between which the paper passes, namely, the steel roller, having pyramidal or truncated cone shaped projections, instead of recesses on its surface. According to the fineness of the grain, there are 750, 1,000, 1,500, 2,500, &c. grains per square centimeter. The granulated paper is eminently suitable for photographic and lithographic purposes, and especially for all processes necessitating the use of a transfer plate.—E. G. P. T.

## XXII.—EXPLOSIVES, MATCHES, Etc.

## PATENT.

*Improved Manufacture of Explosives from Nitrate of Ammonia and Resins.* C. D. Abel, London. From L. König, Berlin. Eng. Pat. 3024, February 12, 1894.

AMMONIUM nitrate and resin are powdered finely, separately or together, are well mixed, and then heated, preferably in a

closed apparatus, until the resin begins to melt. The mixture thus prepared, contains the particles of the salt partially, but not completely coated by resin, so that while on the one hand, no mechanical shaking devices will separate the constituents of the mixture, on the other hand, when ignited, as by a detonator, the combustion extends more rapidly than it would do if the particles of the salt were completely coated with the resin.—E. S.

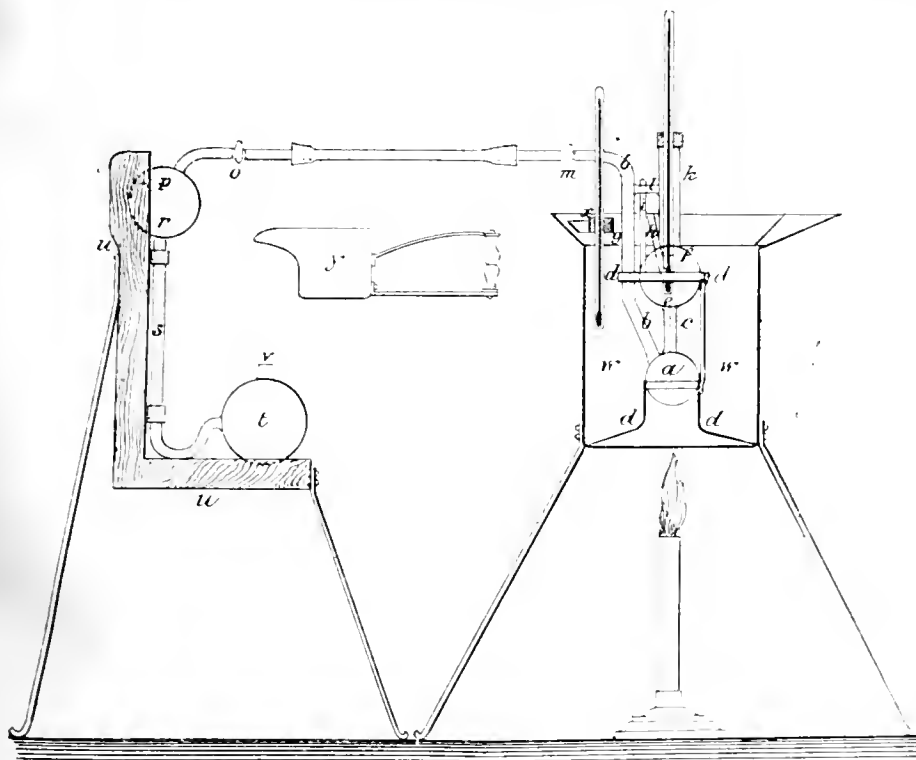
## XXIII.—ANALYTICAL CHEMISTRY.

## APPARATUS, ETC.

*A Viscosimeter for Lubricating Oils.* A. Kinkler. Dingl. Polyt. Journ. 290, 281—283.

IN the examination of lubricating oils, consisting of a mixture of fatty and mineral oils, at least the mineral oil should be tested by means of a viscosimeter. The quantities required for this test being necessarily somewhat large when the existing viscosimeters are used, the author has constructed a new apparatus requiring only 30 cc. The employment of a capillary tube as delivery tube (outflow) is claimed as a further advantage, the oil flowing through it under varying pressures, at different temperatures, whereas, in the case of the old viscosimeters, the determinations being made under the same conditions at different temperatures, inaccuracies arose, in consequence of the times of outflow being necessarily very short. In fact, to arrive at correct results, it would be necessary to employ delivery tubes of different size for higher or lower temperatures.

The new apparatus consists of a sheet brass (oil or water bath *w*, provided with a copper bottom, the contents of which may be heated by a gas lamp, the temperature of the heating liquid being read off by a thermometer held by *a*, *w*



contains the removable stand *d* placed firmly on four legs and supported by two brackets *h*. In this stand fits the viscosimeter, made of strong glass, and consisting of the neck or filling-tube *k*, through which also the thermometer passes, the bulb *c* bearing the mark *f*, the capillary tube *e*, the lower bulb *a*, and the ascending tube *b*; the whole apparatus is held in position by the spring-clamp *i*. The temperature of the contents of the viscosimeter is controlled by thermometer at *k*. The ascending tube *b* is supported by *l*; it is fitted with a tap *m*, and connected by means of india-rubber tubing with the suction apparatus *r*, in which the mercury used for aspirating the oil is allowed to rise up to the mark *p*. Bulb *t* serves as a receptacle for the mercury; can *y* is used for warming the oil to be tested to the desired temperature.

The apparatus must be gauged with a dilute glycerol solution of 1.110 sp. gr. at 20° C., and the time required for its outflow at 20° C. is taken as unity. For temperatures up to 100°, mercury is used as aspirating liquid, for higher temperatures water is preferred. For the heating vessel *w*

water is used for temperatures up to 100°, and above 100° an oil of high boiling point.

The test is carried out in the following manner. Fill *r* up to *p* with the aspirating liquid and heat the bath. In the meantime warm the oil to be tested in can *y* a few degrees above the required temperature. Take the viscosimeter for a short time—say half a minute—out of the bath, so that the air in *a* may be cooled a little; put it back, filling at the same vessel *e* with the oil up to mark *f*. The air in *a* will then expand so that no oil can enter it. Allow the oil in *e* to assume the temperature of the bath, connect the viscosimeter with the aspirator, and open tap *o*. Then open tap *m* and observe accurately the time required by the oil to rise in the ascending tube *b* up to the mark *g*.

For the accurate dimensions of the various parts of the apparatus (which may be had from C. Desaga, of Heidelberg) the original paper must be consulted.

The following table contains a few viscosimetric constants as determined with the new apparatus, contrasted with the numbers obtained by means of Engler's viscosimeter:—

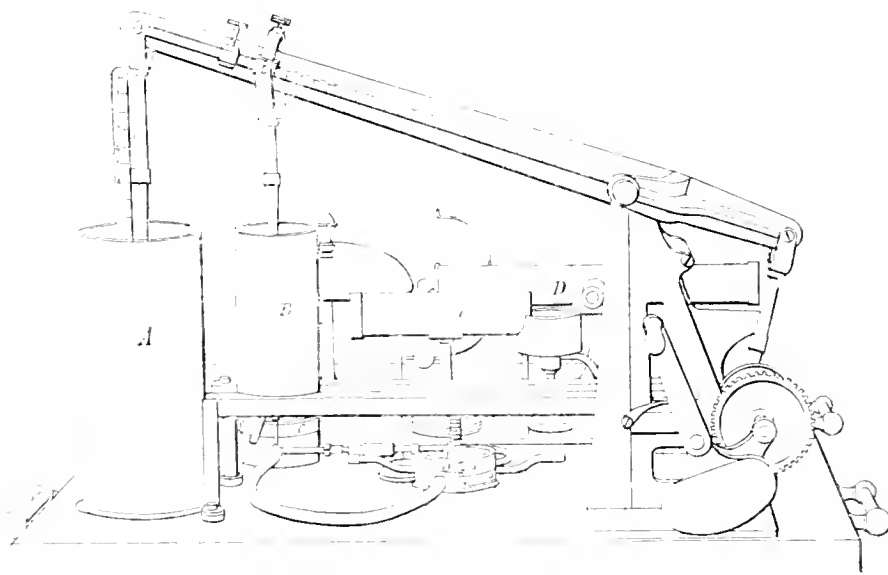
Kind of Oil.	Kunkler's Viscosimeter.						Engler's Viscosimeter.					
	Seconds at			Glycerol Solution.			Seconds at			Water at 20° C.		
	29° C.	59° C.	100° C.	Sp. Gr. 1.110 at 20° C.	50° C.	150° C.	20° C.	50° C.	150° C.	(54 Seconds) = 1.	20° C.	50° C.
Rape oil, refined .....	1.220	380	..	18.48	5.76	..	630	224	..	12.22	4.15	..
Cod-liver oil .....	799	262	..	11.51	3.97	..	430	165	..	7.96	3.06	..
American lubricating oil, pale, 0.905	905	215	..	13.71	3.26	..	475	140	..	8.80	2.59	..
Russian lubricating oil, pale, 0.908	1.320	595	..	65.5	9.01	..	2,310	325	..	13.33	6.57	..
American cylinder oil, pale .....	..	..	759	..	..	11.36	..	..	75	..	..	1.39
" " " dark .....	..	..	1,005	..	..	15.23	..	..	95	..	..	1.76
" Valvoline " cylinder oil .....	..	..	885	..	..	13.41	..	..	85	..	..	1.57

—J. L.

#### Apparatus for the Rapid Determination of Combustible Gases. G. G. Pond. Ber. 27, 692—696.

This apparatus, invented by Thomas Shaw, of Philadelphia, chiefly consists of two pumps, A and B (see figure); the

larger pump A constantly pumps the same volume, being fixed in position, as also is the length of the beam working the pump rod; the smaller pump B, on the other hand, is movable and the pump rod can be so arranged by means of





a scale along the beam as to deliver any required fraction of the maximum. A pumps air, or a mixture of air and combustible gas, whilst B pumps combustible gas only, viz., hydrogen, methane, illuminating gas, &c. Any required mixture of air and combustible gas can accordingly be made as required by pumping air by means of A, and the requisite proportion of combustible gas by means of B. In order to use the arrangement the sample of air or other mixture of gases containing inflammable gas (contained in an india-rubber bag) is first of all tested by pumping, together with air, into the testing cylinder C, and applying a light; if the explosion produced is violent a smaller quantity of the gas to be tested is employed relatively to the air, the mixture being pumped into the explosion cylinder D, and fired by a flame; if the explosion then produced is not too weak, the concussion projects a striker on to a bell, so as to ring a signal audibly. A series of trials is then made with gradually decreasing quantities of combustible gas until the limit is reached, when the violence of the explosion is not sufficient to ring the bell. For samples containing only small quantities of combustible gas, a series of observations is made with coal-gas (or hydrogen, &c.) and air, until a corresponding limit is arrived at, e.g., 8.0 per cent. of combustible gas; another series is then made with the air containing combustible gas to be tested and the same coal-gas (or hydrogen, &c.); if in this case the limit found is 5.3 per cent. of coal-gas, &c., it results that the quantity of combustible gas in the air tested is practically equivalent to  $8.0 - 5.3 = 2.7$  per cent. of the coal-gas, &c. used.—C. R. A. W.

## PATENTS.

*Improvements relating to the Sealing of Wires in Glass.*  
H. H. Lake, London. From J. B. Tibbatts, New York.  
U.S.A. Eng. Pat. 2048, January 30, 1903.

The method consists in using an annular collar made in the form of a dice-box, surrounding the wire to be sealed into glass at the point where the sealing-in is effected. The collar is of platinum or other suitable metal, and is soldered to the wire to be sealed in, and is in its turn fused into the glass by its edges, the waist being left free so that alteration of diameter of the wire by change of temperature can take place without straining the joint with the glass. The method is said to be adapted for the manufacture of incandescent lamps, and to be preferable to the plan of sealing in several small wires which is sometimes adopted when a large current has to be carried.—B. B.

INORGANIC CHEMISTRY.—  
QUANTITATIVE.

*Quantitative Analysis by Electrolysis.* O. Piloty.  
Ber. 27, 25, 280—282.

The author has made a number of electrolytic determinations of metals by Classen's method with the view of dispelling the doubt felt by many chemists as to their accuracy and applicability. He obtained good results in the precipitation of pure metals from their solutions, and the following figures in the separation of one metal from another or others:—

Separation.	Quantities taken and Precipitated.	Results per Cent.		Time of Electrolysis.
		Electrolytic.	Standard Method.	
Cu from Fe .....	1.0653 grms. $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ + about 0.5 grm. of "an iron salt."	25.33	25.39	2½ hours.
Cu from Fe, Ni, Co ....	Copper sulphate (0.5—1 grm.) + about 0.5 grm. of "iron, nickel, and cobalt salts."	25.63 25.77	.. ..	3 hours.
Fe from Cr .....	Iron ammonium sulphate (0.5845) + about 0.5 grm. chrome alum.	14.27	14.28	4 hours.
Fe from Al .....	Iron-ammonium sulphate (0.861 grm.) + about 5 grm. alum.	14.53	.. ..	4 hours.
Ni from Cr .....	Nickel ammonium sulphate (0.684 grm.) + chrome alum.	15.44	15.15	4 hours.
Pb from Cu .....	Lead nitrate (0.6—0.7 grm.) + copper sulphate .....	72.42—72.7	72.24	5 hours.
Sb from Sn .....	Antimony trisulphide (0.3—0.6 grm.) + about 0.5 of "tin salt."	72.6—72.85	.. ..	Left overnight.
Sb from As .....	Antimony trisulphide (about 0.8 grms.) + about 0.5 grm. sodium arsenate.	72.69 72.86	.. ..	Left overnight.

—B. B.

*The Volumetric Estimation of the Bases in certain Metallic Salts.* F. H. English, Chem. Trade J. 14, 1894, 258.

WHEN titrating soluble metallic salts capable of being precipitated as carbonates with sodium or potassium carbonates, using phenolphthalein as indicator, the characteristic red coloration does not appear until all the bases are completely precipitated. From the foregoing it will be seen that certain bases belonging to the first five groups may be estimated by means of a solution of sodium carbonate. The method of analysis is as follows:—Solutions required (1) seminormal sodium carbonate, and (2) an alcoholic solution of phenolphthalein. From 1 to 2 grms. of the salt are dissolved in water and allowed to boil, and then titrated while still hot with the soda solution, using the phenolphthalein as indicator. The solution should be stirred briskly during the titration. With a little practice the final reaction can be easily detected.

In the case of oxides and carbonates or salts insoluble in water, but soluble in HCl, the method of analysis has to be slightly varied. The substance is weighed into a beaker and dissolved in the slightest excess of HCl. When completely dissolved the excess of acid is neutralised by Na,  $\text{CO}_3$  solution, using as indicator a solution of methyl

orange. When neutral, add phenolphthalein and a little water, boil, and proceed as before. The results obtained by the above method compare favourably with other well-known methods as regards accuracy and speed. The following analyses are the mean of several determinations, and show the suitability of the test for technical work:—

Base sought.	Salt taken.	Soda Method.	Gravimetric Method.
PbO .....	$\text{Pb}(\text{NO}_3)_2$ .....	67.17	67.13
$\text{Al}_2\text{O}_3$ .....	$^*\text{Al}_2(\text{SO}_4)_3 \cdot 15\text{H}_2\text{O}$ .....	17.99	17.98
ZnO .....	$\text{ZnSO}_4 + 7\text{H}_2\text{O}$ .....	28.38	28.38
CaO .....	$\text{CaCO}_3$ .....	55.86	55.88
BaO .....	$\text{BaCl}_2 + 2\text{H}_2\text{O}$ .....	62.50	62.50

\* Commercial salt.

This method is admirably adapted for the rapid estimation of lime in limestone and sifted lime, and is useful for those engaged in chemical works laboratories, where these

bodies have to be tested daily. It may also be of use to the practical dyeworks chemist for the estimation of aluminium in red liquors, and lead in lead nitrate liquors; of course, in estimating any individual base no other base capable of being precipitated as carbonate must be present. The method is not applicable in presence of ammonium compounds, which must be got rid of before applying the test.

*Estimation of Calcium Sulphide in Animal Charcoal by Means of Bromine.* P. Herrmann. *Die Deutsche Zuckerei* 1893, 18, 225.

25 grams of the finely-powdered charcoal are moistened, in a 250 cc. flask, with water, 2 to 3 cc. bromine added, and then 100 cc. luke-warm water. The whole is well shaken and then digested on the water-bath for half an hour. 80 cc. of hydrochloric acid of sp. gr. 1.1 are then slowly added, care being taken that the bromine vapours are not dispersed during this operation, so that there may be no chance of escape of hydrogen sulphide. The excess of bromine is then driven off over a naked flame, the volume, after cooling, made up to 250 cc., and then filtered and the sulphuric acid estimated in 200 cc. of the filtrate. After subtracting the barium sulphate due to the calcium sulphate originally present in the charcoal, multiplication of the residual weight of barium sulphate by the factor 1.515 gives the percentage of calcium sulphide in the charcoal. This method is quite as accurate as the nitric acid or potassium chlorate methods.—L. T. T.

*Method for the Determination of Iron in Iron Ore.* Messrs. Mixer and Dubets. *Eng. and Mining J.* 1894, 342.

It is rather surprising that the method described below, although in general use in the Lake Superior region, should be so little known outside of it. The method may be named, in a descriptive sense, the "stannous chloride, hydrochloric acid, and permanganate" method, and the authors do not know to whom credit should be given for having first formulated it.

For ordinary work less than half a gram. of ore is weighed out, the particular weight and the strength of the permanganate solution being so adjusted that by using a 50-cc. burette the per cent. may be read directly, the value of 1 cc. being 2 per cent.

A carboy is filled with a solution containing 250 grms. of permanganate and connected directly with the burette by glass tubing.

For extremely accurate work a 100-cc. burette may be used, with a more dilute solution.

The ore weighed out is placed in a suitable beaker, and 2½ cc. of stannous chloride solution added. (This solution is made up as follows:—One pound of stannous chloride is dissolved in one pound of concentrated hydrochloric acid and water, and diluted to 2 litres.) Then 10 to 15 cc. of hydrochloric acid (1:1) are added, a watch glass put on the beaker, which is then placed on an iron plate, and the contents boiled until the ore is completely dissolved. This will generally require from one to five minutes, depending upon the character of the ore, and the proportion of stannous chloride used, it being most advantageous to have enough stannous chloride present to reduce nearly all the iron to the ferrous state, that is, so that the solution only has a light yellowish-green colour. The rapidity with which the ore dissolves, owing to the presence of the stannous chloride, is a great advantage in shortening the time required for the analysis, and is of considerable theoretical interest as to its exact cause.

When the ore is dissolved, and whilst still hot, additional drops of stannous chloride are cautiously added from a burette, until the yellow colour just disappears, the solution being constantly agitated by giving the beaker a slight rotating motion, thus doing away with the use of a glass rod. To the contents of the beaker are added about 5 cc. of a saturated solution of mercuric chloride, to take up the slight excess of stannous chloride, which reaction is shown by the formation of mercurous chloride, a white, silky

precipitate. The solution is now poured into the titrating beaker, of about 500 cc. capacity, diluted with water, and 5 to 10 cc. of the titrating solution added. The titrating solution is prepared by dissolving 160 grms. of manganese sulphate in water and diluting to 1,750 cc., to which are added 330 cc. of phosphoric acid and 320 cc. of sulphuric acid.

The solution is now ready for titrating, which is accomplished in the usual way. After the mercuric chloride has been added the titration must be performed immediately.

As is well known, the permanganate solution rarely varies, when protected from light and changes in temperature, by easing around the carboy.

Nevertheless, the authors always make sure of it by making one or two analyses of a standard ore with every set of determinations, which, being under precisely the same conditions, will immediately indicate any error in weight or solution; this precaution we consider very important, as it takes little extra work and insures the accuracy of the results.

Duplicates generally agree, and rarely differ more than one-tenth of one per cent. In a standard ore that was recently submitted to several chemists for analysis, the three chemists using this method reported 67.02 per cent., 67.02 per cent., and 67.08 per cent. as their respective results, and the average by all methods was 67.04 per cent.

There is claimed for this method greater rapidity of working and equal accuracy with any other method now employed in iron analysis of ores.

*Estimation of Chromium and Manganese in Steel.* L. Schneider. *Oester. Zeits. f. Berg- und Hüttenkunde*, 1892, 235.

The author's process is based upon the conversion of the lower oxides of these metals into the highest (acid) oxides by the action of lead peroxide in presence of nitric acid. The metal (2 grms.) is dissolved in dilute sulphuric acid (100 cc.), and after oxidising with nitric acid (5 cc.) lead peroxide (5 grms.) is added, and boiled with the solution for 15 minutes. The chromic and manganic acids resulting are estimated volumetrically in the usual way.—C. F. C.

*Colorimetric Estimation of Iron in Spring Waters.* F. Gerhard. *Archiv. der Pharm.*, 230, 705.

The author makes use of the well-known "tannin" reaction. The colour develops most satisfactorily and uniformly in presence of sodium pyrophosphate, which whilst giving the necessary slightly alkaline reaction forms a soluble double salt with ferric compounds. The standard solutions of the latter (0.0001 Fe per cc) is prepared by taking the necessary quantity of a ferric salt, dissolving together with 2.5 grms. sodium pyrophosphate and diluting to 1000 cc. The details of the estimations are of the usual order.—C. F. C.

*The Effect of Platinum in Iron Solutions.* R. W. Mahon. *Americ. Chem. J.* 1893, 15, 578—582.

The minute quantity of platinum which a platinum crucible, used for the fusion of insoluble iron ore residues with sodium carbonate, may lose, is imparted to the subsequent solution of the fusion in water and hydrochloric acid. These traces of platinum form, according to the author, a source of error, when the iron is estimated by titration with potassium bichromate, after reduction by stannous chloride, dilution, and addition of mercuric chloride in excess. Stannous chloride added to the hot hydrochloric acid solution of ferric and platonic chlorides is described as first reducing the iron, and then the platinum. The solution which becomes gradually colourless during the reduction of the iron, again assumes a yellow tinge, due to the trace of platinous chloride which is formed. Moreover, when mercuric chloride is added, as above described, the substance sometimes has a tinge of colour, whilst, when platinum is absent, a colourless solution with pure white mercurous chloride in suspension

occurs. The titration with potassium bichromate shows the usual reaction with potassium ferri-cyanide, but when platinum is present, a recurrent bluing after the completion of this colour reaction may be encountered. This recurrence is sometimes rapid enough to amount to a continuous coloration. Thus a small quantity of platinum may introduce a considerable error into the estimation.

To prevent this, the author recommends two methods:—Firstly, to add the stannous chloride with great care, and to cease when the colour due to the ferric compound has disappeared, and before the above-described yellow tinge due to platinous chloride has made its appearance. Secondly, to precipitate the iron in the fused and dissolved residue by means of ammonia, to collect the precipitate on a small filter, and after thorough washing to redissolve it in hydrochloric acid and hot water, whereby a solution is obtained which is entirely free from platinum. In cases where great accuracy is required this second method is preferable to the first.—H. S.

#### Quantitative Electrolytic Estimation of Lead.

A. Kreichgauer. Ber. 27, 315—318.

CHECK experiments on the electrolytic formation of lead dioxide from an acid solution of lead nitrate indicated that with a solution containing 1 part of nitric acid sp. gr. 1.14 and 5 to 7 of water, an excess of lead was indicated of a few tenths per cent., which was lessened by washing twice with alcohol to slightly below the theoretical amount. A current capable of furnishing 0.2 cc. of detonating gas per minute precipitated all lead from solution in 14 hours, but no error was noticed on prolonging the duration to upwards of 40 hours; no difference was observed whether the dioxide thrown down was weighed after drying at 110° or at 200°. A set of four Meidinger cells and the dish electrode arrangement recommended by Claisen were employed.

—C. R. A. W.

#### The Use of Lead Pipes for Conveying Water. M. T. Leeco. Chem. Zeit. 17, 1431—1432.

THE tests described in this paper were made upon the new water supply of the city of Belgrade (a full analysis of the water is given). This water contains iron, calcium, and magnesium salts, is of 24.8° total hardness (German scale), and holds in solution more than 0.5 gm. CO<sub>2</sub> per litre.

Lead was tested for, in water which had stood over night in the pipes, as follows:—One litre of water acidified with acetic acid was evaporated down to 100—200 cc., filtered, and a drop of dilute sulphuretted hydrogen water added. If lead is present, even in the most minute quantity, a darkened coloration of the water is observed. As this reaction might be partially obscured by the precipitation of the iron present in the water, it is necessary to make a test at the same time with a sample known to be free from lead.

An estimation of the amount of lead may be carried out with great accuracy in the following way:—One litre of the water is acidified with 5 cc. glacial acetic acid, concentrated to 100 cc., filtered, and one or two drops of dilute H<sub>2</sub>S water added (one part saturated H<sub>2</sub>S water with two parts distilled water). At the same time the sample known to be free from lead is treated in an exactly similar manner. If lead is detected in the first sample, add to the second such an amount of a solution containing 0.1 mg. lead in 1 cc. That the two solutions have exactly the same colour. The amount of lead added to the second sample is the amount present in the first. By this means 0.05 mg. lead per litre may be estimated.

In carrying out the tests here described a piece of lead pipe 7 metres in length, and 22 mm. in diameter was employed. The solvent effect of the water on the lead was greater at first than after the tube had been some time in use. But even after three months, about 0.3 mg. lead per litre was found in water which had stood 24 hours in the pipe. After three hours standing a trace of lead was detected, but in water which had merely run through the

pipe no lead could be found. Therefore, where lead pipes are used the water should be allowed to run for a few minutes before taking it for drinking purposes.

The somewhat large amounts of lead dissolved by this particular water may be due to its richness in free carbonic acid. The author recommends that subsequent papers on this subject should include a full analysis of the water, in addition to the method adopted for estimating lead. If this suggestion were fully carried out, it would be possible, with the help of medical men who would inform us as to the amount of lead rendering water unfit for drinking, to definitely solve the question of the suitability of lead pipes for conveying water.—R. B. B.

#### Quantitative Analysis by Electrolysis.—The Electrolytic Determination of Lead. A. Claisen. Ber. 27, 163—165.

AS Luckow has shown, the complete oxidation of lead to peroxide is, in the first place, dependent upon the presence of a certain amount of nitric acid. The quantity requisite, as was first proved in the electro-technical laboratory at Munich, must be regulated by the temperature of the solution and the density of the current used. It was found that the latter depended upon the nature of the anode; with an anode with a very smooth surface the current density must only be 0.05 ampère per 100 sq. cm., under other conditions, 0.5 ampère. Whilst observing all these conditions, the quantity of peroxide that can be precipitated in an adhering form is relatively small.

The rapid deposition of large amounts of lead peroxide can, however, be effected if the platinum dish used as anode is roughened on its inner surface by a sand-blast. By this means in a few hours as much as 4 grms. of peroxide per 100 sq. cm. of surface can be precipitated with a current of 1.5 ampères.

To make this estimation, after dissolving the lead salt, 20 cc. of nitric acid (sp. gr. 1.35—1.38) is added, the liquid is diluted to about 100 cc., then warmed to 50°—60° and electrolysed with a current of  $N.D_{100} = 1.5—1.7$  ampère. By continuing the warming during the electrolysis as much as 1.5 grms. of lead peroxide can be precipitated in three hours, and larger quantities in from four to five hours. To prove the complete separation of the lead, about 20 cc. of water are added, and it is observed whether or not there is any darkening of the freshly-wetted surface of the anode. If after 10—15 minutes, no darkening is visible, the precipitate is washed with water and alcohol, and dried at 180°—190°. A temperature of 110° to 130°, as prescribed by some other chemists, does not suffice. The intensity of the current does not influence the character of the precipitate; in the author's experiments it varied from 4 to 8 volts.

When copper and lead are to be estimated in the same solution, after adding 20 cc. of nitric acid, the volume of liquid is only made up to about 75 cc., and the warm solution is then electrolysed with a current of  $N.D_{100} = 1.5$  to 1.7 ampère; but at the end of an hour the operation is interrupted. By this time from 98—99 per cent. of the lead will have deposited upon the anode, but none of the copper will have deposited on the cathode. The liquid is now transferred to another tared dish, and the precipitated lead peroxide washed, dried, and weighed. The washings are added to the liquid in the second dish. To the solution containing the whole of the copper and the rest of the lead, ammonia is added until the blue colour of an ammoniacal copper solution is permanent, after which about 5 cc. of nitric acid are added, the solution is made up to 120—150 cc., and is then electrolysed with a current of  $N.D_{100} = 1$  to 1.2 ampère. In this electrolysis, however, the tared dish is made the cathode upon which the copper deposits, and the perforated platinum foil or "bucket-electrode" already described by the author (Handbuch der Elektrolyse, p. 56), is used as anode upon which the remainder of the lead peroxide in deposits. From three to four hours are required to separate about 0.25 grms. of copper and the rest of the lead.

When dealing with substances containing sulphur, the separation of lead sulphate frequently causes trouble owing to the difficulty of bringing it into solution in nitric acid.

When lead sulphate forms during solution of the substance to be analysed, ammonia must be added to the liquid in slight excess, and heat applied for a few minutes. This converts the dense lead sulphate into the bulkier lead hydrate. The liquid is then poured, little by little, into the platinum dish, into which about 20 cc. of nitric acid have previously been placed and warmed, whilst it is stirred continuously with the electrode. The lead sulphate that is thereupon formed now redissolves at once either the whole of it or the greater part, so that the remainder dissolves after warming for a short time. The vessel in which the treatment with ammonia was carried out is first rinsed out with a little nitric acid and then with water.

The above described modification can be applied to the separation of large quantities of manganese peroxide, and also to the precipitation of some metals, antimony for example, which hitherto could only be separated in minute quantities. The author intends to make further communications on his experiments on these and other allied points.—H. S. P.

*The Separation and Estimation of Metals in Alkaline Solution by means of Hydrogen Peroxide.* P. Jannasch and J. Lesinsky. Ber. 26, 2908—2912.

SEPARATION of bismuth from copper:—

(1.) *In the Cold.*—A mixture of 50 cc. of 3 per cent. hydrogen peroxide with 15 cc. of concentrated ammonia was gradually added to a solution of 0.3 gram. of each of the pure metals. The faintly yellow precipitate of bismuth hydroperoxide is washed first with a mixture of 2 vols. of hydrogen peroxide, 1 vol. of strong ammonia, and 8 vols. of water, then with warm diluted ammonia (1:8) and ultimately with hot water, care being taken to ensure the complete removal of the copper. After drying at  $90^{\circ}$ — $95^{\circ}$  the precipitate is ignited in a platinum crucible, redissolved in nitric acid and ignited a second time until the weight remains constant. The barium salts and silicic acid contained in commercial hydrogen peroxide are carried down with the bismuth precipitate, hence the results turn out too high and must, if absolute accuracy be desired, be corrected by subtracting the estimated amount of these impurities from the original weight of the bismuth oxide. The copper contained in the filtrate from the bismuth precipitate is, after expulsion of the ammonia, separated from the re-acidulated and diluted solution as sulphide, converted into oxide by ignition in a current of oxygen and weighed as such.

(2.) *With the Application of Heat.*—The solution to which the hydrogen peroxide mixture was added in the cold is heated from 5 to 10 minutes on the water-bath, filtered hot, and the precipitate washed as in (1), but with hot liquors exclusively. If no hydroxylamine be present in the solution the bismuth precipitate retains but extremely small traces of copper, in the presence of a more considerable quantity of hydroxylamine these traces become more noticeable, but may be completely removed by a second precipitation.

Compared with the usual method of separating bismuth from copper by means of ammonia or ammonium carbonate the separation of these metals by means of hydrogen peroxide presents the advantages of greater speed and accuracy.—F. M.

*The use of Ammoniacal Mercuric Cyanide in Quantitative Analysis.* F. W. Schmidt. Ber. 27, 225.

THE conversion of metallic sulphides, precipitated in the ordinary course of analysis, into some form capable of satisfactory weighing, involves either re-solution and precipitation as oxide or carbonate or treatment by Rose's method with sulphur in a stream of hydrogen to obtain an anhydrous sulphide. The process of ignition with mercuric oxide or with a mixture of that substance and mercuric nitrate is not invariably available as basic sulphates are sometimes formed, difficult of decomposition at the temperature of ignition. The conversion can, however, be effected by the use of ammoniacal mercuric cyanide, a cyanide of the metal to be determined being formed, whilst mercuric sulphide is produced in equivalent amount. The metallic cyanide is decomposed on heating in the air, the

mercuric sulphide volatilises, and an oxide of the metals remains. No formation of mercuric sulphocyanide occurs and thus the removal of mellone (a fairly fixed substance) is avoided. Similarly para cyanogen from the decomposition by heat of the excess of mercuric cyanide is not obtained when an ammoniacal solution of the cyanide is used, and thus the difficulty of volatilising so fixed a body, does not arise. This difference in the behaviour of mercuric cyanide and the same salt in ammoniacal solution is due to the formation of the body  $\text{Hg}(\text{CN})_2\cdot\text{NH}_3$ , which the author has prepared and examined. It can be obtained by saturating a strong solution of ammonia with mercuric cyanide, at a gentle heat on the water-bath, and strongly cooling the solution. Crystals several centimetres in length separate, and can be dried in an atmosphere of ammonia; on exposure to air they lose ammonia, and rapidly decompose.  $\text{Hg}(\text{CN})_2\cdot\text{NH}_3$  is with difficulty soluble in water, its aqueous solution is immediately precipitated by  $\text{H}_2\text{S}$ , but when previously made acid with hydrochloric acid, it gives with  $\text{H}_2\text{S}$  at first a yellow, and then a white, precipitate, the latter becoming black on dilution.

The general method of using ammoniacal mercuric cyanide for converting metallic sulphides into oxides, consists in placing the moist precipitate together with the filter in a crucible, covering it with a solution of the reagent, evaporating to dryness, then heating first gently with a Bunsen flame, then strongly, if necessary, over the blow-pipe. In the case of copper and zinc, this treatment suffices and no further description is necessary, but with bismuth, reduction of a portion to metal takes place and must be corrected by subsequent evaporation with nitric acid and ignition of the nitrate to oxide. The conversion of ferrous sulphide to ferric oxide is also certain and complete. A similar result is to be expected for nickel and cobalt.

—B. B.

ORGANIC CHEMISTRY.—QUALITATIVE.

*The Detection of "Abrastol," Calcium  $\beta$ -naphthol Sulphonate in Wines.* Sinibaldi. Monit. Scient. 7, 842.

ABRASTOL forms a pinkish-white powder, of which 100 parts of water or alcohol dissolve respectively 167 and 50 parts at  $15^{\circ}\text{C}$ . Its most characteristic reaction is the slatey-blue coloration it yields with ferric chloride. This coloration changes to green on warming. Abrastol when heated with hydrochloric acid, is converted into naphthol, sulphuric acid, and calcium sulphate. The detection of abrastol in wine may be effected as follows:—25 cc. of wine are neutralised by the addition of a few drops of ammonia, and shaken with 25 cc. of pure amyl alcohol. After separation the amyl alcohol is boiled to expel any traces of ammonia, and after cooling is agitated with  $\frac{1}{4}$  cc. of ferric chloride solution. In presence of abrastol the amyl alcohol assumes the slatey-blue coloration already mentioned. In the case of wines containing but little abrastol, 250 cc. should be evaporated at a low temperature (in vacuo, if possible), the residue extracted by 30 cc. of absolute alcohol, and the alcoholic extract evaporated to dryness. The residue is dissolved in water and treated with amyl alcohol as above (see page 534).—H. T. P.

ORGANIC CHEMISTRY.—QUANTITATIVE.

*A New Method of Estimating Naphthol- and Naphthylamine Sulphonic Acids.* W. Vaubel. Chem. Zeit. 1893, 1265 and 1897.

THE property of these sulphonic acids of combining with one or more molecules of bromine is utilised for their volumetric estimation. The *modus operandi* consists in adding a standard solution of potassium bromate to a mixture of an aqueous solution of the sulphonic acid with potassium bromide and sulphuric acid until the liquor permanently shows the presence of free bromine. The acids, according to their capacity of taking up bromine, may be classed as follows:—

1. Sulphonic acids, which at the ordinary temperatures, generally take up only one atom of bromine, and in case of

which the completion of the reaction is easily recognised. This group includes—

	NH <sub>2</sub>	SO <sub>2</sub> H	SO <sub>3</sub> H
<b>A. of α- and β-naphthylamine sulphonic acids:</b>			
(a.) α-naphthylamine sulphonic acid	1	2	..
(b.) Naphthosmic acid	1	1	..
(c.) Dahl's disulphonic acid II.	1	1	6
(d.) " " III.	1	1	7
(e.) α-naphthylamine-δ-disulphonic acid.	1	4	8
(f.) Amido-R-acid	2	3	6
(g.) p-monosulphonic acid (2Br) also	2	5	..
Naphthylene monosulphonic acid	NH <sub>2</sub> 1	NH <sub>2</sub> 6	SO <sub>2</sub> H 1

	OH	SO <sub>2</sub> H	SO <sub>3</sub> H	SO <sub>3</sub> H
<b>B. of α- and β-naphthol sulphonic acids:</b>				
(a.) Neville and Wither's acid.	1	4	..	..
(b.) α-naphthol disulphonic acid.	1	1	8	..
(c.) Schaeffer's acid	2	6	..	..
(d.) F-acid	2	7	..	..
(e.) R-acid	2	3	6	..
(f.) β-naphtholtrisulphonic acid.	2	3	6	8

II. Sulphonic acids taking up more than one atom of bromine and showing less plainly the end of the reaction—

	NH <sub>2</sub>	SO <sub>2</sub> H	SO <sub>3</sub> H
<b>A. α- and β-naphthylamine sulphonic acids:</b>			
(a.) α-naphthylamine-δ-sulphonic acid (3Br).	1	7	..
(b.) α-naphthylaminemonosulphonic acid S (2Br).	1	8	..
(c.) α-naphthylamine-β-disulphonic acid (2Br).	1	3	7
(d.) α-naphthylamedisulphonic acid Kalle (2Br).	1	2	7
(e.) Bronner's acid (3Br)	2	6	..
(f.) β-naphthylamine-δ-monosulphonic acid I (3Br).	2	7	..
<b>B. α-naphtholmonosulphonic acid S</b>			
	OH 1	8	..

III. Sulphonic acids combining with bromine only at high temperatures. The following take up one bromine atom at 65°—70°.

	NH <sub>2</sub>	SO <sub>2</sub> H	SO <sub>3</sub> H
β-naphthylamine-α-monosulphonic acid	2	8	..
Amido-G-acid	2	6	8
<b>OH</b>			
Croceine sulphonic acid	2	8	..
G-acid	2	6	8

The combination of the bromine method with the well-known method based upon the formation of diazo-com-

pounds in many cases furnishes a means of estimating separately the sulphonic acids contained in a mixture.

—F. M.

#### Errata.

This Journal 1894, 428, 35 lines from top of first column, for "The best" read *Isolu*.  
"The best" lines 20 and 23 from bottom of col. 1, for "Chloroscope" read *Chloroscope*.

## New Books.

**PRACTICAL PAPER-MAKING.** A Manual for Paper Makers and Owners and Managers of Paper Mills, to which are appended useful Tables, Calculations, Data, &c. By GEORGE CHAMBERLAIN. London: Crosby, Lockwood, and Son, 7, Stationers' Hall Court, Ludgate Hill. 1894. Price 5s.

8vo. volume, bound in cloth, and containing 198 pages of subject-matter, exclusive of preface, table of contents, and alphabetical index. There is also a frontispiece containing two figures representing the microscopic structure of cotton, manilla, and hemp. The text is illustrated with 16 microphotographs representing the appearances of cellulose fibres and their mineral adulterants, or "filling" agents. The Appendix supplies various data of value and interest to manufacturers. For example, we have the "Equivalent Weights and Sizes of Writing Papers," "Sizes of Book and Drawing Papers," "Various Calculations relating to Weight of Reams and Webs," &c., &c.

The following subjects are treated of in the work:—I. Chemical and Physical Characteristics of Various Fibres. II. Cutting and Boiling of Rags. Jute Boiling and Bleaching. III. Wet Picking, Washing, Breaking, and Bleaching. Electrolytic Bleaching. Antichlor. IV. Cellulose from Wood. Mechanical Wood Pulp. V. Esparto and Straw. VI. Beating. VII. Loading. Starch. Colouring Matter. VIII. Resin, Size, and Sizing. IX. The Fourdrinier Machine and its Management. X. Animal Sizing. Drying. XI. Glazing and Burnishing. XII. Cutting. Finishing. XIII. Microscopical Examination of Paper. XIV. Tests for Ingredients of Paper. XV. Recovery of Soda. XVI. Testing of Chemicals. Testing Water for Impurities.

**LAW AND THEORY IN CHEMISTRY.** A Companion Book for Students. By DOUGLAS CARNEGIE, M.A. London: Longmans, Green, and Co. New York: 15, East 16th Street. 1894. Price 6s.

This book is stated to contain the substance of a course of eight lectures delivered before an audience of school teachers of Elementary Chemistry at Colorado College, U.S.A. The character of the work may be summed up as the history of the development of modern chemical theory, and the subject is subdivided as follows:—I. Alchemy and the Birth of Scientific Chemistry. II. The Phlogistic Period and the Beginnings of Chemical Theory. III. Chemical Classification. Mixtures. Compounds. Elements. IV. The Atomic Theory. V. The Classification of Compounds, Acids, Bases, Salts. VI. Isomerism and Molecular Architecture. VII. Chemical Equilibrium. The text occupies 222 pages, the book is of 8vo. size, and is strongly bound in cloth.

**ENGINEERING CHEMISTRY.** A Practical Treatise for the Use of Analytical Chemists, Engineers, Ironmasters, Ironfounders, Students, and others. Comprising Methods of Analysis and Valuation of the Principal Materials used in Engineering work with numerous Analyses, Examples, and Suggestions. By H. JOSHUA PHILLIPS, F.I.C., F.C.S. Second Edition, Revised and Enlarged. London: Crosby, Lockwood, and Son, 7, Stationers' Hall Court, Ludgate Hill. 1894. Price 10s. 6d.

This is a book the object of which is mainly to give precise methods for analysing and valuing the most important of

the materials in general use by engineers. The author having long entertained the idea of writing such a volume, and having been continuously engaged in the laboratories of railway companies, has gradually collected together the materials for it. This volume, which is strongly bound in cloth, forms an 8vo. containing 388 pages of subject matter, to which is added an alphabetical index. In the text are to be found some 50 woodcuts, and numerous tables. The sub-division of the text into chapters under the primary subjects or themes of which they treat, will give most concisely the plan of the work and its detailed method of arrangement. **EXCEPTION:** Reddrop's System of Reagents. I. Metals, Alloys, &c. II. Ores, Limestones, &c. III. Fuels.—Solid, Liquid, and Gaseous. IV. Water. V. Oils. VI. Materials used in Grease Making. VII. Gasworks Products. VIII. Disinfectants. IX. Explosives. Appendix, with a variety of tabular matter.

**THE TANNINS.** A Monograph on the History, Preparation, Properties, Methods of Estimation, and Uses of the Vegetable Astringents, with an Index to the Literature of the Subject. Vol. II. By HENRY TRIMBLE, PH.D., Professor of Analytical Chemistry in the Philadelphia College of Pharmacy, Philadelphia, U.S.A. J. B. Lippincott Company. 1891.

THIS second volume by Prof. Henry Trimble on the Tannins, is devoted to the results of investigation by him on the astringent principles from nine species of oaks and one species each of mangrove, canaigre, and chestnut.

Oak barks have been collected and examined from three different quarters of the globe, so that conclusions regarding the oak tannins are not based on too narrow foundations.

A valuable index to the literature of the tannins is given and occupies some 30 pages, the total text filling 170 pages. The work concludes with an alphabetical index of subject matter and another of authors. It is an 8vo. volume and bound in cloth.

## Trade Report.

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

#### NEW CUSTOMS TARIFF OF BRITISH INDIA.

A copy of the Indian Tariff Act of 1894, which received the assent of the Governor-General on the 10th March last, has been received at the Board of Trade from the India Office. The following is a statement of some of the rates of import duties now levied under this Act:—

#### Import Tariff.

No.	Names of Articles.	Tariff Valuation.	Duty.
<i>Chemicals, Drugs, Medicines, and Narcotics, and Dyeing and Tanning Materials.</i>			
7	Chemical products and preparations:	Rs. a.	
	Acid, sulphuric .....	Lb. 0 2	5 per cent.
	Alkali, country (sajji-khâr) Cwt.	2 0	"
	Alum .....	" 5 0	"
	Arsenic .....	" 25 0	"
	Do., China mansil .....	" 16 0	"
	Bicarbonate of soda .....	" 7 0	"

#### Import Tariff—continued.

No.	Names of Articles.	Tariff Valuation.	Duty.
	Chemical products, &c.— <i>cont.</i>	Rs. a.	
	Copperas, green .....	Cwt. 3 0	5 per cent.
	Explosives, namely, blasting gelatin, dynamite, roborite, tonite, and all other descriptions, Sal ammoniac .....	Cwt. 30 0	"
	Sulphur (brimstone), flour ..	" 5 8	"
	Do., do., roll .....	" 5 0	"
	Do., do., rough .....	" 4 0	"
	All other sorts of chemical products and preparations.	<i>ad val.</i>	"
8	Drugs, medicines, and narcotics:		
	Aloes, black .....	Cwt. 14 0	"
	Do., Socotra .....	" 25 0	"
	Aloe wood .....	Lb. 3 0	"
	Asafoetida (hing) .....	Cwt. 80 0	"
	Do., coarse (hingra) .....	" 18 0	"
	Atary, Persian .....	" 15 0	"
	Brimstone (amalsara) ..	" 45 0	"
	Camphor, Bhemasani (baras) ..	Lb. 100 0	"
	Do., refined, cake .....	" 1 0	"
	Do., crude, in powder ..	" 0 12	"
	Cassia lignea .....	Cwt. 20 0	"
	China root (chobchini), rough ..	" 7 0	"
	Do., do., scraped .....	" 16 0	"
	Galangal, China .....	" 8 0	"
	Pellitory (takalkara) .....	" 30 0	"
	Salep .....	" 100 0	"
	Senna leaves .....	" 4 8	"
	Storax, liquid (rose mel-foet) ..	" 46 0	"
	Tobacco, manufactured and un-manufactured.	<i>ad val.</i>	"
	All other sorts of drugs, medicines, and narcotics, except quinine, which is free, and opium (for which see Schedule of liquors, opium, salt, and salted fish).	"	"
9	Dyeing and tanning materials:		
	Aizarin dyes, all colours .....	"	"
	Aniline, do., do. ....	Lb. 1 8	"
	Cochineal .....	" 1 0	"
	Gallnuts, myrabolans ..	Cwt. 4 8	"
	Do., Persian .....	" 37 0	"
	Madder or manjit .....	" 12 0	"
	Orchilla weed .....	" 9 8	"
	Sappan wood and root ..	" 5 8	"
	All other sorts of dyeing and tanning materials.	<i>ad val.</i>	"
<i>Oils.</i>			
	Cassia .....	Lb. 3 0	"
	Cocunut .....	Cwt. 13 0	"
	Grass .....	Lb. 1 12	"
	Junjili or til .....	Cwt. 15 0	"
	Linseed, European .....	Imp. gall. 1 12	"
	Oil, of sorts .....	Ounce 10 0	"

## Import Tariff—continued.

No.	Names of Articles.	Tariff Valuation.	Duty.
	Petroleum, including also naphtha and the liquids commonly known by the names of rock-oil, Ranceon oil, Borina oil, kerosene, paraffin oil, mineral oil, petrolene, gasoline, benzol, benzoline, benzine, and any inflammable liquid which is made from petroleum, coal, schist, shale, peat, or any other bituminous substance, or from any products of petroleum.	Rs. 36.	1 anna per imp. gall.
	Do., which has its flashing point at or above 200° of Fahrenheit's thermometer, and is proved to the satisfaction of the Customs collector to be intended for use exclusively for the latching of jute or other fibre or for lubricating purposes.	<i>ad val.</i>	5 per cent.
	Sandalwood..... Lb.	8 0	"
	Turpentine..... Imp. gall.	1 14	"
	Whale (except spermaceti) Cwt.	15 0	"
	Wood..... "	25 0	"
	All other sorts of oil.....	<i>ad val.</i>	"
<i>Other Articles. Unmanufactured and Manufactured.</i>			
7	Candles, paraffin..... Lb.	0 5	"
	Do., spermaceti..... "	0 6	"
	Do., wax..... "	1 0	"
	Do., all other sorts, including composition..... "	0 5	"
31	Gums, gum-resins, and articles made of gum or gum resin:		
	Copal..... Cwt.	55 0	"
	Cuteh and gambier..... "	17 0	"
	Gum ammoniac..... "	14 0	"
	Do., arabic..... "	18 0	"
	Do., Bellium (common gum)..... "	10 0	"
	Do., Benjamin..... "	40 0	"
	Do., Bysibol (coarse myrrh)..... "	14 0	"
	Do., Oilinum or frankincense..... "	12 0	"
	Do., Persian (false)..... "	10 0	"
	Do., Kino..... "	10 0	"
	Myrrh..... "	30 0	"
	Rosin..... "	1 4	"
	All other sorts of gums, gum-resins, and articles made of gum or gum resin.....	<i>ad val.</i>	"
39	Leather, and articles made of leather, including boots and shoes, harness and saddlery, but excluding belting for driving machinery, which is free.	"	"
41	Matches, lucifer, and all other sorts.....	<i>ad val.</i>	"
43	Oilcloth and floorecloth.....	"	"
44	Paints, colours, painters' materials, and compositions for application to leather and metals:		
	Ochre, other than European, all colours..... Cwt.	1 5	"
	Paints of sorts..... "	9 0	"
	Do., composition..... "	55 0	"
	Do., patent driers..... "	10 0	"
	Prussian blue, China..... Lb.	0 10	"
	Do., European..... "	1 0	"

## Import Tariff—continued.

No.	Names of Articles.	Tariff Valuation.	Duty.
	Beets, colours, &c. ....	Rs. 36.	5 per cent.
	Red-flower..... Cwt.	12 0	"
	Turpentine..... Imp. gall.	1 14	"
	Verdigris..... Cwt.	70 0	"
	Vermillion, Canton..... { Box of }	94 0	"
	White lead..... Cwt.	14 0	"
	All other sorts.....	<i>ad val.</i>	"
45	Paper of all kinds.....	"	"
46	Perfumery:		
	Rose flowers dried..... Cwt.	12 0	"
	Rosewater..... Imp. gall.	1 14	"
	All other sorts, except perfumed spirit (for which see Schedule of liquors, opium, salt, and salted fish).....	<i>ad val.</i>	"
49	Pitch, tar, and dammer:		
	Bitumen.....	"	"
	Dammer..... Cwt.	6 0	"
	Pitch, American and European.....	6 0	"
	Do., coal.....	2 8	"
	Tar, American and European.....	6 0	"
	Do., coal.....	2 8	"
	Do., mineral.....	<i>ad val.</i>	"
53	Soap.....	"	"
54	Stationery, excluding paper (for which see No. 45).....	"	"
55	Stone and marble.....	"	"
56	Tallow and grease..... Cwt.	25 0	"

## THE NEW RUSSO-GERMAN COMMERCIAL TREATY.

We give below the principal pharmaceutical articles on which Russia has agreed to lower the Customs duty in the new commercial treaty with Germany. In cases where these rates are lower than those hitherto levied upon imports from countries with which Russia trades on the "most-favoured-nation" basis, those countries (of which the United Kingdom is one) participate in the benefit. The rates are given in roubles per pound, the figures in ( ) denoting the rates at present levied under the "war" tariff:—

Salts of chromic acid soluble in water—*e.g.*, bichromate of potash, neutral chromate of potash, chromate of soda, 2·15 (2·65); tannic acid (taunin) 5·00 (6·00); blue copperas, except anhydrous copperas, Salzburg copperas (a mixture of sulphates of iron and copper), white copperas or sulphate of zinc, chloride of zinc, 0·80 (1·00); tartar emetic, 3·00 (4·00); chemical and pharmaceutical products not specially tariffed, gross weight, 1·50 (gross weight, 1·80); proprietary medicines the importation of which is authorised in accordance with the special list, gross weight, 16·00 (gross weight, 16·00); precipitated or washed chalk, chalk and tale ground, gross weight, 0·12 (gross weight, 0·15); white-lead and zinc-white, 0·50 (0·60); red-lead, 0·35 (0·40); colours with base of copper (except verdigris, or of arsenic), 3·00 (4·00); verdigris (basic acetate of copper), 3·60 (4·00); dyeing substances (pigments) prepared with products from the distillation of coal-tar; alizarine, extract of madder, lac of madder or alizarine; cochineal carmine, carmine lac; indigotine (indigo extract in a dry state), 14·00 (17·00). Colouring substances mixed with non-colouring materials, such as clay and oil, pay duty at the rate of 3r. per pound when the colouring substance does not exceed 10 per cent. of the total weight of the mixture.—*Chemist and Druggist.*



### SOME OF THE CONTEMPLATED CHANGES IN THE UNITED STATES TARIFF.

Name of Article.	Present Tariff, McKinley Bill.	Wilson Bill as it left the House of Representatives.	Wilson Bill as it is now before the United States Senate.	Remarks.
Acetate of lead, white.....	5½ c. per lb.	2½ c. per lb.	2½ c. per lb.	
"    "    br. wn.....	3½ c. "	1 c. "	1½ c. "	
Acid, acetic, sp. gr. 1.170 and under.....	1½ c. "	20 per cent.	20 per cent.	
"    "    "    "    over.....	1 c. "	20 "	20 "	
"    boracic.....	5 c. "	20 "	20 "	Commercial and pure same duty.
"    chromic.....	6 c. "	10 "	10 "	
"    cupric.....	10 c. "	20 "	20 "	Present duty about 30 per cent.
"    sulphuric.....	¾ c. "	Free	Free	
"    tannic.....	75 c. "	35 c. per lb.	35 c. per lb.	Duty under Wilson Bill, 85 per cent.
"    tartaric.....	10 c. "	20 per cent.	10 per cent.	
Alcoholic perfumery.....	\$2.00 per gall. and 50 per cent.	\$2.00 per gall. and 25 per cent.	\$2.00 per gall. and 25 per cent.	
Alum, alum cake, patent alum, sulphate alumina and alum, crystals or ground.	⅙ c. per lb.	20 "	30 "	
Alumina.....	⅙ c. "	10 "	10 "	
Ammonia, carbonate.....	1½ c. "	Free	Free	Present duty about 27 per cent.
"    muriate or sal ammoniac.....	¾ c. "	Free	Free	
"    sulphate.....	½ c. "	Free	Free	
Antimony, regulus or metal.....	¾ c. "	Free	Free	
Baryta, sulphate of, manufactured.....	\$6.72 per ton	\$3.00 per ton	25 per cent.	Ton of 2,240 lb. Sulphate unmanufactured, free.
Bay rum.....	\$1.50 proof gallon	\$1.60 proof gallon	\$1.60 proof gallon	
Blue vitriol or sulphate copper.....	2 c. per lb.	Free	Free	
Borax, crude, or borate of soda or lime.....	3 c. "	Free	Free	
"    refined.....	5 c. "	20 per cent.	20 per cent.	
Castor beans.....	50 c. per bushel	25 c. per bushel	25 c. per bushel	Bushel of 50 lb.
"    oil.....	50 c. per gallon	35 c. per gallon	35 c. per gallon	
Calomel.....	35 per cent.	25 per cent.	25 per cent.	
Camphor, refined.....	4 c. per lb.	Free	Free	
Capsicum or red pepper unground.....	2½ c. "	2½ c. per lb.	30 per cent.	
Caustic potash, in sticks or rolls.....	1 c. "	Free	Free	
Chalk, prepared, precipitated, French, and red.	1 c. "	20 per cent.	20 per cent.	
Chloral hydrate.....	50 c. "	25 "	25 "	
Chromate and bichromate potash.....	3 c. "	20 "	25 "	
Coal-tar colours or dyes. (Aniline colours).....	35 per cent.	20 "	20 "	
"    preparations, not colours or dyes.....	20 "	Free	Free	All products of coal-tar, also free.
Cobalt, oxide of.....	30 c. per lb.	Free	Free	
Cocoa butter.....	3½ c. "	3½ c. per lb.	15 per cent.	
Cod-liver oil.....	15 c. per gallon	20 per cent.	20 "	
"    oil.....	8 c. "	15 "	Free	
Copperas or sulphate of iron.....	⅓ c. per lb.	Free	Free	
Cotton-seed oil.....	10 c. per gallon	Free	Free	Gallon of 7½ lb.
Cream tartar.....	6 c. per lb.	25 per cent.	20 per cent.	
Croton oil.....	30 c. "	Free	Free	
Dextrine or British gum.....	1½ c. "	1 c. per lb.	50 per cent.	
Drugs which have been advanced in value by refining, grinding, or other process of manufacturing.	10 per cent.	Free	Free	10 per cent. proposed in Senate.
Ether, fruit, oils, or essences.....	\$2.50 per lb.	\$1.00 per lb.	\$1.60 per lb.	
"    nitrous.....	25 c. "	20 c. "	20 c. "	
"    sulphuric.....	40 c. "	35 c. "	35 c. "	

SOME OF THE CONTEMPLATED CHANGES IN THE UNITED STATES TARIFF—*continued*.

Name of Article.	Present Tariff, McKinley Bill.	Wilson Bill as it left the House of Representatives.	Wilson Bill as it is now before the United States Senate.	Remarks.
Extracts of logwood, sumac, &c.	$\frac{1}{2}$ c. per lb.	10 per cent.	10 per cent.	
" hemlock bark	1 c. "	"	"	
Flax seed	30 c. per bushel	20 c. per bushel	20 c. per bushel	Bushel of 56 lb.
Fuller's earth, Manufactured	\$1.00 per ton	\$1.00 per ton	\$1.00 per ton	Unmanufactured free.
Glauber salts. (Sulphate soda)	\$1.25 "	Free	Free	
Glycerine, crude, not purified	1 $\frac{1}{2}$ c. per lb.	1 c. per lb.	1 c. per lb.	
" refined	1 $\frac{1}{2}$ c. "	3 c. "	3 c. "	
Gold leaf. Packages of 500 leaves	\$2.00 per package	34 per cent.	30 per cent.	
Honey	20 c. per gall.	10 c. per gallon	20 "	
Hops	15 c. per lb.	8 c. per lb.	20 "	
Indigo, extract or paste	$\frac{1}{2}$ c. "	Free	Free	
Indigo, carmined	10 c. per lb.	Free	Free	
Ink, printing ink and ink powders	30 per cent.	20 per cent.	20 per cent.	
Iodide of potash	50 c. per lb.	25 c. per lb.	10 "	
Iodine, resublimed	30 c. "	Free	Free	
Iodoform	\$1.50 "	\$1.00 per lb.	25 per cent.	
Licorice, extracts of	5 $\frac{1}{2}$ c. "	5 c. "	1 c. per lb.	
Linseed oil, raw or boiled	32 c. per gall.	15 c. per gall.	15 c. per gall.	Per gallon of 7 $\frac{1}{2}$ lb.
Litharge	3 c. per lb.	1 $\frac{1}{2}$ c. per lb.	1 $\frac{1}{2}$ c. per lb.	
Magnesia, carbonate	4 c. "	3 c. "	30 per cent.	
" calcined	8 c. "	7 c. "	30 "	
" sulphate	1 c. "	Free	Free	
Mica	35 per cent.	Free	Free	
Morphine and its salts	50 c. per oz.	75 c. per oz.	75 c. per oz.	Present rate about 47 per cent.
Mustard, ground or preserved	10 c. per lb.	10 c. per lb.	25 per cent.	Present duty, 38 per cent.
Olive oil, fit for salad purposes	35 c. per gall.	35 c. per gall.	25 "	
Opium, aqueous extract, tincture and other liquid preparations.	40 per cent.	25 per cent.	20 "	
Opium, containing less than 9 per cent. morphine	\$12.00 per lb.	\$6.00 per lb.	\$6.00 per lb.	
" prepared, for smoking	\$12.00 "	\$6.00 per lb.	\$7.00 "	
" crude, containing 9 per cent. and over morphine.	Free	\$1.00 "	\$1.00 "	
Peppermint oil	80 c. per lb.	25 per cent.	20 per cent.	
Phosphorus	20 c. "	25 "	25 "	
Prussiate potash, red	10 c. "	20 per cent.	20 per cent.	
" " yellow	5 c. "	20 "	20 "	
Quicksilver	10 c. "	Free	Free	
Red lead	3 c. "	1 $\frac{1}{2}$ c. per lb.	1 $\frac{1}{2}$ c. per lb.	
Rochelle salts	3 c. "	10 per cent.	10 per cent.	
Sage	3 c. "	1 c. per lb.	30 "	Present duty 170 per cent.
Saltpetre, refined	1 c. "	$\frac{1}{2}$ c. "	10 "	
Santonine	\$2.50 per lb.	\$1.00 per lb.	\$1.00 per lb.	
Silver leaf, per package of 500 leaves	75 c. per package	15 per cent.	30 per cent.	
Soap, Castile	1 $\frac{1}{2}$ c. per lb.	20 "	20 "	
" common	20 per cent.	10 "	10 "	
" fancy and toilet	15 c. per lb.	25 "	30 "	
Soda, ash	$\frac{1}{2}$ c. "	$\frac{1}{2}$ c. per lb.	20 "	
" bicarbonate	1 c. "	$\frac{1}{2}$ c. "	30 "	
" bichromate and chromate	3 c. "	20 per cent.	25 "	

SOME OF THE CONTEMPLATED CHANGES IN THE UNITED STATES TARIFF—*continued.*

Name of Article.	Present Tariff, McKinley Bill.	Wilson Bill as it left the House of Representatives.	Wilson Bill as it is now before the United States Senate.	Remarks.
Soda, caustic.....	1 c. per lb.	$\frac{1}{2}$ c. per lb.	25 per cent.	
" crystals or sal soda.....	$\frac{1}{2}$ c. "	$\frac{1}{2}$ c. "	20 "	
" silicate .....	$\frac{1}{2}$ c. "	$\frac{1}{2}$ c. "	20 "	
Spices, ground or powdered.....	4 c. "	3 c. "	30 "	
Sponges .....	20 per cent.	10 per cent.	10 "	
Strychnine, and its salts .....	40 c. per oz.	30 "	30 "	
Sugar of milk .....	8 c. per lb.	20 "	5 c. per lb.	Present duty 65 per cent.
Sulphur, refined.....	\$8.00 per ton	Free	Free	
" sublimed, or flowers of.....	\$10.00 "	20 per cent.	20 per cent.	
Sumac, ground.....	$\frac{1}{16}$ c. per lb.	10 "	10 "	
Toilet preparations.—Cosmetics, dentifrices, pomades, &c.	50 per cent.	40 "	30 "	
Turkey-red oil.....	40 c. per gallon	30 "	30 "	
Ultramarine blue, dry, pulp, or mixed with water.	$\frac{1}{2}$ c. per lb.	20 "	20 "	
Varnishes, including gold size and Japan.....	35 per cent.	25 "	25 "	
Vermilion red, and colours containing quicksilver	12 c. per lb.	20 "	20 "	
White lead, dry or ground, in oil.....	3 c. "	$1\frac{1}{2}$ c. per lb.	$1\frac{1}{2}$ c. per lb.	
Whiting and Paris white, dry.....	$\frac{1}{2}$ c. "	25 per cent.	35 per cent.	
Zinc, oxide, dry or ground, in oil .....	$1\frac{1}{2}$ and $1\frac{1}{4}$ c. per lb.	20 "	25 "	

*Schoellkopf, Hartford, and MacLagan, Limited.*

April 27th, 1894.

## STATISTICS.

## MINERAL PRODUCTION OF THE UNITED KINGDOM.

The official returns of mineral production in Great Britain and Ireland in 1893 have just been made public, and we give below a table showing the output for the year, compared with that for 1892 and 1891. The statistics covering a long series of years will be found in the new volume of the "Mineral Industry." The figures given below are in long tons (2,240 lb.) :—

*Mineral Production of the United Kingdom.*

Description of Mineral.	1891.	1892.	1893.
	Tons.	Tons.	Tons.
Alum clay (banxite).....	10,763	7,322	8,740
Arsenic.....	6,049	5,114	5,976
Arsenical pyrites .....	5,995	4,497	3,036
Barytes.....	26,876	21,217	22,343
Clays (excepting ordinary clay).....	827,970	891,519	878,965
Coal.....	185,479,126	181,786,871	164,325,795
Copper ore.....	8,836	5,995	5,346
Copper precipitate .....	322	270	230
Fireclay.....	2,334,065	2,212,333	2,186,243
Fluorspar.....	141	171	215
Gold ore .....	14,117	9,990	4,489
Gypsum.....	151,768	147,540	143,686
Iron ore.....	12,777,689	11,312,975	11,203,476

*Mineral Production of the United Kingdom—cont.*

Description of Mineral.	1891.	1892.	1893.
	Tons.	Tons.	Tons.
Iron pyrites.....	15,163	13,997	15,837
Lead ore.....	43,859	40,024	40,808
Manganese ore.....	9,476	6,078	1,336
Ochre, maber, &c. ....	13,502	12,131	10,534
Oil shale.....	2,361,119	2,089,937	1,953,520
Phosphate of lime .....	10,000	12,200	3,300
Salt.....	2,043,571	1,956,524	1,921,029
Slates and slabs.....	415,029	418,241	438,093
Strontian sulphate .....	8,061	5,066	5,812
Tin ore .....	14,488	14,329	13,689
Wollfram .....	138	125	22
Zinc ore .....	22,216	26,880	23,754

With a few exceptions the production shows a slight decrease in 1893. The great falling off in coal production was due to the miners' strike, and some comment will be found elsewhere.

The total number of mines reported in 1893 was 4,208, of which 3,383 mines, employing 683,008 persons, were classed under the "Coal Mines Regulation Act," and 825 mines, employing 35,739 persons, were under the "Metaliferous Mines Act." The total number of persons employed in mining was thus 718,747, of whom 5,760 were women, of whom 4,725 were at coal mines and 1,035 at

other mines. The mining employees were divided as follows:—

	Underground.	On Surface.	Total.
Males.....	570,978	142,000	712,978
Females.....	..	5,700	5,700
Totals.....	570,978	147,700	718,747
Totals, 1892.....	571,840	149,968	721,808
Totals, 1891.....	559,189	148,222	707,411

The total number of deaths resulting from accidents in and about mines last year was as follows:—

	Coal Mines.	Other.	Total.
Accidents underground.....	940	62	1,002
Accidents on surface.....	120	3	123
Totals.....	1,060	65	1,125
Totals, 1892.....	1,016	52	1,068
Totals, 1891.....	1,005	51	1,056

In the accidents underground, 160 deaths were caused by explosions of firedamp, 435 by falls of rock, and 115 were in shafts.—*Engineering and Mining Journal*.

#### MINERAL PRODUCTION OF CANADA, 1893.

The following summary of the mineral production of Canada during 1893 has just been issued by Mr. Elfric Drew Ingall, in charge of the Division of Mineral Industries and Mines. The figures are still held subject to revision:—

Product.	Quantity. <sup>(1)</sup>	Value.
<i>Metallic.</i>		
Copper (?).....	Lb. 8,102,856	\$75,864
Gold (?).....	Ozs. 51,609	927,244
Iron ore (?).....	Tons 124,702	298,918
Lead (?).....	Lb. 2,435,923	80,296
Nickel (?).....	.. 3,992,982	2,076,351
Platinum.....	Ozs. ..	1,800
Silver (?).....	.. 414,975	321,423
Zinc (?).....	Lb. 11,763	470
Total metallic.....	..	4,582,106
<i>Non-Metallic.</i>		
Asbestos.....	Tons 6,473	313,806
Coal.....	.. 3,719,17	8,422,259
Coke (?).....	.. 161,730	61,078
Felspar.....	.. 575	4,525

<sup>(1)</sup> Quantity marketed, except when otherwise specified. Tons are of 2,000 lb.

<sup>(2)</sup> Copper contents of ore, matte, &c., at 10¢ cents per lb.

<sup>(3)</sup> Nova Scotia and Ontario gold at 19'50 dols., Quebec at 18 dols., and British Columbia and Yukon District at 17 dols. per oz.

<sup>(4)</sup> Of this quantity 124,053 tons were converted into pig iron, producing 55,917 tons, valued at the furnaces at 790,283 dols.

<sup>(5)</sup> Lead contents of ores at 3'7 cents per lb.

<sup>(6)</sup> Nickel contents of ore, matte, &c., at 52 cents per lb. This represents the final market value of the nickel. In the matte its spot value would be much less, being quoted at 13 cents only in this condition, which would bring the figures for value down to 519,088 dols.

<sup>(7)</sup> Silver contents of ore at 76'9 cents per oz.

<sup>(8)</sup> Zinc contents of ore at 4 cents per lb.

<sup>(9)</sup> Oven coke, all the production of Nova Scotia.

#### MINERAL PRODUCTION OF CANADA, 1893—cont.

Product.	Quantity.	Value.
Fireclay.....	Tons 540	\$68,700
Grindstones.....	.. 4,000	38,570
Gypsum.....	.. 197,568	136,150
Limestone for flux.....	.. 27,737	27,510
Manganese.....	.. 228	14,458
Mica (?).....	..	6,9622
Mineral water.....	Galls. 725,593	168,317
Moulding sand.....	Tons ..	1,000
Natural gas (?).....	..	366,233
Ochres.....	Tons 1,070	17,710
Petroleum (?).....	Bbls. 708,403	84,334
Phosphate (?).....	Tons 8,198	707,42
Precious stones.....	..	150
Pyrites.....	Tons 58,542	175,626
Salt.....	.. 62,324	165,926
Soapstone.....	.. 717	1,920
<i>Structural Materials.</i>		
Bricks, M.....	205,000	1,275,000
Building stone.....	Cub. yds. 220,000	610,000
Cement.....	Bbls. 134,745	201,786
Flaestones.....	Sq. ft. 40,800	3,487
Granite.....	Tons 22,521	94,393
Lime.....	Bush. 2,400,000	410,000
Marble.....	Tons 500	5,100
Pottery.....	..	189,107
Roofing cement.....	Tons 951	5,441
Sands and gravels (exports).....	.. 329,116	121,795
Sewer pipe.....	..	194,162
Slate.....	Tons 7,112	90,825
Terra-cotta (?).....	..	55,704
Tiles, M.....	16,000	191,000
Total non-metallic.....	..	14,394,294
Total metallic.....	..	4,582,106
Estimated value of mineral products not returned, largely structural materials.....	..	276,518
1893. Total.....	..	19,250,000
1892 ..	..	19,500,000
1891 ..	..	21,500,000
1890 ..	..	18,000,000
1889 ..	..	14,500,000
1888 ..	..	13,500,000
1887 ..	..	12,500,000
1886 ..	..	12,000,000

\* Estimated.

<sup>(1)</sup> See note (1) in preceding column.

<sup>(2)</sup> Exports, plus quantities sold to Canadian electrical works and stove foundries.

<sup>(3)</sup> Gross amount received through sale of gas.

<sup>(4)</sup> Calculated from inspection returns at 100 galls. crude to 38 refined, and computed at 1'04 dols. per bbl. of 55 imp. galls. The barrel of refined oil is assumed to contain 42 imp. galls.

<sup>(5)</sup> Exports, plus quantities sold to Canadian superphosphate works.

<sup>(6)</sup> Includes porous fire-proof terra-cotta.

—*Ibid.*

## THE MINERAL PRODUCTION OF GERMANY.

The following figures for the mineral production of Germany, including Luxemburg, for the year 1893, are given by the "Kohlen Zeitung," and have been prepared from the reports made by the various mines and works. The production is given in metric tons; the values we have reduced to dollars:—

	Quantities.		Values.	
	1892.	1893.	1892.	1893.
Coal.....	Met.Tons.	Met.Tons.	Dols.	Dols.
	71,372,193	73,908,399	131,714,794	124,616,605
Brown coal (lignite)...	21,171,857	21,567,218	11,626,475	13,759,759
Graphite.....	4,036	3,149	63,210	52,000
Asphalt.....	53,279	47,238	14,4713	89,215
Petroleum.....	14,527	13,971	219,941	195,733
Rock salt.....	662,577	669,042	708,046	736,929
Kainit.....	548,445	661,986	1,955,717	2,307,576
Other potash salts.....	892,639	861,162	2,532,169	2,562,060
Glauber salts.....	19,297	8,818	22,356	17,657
Boracite.....	179	181	13,614	11,289
Iron ore.....	11,539,013	11,157,491	19,319,713	9,950,111
Zinc ores.....	809,237	788,394	5,305,324	3,574,163
Lead ores.....	163,372	168,411	3,671,881	3,336,012
Copper ores.....	567,738	584,875	5,128,346	4,539,683
Gold and silver ores...	17,536	18,578	910,509	773,931
Tin ores.....	63	69	23,091	18,888
Cobalt, nickel, and bismuth ores.....	3,185	4,379	198,298	186,410
Uranium and wolfram ores.....	48	43	11,557	10,778
Antimony and quick-silver ores.....	..	16	..	100
Manganese ores.....	32,961	40,788	126,024	122,988
Arsenic ores.....	2,146	2,756	19,491	25,363
Pyrites.....	115,213	121,334	215,887	219,427
Other sulphur and alum. ores.....	2,973	791	2,916	1,250

The fuel production, it will be noted, showed only a moderate increase last year. The important minerals, iron, zinc, and lead ores, were substantially the same in both years. Prices generally showed a slight decline; coal, for instance, was valued at 1.68 dols. in 1893, against 1.84 dols. in 1892.

The products of the German salt works, an important item in the mineral industry of that country, were as follows:—

	Quantities.		Values.	
	1892.	1893.	1892.	1893.
Salt.....	Met.Tons.	Met.Tons.	Dols.	Dols.
	504,687	505,923	3,469,805	3,494,194
Potassium chloride....	123,362	137,216	4,109,559	4,396,285
Magnesium chloride....	14,386	12,764	59,578	44,443
Glauber salts.....	74,998	75,965	5,627,888	496,751
Potassium sulphate....	22,968	23,555	931,861	956,439
Double sulphate of potassium and magnesium.....	11,263	14,199	118,152	250,792
Magnesium sulphate....	23,879	27,518	84,008	79,240
Alumina sulphates.....	29,218	17,859	409,735	345,166
Sum.....	7,929	3,412	97,444	87,556

The chemical manufacture, as well as the mining industry, appears to have last year just about held its own, the average prices showing little variation.—*Ibid.*

## EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

## SWEDISH BEET SUGAR INDUSTRY.

## U.S. Consular Reports, 1894, 44, 704.

The sugar of Sweden is made from beets, and the following table shows the number of beet sugar factories in the Kingdom and their output for the 11 years 1882—92:—

Year.	Number of Factories.	Raw Sugar produced.	Year.	Number of Factories.	Raw Sugar produced.
		Tons.			Tons.
1882	1	1,472	1888	4	9,358
1883	2	1,367	1889	4	16,822
1884	2	3,655	1890	6	16,397
1885	3	3,940	1891	8	27,241
1886	3	5,958	1892	..	39,781
1887	3	5,953			

This sugar is all the product of beets grown in Sweden, and the increase in production is simply enormous. The amount of sugar consumed in Sweden is given in the following table, which shows the number of tons used for each year from 1883 to 1891. It must be borne in mind that the figures in the following table represent refined sugar, whereas those in the foregoing are for raw sugar. For purposes of comparison, 100 tons of raw sugar may be taken as equal to 99 tons of refined:—

Year.	Refined Sugar consumed.	Year.	Refined Sugar consumed.
	Tons.		Tons.
1883	38,294	1888	44,589
1884	49,753	1889	48,163
1885	41,253	1890	51,343
1886	11,392	1891	54,336
1887	42,651		

It will be seen that we have here to deal with an increasing consumption, as well as with an increasing output. Reckoning that the consumption will continue to increase in the same ratio as heretofore, we find that in the year 1896 Sweden will make use of 64,000 tons of refined sugar.

A careful estimate of the output of the Swedish factories, adding to the regularly increasing product of the 10 factories now in operation the probable product of four new factories just built or being built, shows that in 1896 there will be produced in Sweden 76,000 tons of raw sugar, equal to 68,400 tons of refined. Hence in 1896—the year after next—Sweden will probably produce not only sufficient sugar for its own inhabitants, but have a surplus for export.

It is true that the Swedish sugar beets are all raised in the southern portion of the country, and nearly all in a comparatively small section in the extreme south west. It is also true that the Gulf Stream, conveying the waters of the tropics to the north-west of Europe, gives to the Scandinavian peninsula a climate milder than that enjoyed by any other northern land. Still, after all allowances have been made, Sweden is an extreme northern country, with long winters, short summers, and in general a hard

soil. That in such a country the production of sugar should have advanced with such rapid strides and reached such grand proportions, constitutes one of the marvels of agriculture on our globe, and might well serve as an example and an incentive to the people of the United States, who live in a more fertile land and under milder skies.

### GENERAL TRADE NOTES.

#### THE AMERICAN CHEMICAL INDUSTRY.

##### *The Relation of Labour to the Cost of Production.*

The proportion of the cost of labour to the total cost of production in any branch of manufacturing industry is necessarily a serious consideration to manufacturers, especially where labour is an important factor in the industry. It is obvious that the supremacy in any given line of production must eventually lie with the country where, other conditions being equal, the cost of production is less than that of competing countries.

Germany is an important factor in this respect, so far as England is concerned; but the present article is not concerned with that country, but rather with the United States, where the chemical industry has of late years made steady progress.

It may be pointed out, by way of introduction, that the United States purchased, in 1893, chemicals, drugs, dyes, and medicines, to the value of 13,250,912 dols., of which 1,915,873 dols. was for coal-tar colours and dyes; 1,171,878 dols. was for caustic soda; 3,803,478 dols. for soda ash; and 4,401,000 dols. for miscellaneous chemicals. This was dutiable. The States also imported, during the same period, 1,444,620 dols. worth of chemicals duty free, among which were alizarine, argol, various kinds of barks, cochineal, &c. The export amounted, in 1893, to about half the total value of the imports of chemicals, namely to 7,001,000 dols. The value of the British exports of chemicals to America, in 1893, was 1,297,000*l.* (6,485,000 dols.) for alkali alone, or about 50 per cent. of the total value exported to all countries. Of bleaching materials, the States took 337,762*l.*, out of a total of 522,625*l.* We thus see the importance of the connection between the two countries, and any information bearing on the cost of production of chemicals in America must be of importance to English manufacturers.

The relation of wages to the cost of production involves a consideration of two elements—Labour and Capital, and two lines of inquiry may be pursued. First, the determination of the share of product which falls to labour in distinction from the share retained by capital. Secondly, the relation which labour cost bears to the total cost of production, as shown by the proportion which wages bear to the other elements of cost. The first inquiry would involve the question of profits, and would take as its basis the selling price of the product, separating the price into the various factors which compose it, such, for instance, as wages (labour's share) and profits and interest (capital's share). The second phase of the subject, which deals only with the actual cost to the manufacturer, eliminates profits, and determines what part of that cost is due to labour, represented by wages, and what to raw material and other necessary expenses. The first inquiry deals with the distribution of wealth, and the second with its production. The question to be dealt with next is, what relation does the cost of labour bear to the cost of the product at the actual time of completion? For instance, the proportions of cost in making 1,000 gallons of *disinfectants* are as follows:—Wages from 4.76 to 9.27 per cent. of the total, and raw materials from 95.24 to 90.73 per cent. respectively. In the manufacture of one ton of *sulphuric acid* the proportions are: Wages from 15 to 19 per cent., raw materials from 53 to 66.50 per cent., and miscellaneous expenses (such as rent, taxes, insurance, &c.) from 32 to 14 per cent. As regards laundry blue, to produce 24,092 gross of this article, costs from 13 to 22 per cent. for wages, about 59 per cent. for materials, and the balance for rent, &c. The labour cost of producing a ton of *guano* is comparatively small, being only 8.30 per cent. of the total outlay. The raw material costs 81.50 per cent., while

about 10.20 per cent. is allowed for general expenses. For the same quantity of *fertilisers*, the wages cost is 20 per cent., and materials 80 per cent. In the manufacture of *phosphates*, the labour cost per ton is a little higher, namely, 14.29 per cent. and materials 85.71 per cent. *Oils and illuminating fluids* cost, per barrel, nearly 7 per cent. for labour, about 67.50 per cent. for raw materials, and the balance goes for miscellaneous expenses. An experiment in making 3,185 tons of *copperas* shows that the labour cost ranges from 22.94 to 33.91 per cent., raw materials from 41.66 to 51.32 per cent., and general charges from 35.43 to 11.77. In making *mixed paints* (per gallon) labour costs about 10 per cent. of the total, and materials 90 per cent., while the same proportions apply to the manufacture of green paints.

In 1893, an investigation was made by the Statistical Bureau of the State of Connecticut, U.S.A., as regards the cost of manufacturing in that district, and particulars were obtained from 11 firms making *chemical products*, the aggregate capital being 438,000 dols.; the total value of goods made in one year amounting to 638,000 dols., the value of stock and materials being 359,300 dols.; the cost of manufacture (less rent, taxes, &c.), 156,550 dols.; rent and taxes, 6,988 dols.; superintendence, as distinct from "wages," 32,950 dols.; wages, 69,337 dols.; gross profits, 122,150 dols.; net profits, 115,161 dols., or a percentage of net profits of capital amounting to 26.29. Reduced to percentages these figures are:—Percentage of wages to the cost of goods made, 13.26; superintendence, 6.30; stock and materials, 68.72; rent, taxes, &c., 1.34; general expenses of cost of goods made, 10.38.

The following table shows the weekly rate of wages paid by nine firms to operatives in the State of Massachusetts, in the industry of making chemical preparations:—

Rates of Wages.	Employees.	
	Males.	Females.
Under 20 <i>s.</i> .....	2	1
20 <i>s.</i> but under 24 <i>s.</i> .....	2	..
24 <i>s.</i> .. 28 <i>s.</i> .....	4	..
28 <i>s.</i> .. 32 <i>s.</i> .....	3	2
32 <i>s.</i> .. 36 <i>s.</i> .....	7	..
36 <i>s.</i> .. 40 <i>s.</i> .....	121	..
40 <i>s.</i> .. 48 <i>s.</i> .....	54	1
48 <i>s.</i> .. 60 <i>s.</i> .....	83	..
60 <i>s.</i> .. 80 <i>s.</i> .....	21	..
80 <i>s.</i> and over .....	4	..
Totals .....	301	3

Dyestuffs (Seven Firms).	Males.		Females.
Under 20 <i>s.</i> .....	3	..	..
20 <i>s.</i> but under 24 <i>s.</i> .....	2	5	..
24 <i>s.</i> .. 28 <i>s.</i> .....	3	6	..
28 <i>s.</i> .. 32 <i>s.</i> .....	20	2	..
32 <i>s.</i> .. 36 <i>s.</i> .....	6	1	..
36 <i>s.</i> .. 40 <i>s.</i> .....	74	..	..
40 <i>s.</i> .. 48 <i>s.</i> .....	85	..	..
48 <i>s.</i> .. 60 <i>s.</i> .....	24	..	..
60 <i>s.</i> .. 80 <i>s.</i> .....	10	..	..
80 <i>s.</i> and over .....	1	..	..
Totals .....	227	14	..

P	Seven Firms.	Males.	Females.
1	12	..	..
2	10	..	..
3	28	2	..
4	32	2	..
5	166	21	..
6	108	41	..
7	188	9	..
8	648	13	..
9	800	3	..
10	an	1	..
Totals		92	..

In printworks, dyeworks, and bleacheries, in which 1,429 males were employed, 50 per cent. received 28s. to 32s. per week; 10 per cent. earned 1*l.*; 10 per cent., 24s.; 10 per cent., 36s., and the remainder sums varying from 2*l.* to 4*l.* In the chemical industries as a whole, 75 per cent. of the people earn about 2*l.* 5s. a week. Women rarely get beyond 30s., except in special cases of skill.

Mr. Charles Booth, when examining into the wages paid to persons employed in the chemical industry in England, found that out of 663 persons, two-thirds earned from 20s. to 40s. a week, so that it may be concluded that wages on both sides of the Atlantic are very similar. Mr. Booth found that foremen earn 40s. to 50s. a week, and "tending hands" make from 30s. and upwards, being paid 6*d.* to 7*d.* per hour. Chemical labourers are paid 5*d.* to 6*d.* per hour, or about 25s. for an ordinary week; but they, as well as the "tending hands," make a good deal of overtime. White lead workers earn 7s. 6*d.* a day, but the men do not work at this more than three days a week. Other persons employed get 4s. 6*d.* a day or 19s. to 22s. a week for such as have regular work. The cost of living in America, outside New York City, would be about 10 per cent. higher than in England, and this includes cost of clothing. Comparing New York City with London, the former would be about 20 per cent. dearer. Operatives work harder in America than in England, and there is rather more "driving" by the "bosses" there than in England. — *Chemical Trade Journal*.

#### THE IMPORTATION OF PATENT MEDICINES INTO TURKEY.

The following observations referring to the prohibition of the import of patent medicines into Turkey are contained in the report of the British Chamber of Commerce at Constantinople for the year 1892—93:—

Your Board is unfortunately unable to report that the prohibition referred to last year has been removed.

Further correspondence has been exchanged with Her Majesty's Embassy, and your Board is aware that Her Majesty's officials have striven to induce the Turkish Government to remove this arbitrary and prejudicial prohibition, but so far with no successful results.

On the 17th June, Sir Arthur Nicolson informed the Chamber that the Embassy had quite recently addressed two further notes to the Sublime Porte in regard to the questions of patent medicines and the banderolle tax, urging in the first case that some reply should be sent to the previous communications on the subject, and in the second requesting that the duty should be abolished as being distinctly contrary to treaty stipulations.

There is a curious feature in connection with this prohibition of patent medicines, which is the absence of this restriction at Smyrna, where all kinds of pharmaceutical

preparations are daily imported without hindrance, and it is no doubt the same in other Turkish ports.

#### NATIONALISATION OF THE GERMAN POTASH AND MAGNESITE DEPOSITS.

Probably animated thereto by the recent discoveries of new potash and magnesite mines in central Germany, the German Government have brought in a Bill for the nationalisation of all potash and magnesite deposits in the country. The output of potash and magnesia salts in Germany in 1892 was 1,361,161 tons, worth over 900,000*l.*—*Chemist and Druggist*.

#### THE PORCELAIN INDUSTRY AT LIMOGES.

According to the *Monde Economique* for the 14th April, the porcelain industry at Limoges is in a depressed condition. At the beginning of this year there were only 139 furnaces against 270 last year, with a diminished production of more than 40,000*l.*

#### CAUCASIAN MANGANESE TRADE.

The Belgian Consul-General at Odessa, in a recent report, supplies the following extract from an article which has appeared in the *Kharkoff Journal des Mines*:—

The owners of the manganese mines of Tchiatoura (Caucasus), have made a contract with the firm of G. Pincher, of Hamburg, for developing the working of manganese.

By the terms of this contract all the ore extracted is taken by the Pincher firm, which has the monopoly for sale to the European and American steelworks.

The quantity which will be annually delivered for consumption is estimated at 1,000,000 pounds at the least.

#### GERMAN IRON-MAKING AND ITS RESIDUAL PRODUCTS.

The advance of Germany as a great iron-producing country, and the strength of her competition in some of the markets of the world which British manufacturers have hitherto regarded as special fields of enterprise have awakened surprise, if nothing more. Twenty years ago, for instance, Germany's total make of iron was 885,000 tons, and of steel 125,000 tons, together 1,011,000 tons; while last year the total was 3,878,000 tons. Of this total one-third is bar and section iron; blooms, billets, and ingots making up 800,000 tons; plates, 425,100 tons; and rails, 891,400 tons. In recent years there have been cases of German iron and steel manufacturers quoting to British clients a lower price than home makers. It is assumed by some that the product is inferior, but where specific tests have to be met this is scarcely tenable. Labour may be slightly cheaper, but even this is doubtful, and certainly wages, especially to coal miners, are on the increase, so that fuel is dearer.

The growing importance of the iron industry in Germany is said by him to be due to two fundamental facts—the introduction of the basic process, which enables cheap and abundant native ores to be used, and the application of chemical skill to the recovery and utilisation of the by-products of coke manufacture. The latter point is of special interest, for, while there is a growing tendency to construct ammonia recovery works for the recovery of the products from the blast furnaces, little has been done in the case of coke making. The difficulty formerly urged was the possibility of injuring the quality of the coke. There are in all about 3,000 coke ovens which save the subsidiary products, and half of these are of the Otto and Otto-Hoffmann type. The oven more largely adopted in Belgium—the Seneet-Solvay—is cheaper to construct, but requires a special mixture of fat and lean coal, which is not always easy of arrangement, and while the coke made is of high and uniform quality, it gives less ammonia, tar, and gas than the German oven. The product from good coal containing from 15 to 17 per cent. of water is about 76 per cent. of coke, 1·15 to 1·25 per cent. of sulphate of ammonia, and from 2·5 to 4 per cent. of tar. Batteries of 60 ovens



in three districts gave the following results in pounds per ton of coal:—

	Coke.	Tar.	Sulphate of Ammonia.
Ruhr .....	1,672	60.5	25.3
Silesia .....	1,436	93.5	26.4
Saar .....	1,749	91.4	18.7

These 180 ovens produce per annum 139,800 tons of coke, Ruhr making 51,300 tons, Silesia 48,000 tons, and Saar 40,500 tons. The aggregate sulphate of ammonia recovered weighed 2,112 tons and the tar 7,260 tons. Moreover, the waste heat, after the recovery of these by-products, was sufficient to work the ovens and give a large surplus for other purposes. This surplus in the case of Ruhr was 12,800 cubic feet per oven per day, Silesia 16,000, and Saar 12,800 cubic feet. In other words, a battery of ovens, in addition to providing the coke and the residual products, self-heating, and gives off per day heat which is equivalent to 45,600 lb. to 57,000 lb. of coal, sufficient to run machinery indicating from 1,200 to 1,600 indicated horse power. Of course, a special plant is necessary to work the process, so that in making comparison with the ordinary coking establishment, allowance must be made for this extra first cost.

The approximate gross value of the process is easily obtained. Taking the case of Silesia, we find that in producing 48,000 tons of coke in the ovens, 3,000 tons of tar are recovered each year, which at 2s. per cwt. yields 6,000*l.*, and 840 tons of sulphate of ammonia, at 11s. per cwt. equals 9,240*l.*—together, 15,240*l.* To this amount must be added the fuel saved. In Silesia the total production of gas is 36,800 cubic feet per oven per day, and after consuming 20,800 cubic feet in roasting the coal in the production of the coke, a surplus of 16,000 cubic feet remains for other purposes, equal, as we have already indicated, to 57,000 lb. of coal for the 60 ovens, sufficient for 1,600 i.h.p. during 18 hours per day at 2 lb. per i.h.p. per hour. The value of this coal—about 9,000 tons per annum—should be added to the 15,240*l.* received for tar and sulphate of ammonia, in determining the gross value of the residual products. This does not include the value of the heat utilised in the oven, which is equivalent to an additional 12,000 tons per annum. Sixty ovens in Westphalia cost in construction 34,500*l.*, or 576*l.* each, and the tar and sulphate of ammonia produced in one year's working provided a revenue of 8,375*l.*, or about 25 per cent. on the capital invested in the plant.

The other instances given might also be worked out, but it is probably sufficient to show that Germany can afford to credit actual cost of production with a large sum, the result of the recovery and sale of by-products. Of course this affects the selling price of their iron and steel, and offers at least a partial explanation for the low quotations made by German makers in competition with British manufacturers. The influence on the price of tar and ammonia of the 3,000 ovens in use in Germany and Austria is pronounced. In 1883 sulphate of ammonia was worth 16s. per cwt., but now sells at 11s., and tar has dropped in price from 3s. to 2s. per cwt. The decline in prices is not alarming, however, as in Westphalia four years' revenue suffices to pay the plant.—*Engineering and Mining Journal*.

#### THE NITRE QUESTION.

H. Polakowsky. *Chem. Ind.* 17, 86—88.

In this paper German capitalists are encouraged to participate in the forthcoming great sale by the Chilean Government of the nitre deposits in Northern and Central Tarapaca, and thus to prevent an English monopoly of the nitre market, to the detriment of the German consumption, which latter amounts to one-third of the world's consumption! About three-fourths of the deposits already sold by the Government are in English hands; the property now on sale embraces one-fourth of the nitre deposits still owned by the State in the province of Tarapaca.

The law enacting the sale of these deposits contains the following regulations (*Diario Oficial*, November 29, 1893, Law 127): Sect. 1 authorises the sale of 37 "oficinas" (nitre deposits with corresponding buildings and apparatus) within five years. The Government having undertaken to redeem the whole of the paper money by June 30, 1896, and the proceeds from the sale of these deposits being intended for such redemption, it is likely that the sales will be proceeded with shortly. Sect. 2 enacts the sale of certain specified deposits without "oficinas." Sect. 3. The sale is advertised six months beforehand in the *Diario Oficial* and three months beforehand in the principal newspaper in London, Paris, Berlin, and New York. Sect. 4. The appraised value to be the lowest bid. Sect. 5. Payments to be rendered in pounds sterling or in good acceptances in London (30 days). Twenty per cent. of the purchase money payable at once, 30 per cent. at the end of 1894, and 50 per cent. at the end of 1895. Sect. 6. A guarantee amounting to 40 per cent. of the appraised value has to be given. On payment of the first two instalments the guarantee is reduced to 20 per cent. Sect. 7. The deposits are sold according to the plans of the "Delegación Fiscal de Salitras." Sect. 8. The State is responsible for the claims of third parties. Sect. 9. In case of boundary disputes the buyers to have the right of calling in the decision of the court. Sect. 10. The proceeds from the sales, up to 1,500,000*l.*, to remain in the keeping of the mint. Sect. 11. The suspension of the law of 26th January 1893 referring to the sale of nitre deposits is enacted.

The property on sale consists of 57 deposits provided with "oficinas" (the latter are out of use since 1879 and will be next to valueless) and of 24 deposits without "oficinas." The first sale takes place on June 15, 1894, when one-third of the deposits, situated in the northern part of Tarapaca, will be put up. The appraised value of this lot amounts to 700,000*l.* out of a total of 1,500,000*l.*

—H. A.

#### EXPERT OPINIONS FROM THE PAPER TESTING DEPARTMENT OF THE BERLIN EXPERIMENTAL INSTITUTES.

*Mitt. Königl. techn. Versuchs.* 11 '93, 248—256.

It frequently happens that the tribunals apply to the Institute for expert opinion on matters connected with paper. Such opinion is required for the purpose either of settling disputed questions between litigants, or of supplying some link in the chain of evidence in cases where punishment is inflicted. Seven typical cases are cited and the following conclusions are drawn:—No careful manufacturer should accept an order to be executed "exactly according to sample." In settling the conditions of delivery, it would be better to rely less upon the sample than upon a clear statement of the nature and strength of the raw materials to be employed. Above all, such general definitions as "extra strong," "durable," "extra hard sized," &c., should be avoided. Parties are also recommended before going to law to obtain the opinion of an expert on the matters in dispute, as it seldom happens that the judge himself can arrive at a conclusion without reference to expert opinion. The question is finally asked: "Would it not be better to avoid altogether the tedious and costly method of settling trade disputes by litigation, and rather to submit them at once to a tribunal of scientifically and practically trained experts?"—E. G. P. T.

#### PURCHASING SILVER, GOLD, AND LEAD ORES.

H. Van Furman. *School of Mines Quarterly*, 15, 1893, 1—7.

Competition between the smelters of Denver, Pueblo, and Salt Lake City, the growing scarcity of desirable flaxing ores, and (late) the declining price of silver, have led to a complex system of valuation, which takes into account, besides the assay value of the ore, its chemical composition, the assumed losses in treatment, the cost of roasting and of smelting (including cost of fluxes, fuel, and labour), the mechanical condition of the ore, the particular desirability of the lot, and the market values of the metals at the time of purchase. The assay-value of the gold, silver, and lead

is always determined by fire assay. Copper when present is determined by volumetric or electrolytic methods. The necessity for determining the silica, iron, &c., is judged of according to the apparent mineralogical composition of the ore, and charges or allowances are made on a sliding scale based upon an equality of silica and iron—the so-called neutral basis. Silica is charged or allowed for at the rate of 15 cents per unit above or below the iron present. Manganese is reckoned as iron. 6 cents per unit is allowed for all fine magnesia, and baryta; zinc below 8 per cent. is not charged for; all above that is charged 50 cents per unit. Special deductions are sometimes made in the case of arsenic. The losses of gold and silver in treatment are usually assumed at 5 per cent. Payment is calculated on New York prices. Lead is paid for on the following scale, based upon a New York price for pig lead of 1 dol. per 100 lb.:—

Under 5 per cent., payment nothing.			
5 per cent. and under 10, payment 25 cents per unit.			
10	"	20	35
20	"	30	40
30	"	40	45
40	"	50	50
50	"	and over	55

The cost of roasting varies with the chemical and mechanical condition of the ore, the kind of furnace used; the cost and amount of labour, &c., from less than 1 dol. up to 2.75 dols. per ton.

The cost of smelting also varies greatly, additions and allowances being made as above, but the charge is based on 4.50 dols. per ton for an assumed neutral ore.

Examples are given of calculations in special cases, according to the foregoing data.—J. H. C.

#### THE BRADFORD CONDITIONING HOUSE.

*United States Consular Reports, May 1894, 2—7.*

A conditioning house is an establishment in which the true weight, length, and condition of articles of trade and commerce are determined scientifically. This is a matter on which there has always existed a difference of opinion between British exporters and American importers. Though England and the United States use the same weights and measures, there are invariably differences between the weights and measurements of yarns, wools, and pieces, from the time of their shipment from this point and their final distribution in America; so great, in fact, as to have a considerable bearing on the profits of the transaction. Certain exporters have a definite understanding of some kind with their American clients on this subject, and manage to evade serious difficulty. Others, however, have constant friction, and only recently one of the largest exporting houses in Bradford was compelled to allow an American customer a 5 per cent. discount because of the shrinkage in a large invoice of goods, though it was alleged by the exporter that it entirely obliterated the profits of the transaction. All this may be prevented by the importer of either raw material, partly manufactured, or wholly finished goods, demanding that the product be accompanied by a certificate from a legally authorised conditioning house. As a rule, the charges would be light; but they would be added to the price of the goods, unless there was a distinct understanding to the contrary. Another plan would be for the establishment of a conditioning house in New York, or any other American port of entry, for the purpose of conditioning goods there on exactly the lines followed here. The system would be equally useful for testing and conditioning American and foreign goods.

#### *The Bradford Establishment.*

The conditioning house here is owned and operated by the municipality. It is the only one in England, and was established three years ago under the Parliamentary Act of 1877. The idea originated with the Bradford Chamber of

Commerce, and was adopted by the corporation on that body's suggestion. Its popularity has been of steady growth, and last year the business transacted was twice that of the previous year. Merchants and spinners engaged chiefly in the home trade are now making great use of it for the testing of bulk lots of wool, tops, yarns, and waste. The tests on this account alone amount to 45 per cent. of the total business transacted, the remaining 55 per cent. of the business of the enterprise being export trade. A large percentage of the tests are made to settle disputes, the certificate of the conditioning house being accepted as a basis of arbitration. Bradford is fortunate in having, as manager of the concern, a gentleman of splendid scientific attainments, who is an enthusiast in the business, and who understands thoroughly the very complicated machinery used in making the tests. Tests are made by the exact and logical metric system, and the results then rendered into the English weights or measurements. They are also figured direct by the English system. Being worked under Parliamentary Act, all certificates granted are legal evidence in a court of law, and are accepted as absolutely correct as to weight, condition, and description of goods, on the date such goods passed through the conditioning house. The objects to be effected are to determine:

- (1) The average amount of moisture contained in wool, tops, noils, and yarns submitted for examination.
- (2) The correct gross weight of bales, bags, cases, skeps, sheets, and packages of goods, and the tare thereof.
- (3) The net conditioned weights of sample lots of wool, tops, noils, and yarns, after scouring, clauing, and drying.
- (4) The true counts, lengths, twists, and strengths of yarns.
- (5) The measurement of length in piece goods.

The system adopted is much the same as that in general use on the Continent. In France, Germany, and in many other countries of Europe there is a large number of these institutions, some of colossal size, notably the one at Roubaix, where about 100,000,000 kilograms of material are "conditioned" annually. As has been stated, the Bradford House, though of recent origio, is meeting with popular favour, both for home and export trade, and is being used as a means of ascertaining and accepting "the correct invoice weight" of goods at the normal (official) condition. Many other branches of testing are undertaken for the settlement of disputes and claims. The tests are made with every care that science and experience can suggest, and by the use of the best machinery and testing apparatus. I was struck with wonder in making a tour of the establishment. The puzzling machinery, the drying of wool in hot receptacles, the mazes of yarns being tested for strength, the piles of different material, the clicking and ticking of curious instruments, all combined, are calculated to have a mystifying effect upon a novice. All tests are made in duplicate, and the results and calculations worked out by separate methods and separate persons, so as to insure perfect accuracy. An official certificate is given showing the result of each test. The strictest confidence is observed, and the results of tests are made known only to the persons who send the articles to be tested. Although the official condition is given only for bulk lots—that is, when the whole consignment has been sent to the conditioning house—a special note marked "Sample Test Note," is given for samples drawn by the buyer or seller elsewhere.

#### *How Tests are made.*

On the arrival of the bulk lots, the gross weights of the sheets, bales, bags, or packages, are taken by two persons independently. The tares, if it be so desired, or if it be practicable, are taken in the same manner. A certain number of samples are then drawn for the purpose of testing their condition, and the bulk can at once be taken away. The official certificate will follow, by post or otherwise, as soon as practicable on the completion of the tests and



[Inclosure.]

FORM OF CERTIFICATE OF BRADFORD CONDITIONING  
HOUSE.

Entry No.

Prog. No.

COUNTY BOROUGH OF BRADFORD.

[SEAL.]

CONDITIONING HOUSE.

The Bradford Corporation (Various Powers) Act, 1887.

Bradford, 18

Delivered by

Gross weight being                      lbs.  
Tare being                                  lbs.  
Net weight being                        lbs.

lbs.    ozs.    drs.

Lets extracted from the  
above, weighing net  
resulting after testing  
absolutely dry

or loss

per lb.

or total dry weight                      lbs. or                      per cent.  
Official regain                      lbs. or                      per cent.  
Correct Invoice weight                      lbs. or                      per cent.  
Loss or gain                      lbs. or                      per cent.  
Original weight                      lbs. or                      per cent.

CHARGES.

Tests.....  
Weighing.....  
Repacking.....  
Puplients.....  
Postage.....  
.....

I hereby certify that the above is  
a correct return of the tests made  
by me of the samples referred to.  
In testimony whereof the corporate  
common seal of the said borough  
has been affixed herunto this  
day of 18 .

Signed \_\_\_\_\_  
Manager.

Verified by

[CORPORATE SEAL.]

## BOARD OF TRADE RETURNS.

## SUMMARY OF IMPORTS.

Articles.	Month ending 30th April.	
	1893.	1894.
	£	£
Metals.....	1,599,233	1,661,457
Chemicals and dyestuffs.....	499,855	509,251
Oils.....	550,430	560,942
Raw materials for non-textile industries.	2,941,535	3,597,390
Total value of all imports ....	34,120,160	35,908,029

## SUMMARY OF EXPORTS.

Articles.	Month ending 30th April.	
	1893.	1894.
	£	£
Metals (other than machinery) ....	2,422,875	2,426,068
Chemicals and medicines .....	818,711	747,794
Miscellaneous articles.....	2,586,400	2,477,315
Total value of all exports.....	16,617,977	17,559,876

## IMPORTS OF METALS FOR MONTH ENDING 30TH APRIL.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Copper:—				
Ore..... Tons	4,112	5,754	23,525	30,755
Regulus .....	8,291	6,523	294,118	136,444
Unwrought .... "	2,382	5,396	112,614	229,778
Iron:—				
Ore..... "	323,127	428,329	217,141	290,618
Bolt, bar, &c. .... "	2,521	2,741	20,691	24,368
Steel, unwrought.. "	391	410	3,880	4,036
Lead, pig and sheet ..	14,822	13,582	147,450	122,961
Pyrites .....	56,496	51,585	96,066	92,510
Quicksilver..... Lb.	45,140	421,200	3,796	30,313
Silver ore..... Value £	..	..	218,714	181,013
Tin..... Cwt.	70,124	80,087	328,466	277,523
Zinc..... Tons	8,951	5,295	69,838	71,613
Other articles ... Value £	..	..	122,944	149,529
Total value of metals ..	..	..	1,599,233	1,661,457

## IMPORTS OF OILS FOR MONTH ENDING 30TH APRIL.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Cocoa-nut..... Cwt.	5,946	4,850	7,695	5,630
Olive .....	2,407	3,234	92,705	111,631
Palm .....	82,304	108,218	106,222	116,721
Petroleum .....	9,333,410	11,928,146	165,118	184,599
Seed .....	2,262	1,169	55,805	42,749
Train, &c..... Tons	1,580	931	30,398	18,675
Turpentine .....	11,212	..	12,348	..
Other articles .. Value £	..	..	80,139	80,937
Total value of oils...	..	..	550,430	560,942

IMPORTS OF CHEMICALS AND DYE-STUFFS FOR MONTH  
ENDING 30TH APRIL.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Alkali..... Cwt.	6,919	7,074	5,828	6,136
Bark (tanners', &c.) ..	22,310	18,424	8,414	6,815
Brimstone..... "	31,985	43,411	7,919	8,530
Chemicals..... Value £	..	..	92,034	124,127
Cochineal ..... Cwt.	289	278	1,666	1,491
Cuteh and gambier Tons	2,529	3,554	56,888	71,425
Dyes:—				
Aniline..... Value £	..	..	12,867	14,646
Alizarin ..... "	..	..	25,760	16,916
Other ..... "	..	..	1,179	1,337
Indigo ..... Cwt.	2,339	5,125	41,781	84,243
Nitrate of potash .. "	12,578	24,551	10,638	23,592
Valonia..... Tons	5,108	1,478	62,671	17,673
Other articles... Value £	..	..	163,290	192,711
Total value of chemicals	..	..	490,875	569,251

IMPORTS OF RAW MATERIAL FOR NON-TEXTILE  
INDUSTRIES FOR MONTH ENDING 30TH APRIL.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Bark, Peruvian .. Cwt.	8,007	4,259	14,616	7,883
Bristles..... Lb.	305,186	247,521	47,899	41,195
Caoutchouc..... Cwt.	21,224	36,584	286,966	444,529
Gum:—				
Arabic..... "	6,681	3,614	13,368	9,778
Lac, &c..... "	18,270	19,735	89,875	99,308
Gutta-percha .... "	1,024	4,853	10,871	52,012
Hides, raw:—				
Dry..... "	34,113	51,806	93,716	117,202
Wet..... "	33,643	37,231	67,772	73,996
Ivory..... "	558	1,063	25,432	44,537
Manure:—				
Guano..... Tons	2,676	6,314	19,268	26,176
Bones..... "	2,683	8,410	12,539	39,448
Nitrate of soda... "	5,171	25,539	53,975	247,386
Phosphate of lime .. "	14,911	19,553	24,463	32,264
Paraffin..... Cwt.	73,102	54,416	76,179	53,862
Linen rags..... Tons	349	1,649	1,890	15,987
Esparto..... "	18,185	19,089	87,921	84,514
Pulp of wood .... "	18,391	21,421	99,842	114,175
Rosin..... Cwt.	101,625	97,856	22,299	18,711
Tallow and stearin .. "	148,369	224,979	269,862	291,187
Tar..... Barrels	2,650	2,084	1,517	2,096

IMPORTS OF RAW MATERIAL FOR NON-TEXTILE  
INDUSTRIES FOR MONTH ENDING 30TH APRIL—cont.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Wood:—				
Hewn ..... Loads	153,167	226,239	288,480	354,781
Sawn ..... "	165,939	246,871	359,730	378,500
Staves ..... "	8,921	9,024	52,673	46,298
Mahogany ..... Tons	8,445	3,902	78,225	28,599
Other articles.... Value £	..	..	929,794	779,316
Total value .....	..	..	2,911,545	3,597,399

Besides the above, drugs to the value of 64,975*l.* were imported, as against 80,363*l.* in April 1893.

EXPORTS OF METALS (OTHER THAN MACHINERY) FOR  
MONTH ENDING 30TH APRIL.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Brass..... Cwt.	8,913	9,959	35,213	37,389
Copper:—				
Unwrought..... "	57,595	35,967	149,957	78,791
Wrought..... "	28,925	22,528	84,719	61,706
Mixed metal .... "	24,943	22,478	59,569	50,470
Hardware..... Value £	..	..	162,857	153,117
Implements..... "	..	..	94,875	97,628
Iron and steel..... Tons	252,459	249,351	1,821,265	1,567,953
Lead ..... "	2,913	4,158	35,523	45,347
Plated wares... Value £	..	..	26,179	21,609
Telegraph wires .. "	..	..	43,475	186,739
Tin ..... Cwt.	6,965	13,364	33,659	49,741
Zinc ..... "	20,567	10,651	17,498	8,460
Other articles .. Value £	..	..	68,446	68,227
Total value .....	..	..	2,692,875	2,426,068

EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING  
30TH APRIL.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Alkali..... Cwt.	617,179	465,999	242,445	132,147
Bleaching materials .. "	143,134	107,491	54,739	42,169
Chemical manures. Tons	24,794	26,694	188,799	192,576
Medicines..... Value £	..	..	87,210	80,628
Other articles .... "	..	..	289,608	309,534
Total value .....	..	..	818,711	747,794

EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH  
ENDING 30TH APRIL.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Gunpowder..... Lb.	858,700	567,400	25,792	11,212
Military stores.. Value £	..	..	75,258	89,431
Candles..... Lb.	1,466,600	1,268,100	27,961	22,195
Caoutchouc..... Value £	..	..	93,312	92,512
Cement..... Tons	39,461	38,831	67,006	63,327
Products of coal Value £	..	..	131,200	117,553
Earthenware ... ..	..	..	150,176	127,655
Stoneware..... ..	..	..	14,805	14,262
Glass:—				
Plate..... Sq. Ft.	125,087	95,187	6,371	4,974
Flint..... Cwt.	7,292	6,526	19,031	15,576
Bottles..... ..	57,080	59,968	26,092	28,591
Other kinds.... ..	19,918	13,403	16,577	10,586
Leather:—				
Unwrought .... ..	11,568	12,467	110,177	110,519
Wrought ..... Value £	..	..	23,010	20,507
Seed oil..... Tons	5,366	6,743	118,705	139,082
Floorcloth ..... Sq. Yds.	1,306,900	1,725,400	54,380	64,745
Painters' materials Val. £	..	..	128,402	119,832
Paper ..... Cwt.	80,247	76,974	126,259	121,206
Bags..... Tons	5,057	4,536	49,426	29,576
Soap..... Cwt.	11,722	17,180	48,705	50,566
Total value .....	..	..	2,586,400	2,477,315

## Monthly Patent List.

\* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### APPLICATIONS.

7492. C. F. Young. Improvements in agitators or stirrers for pans in which liquids, starch, and other pastes, colours, and other substances are heated or cooled. April 16.

7626. W. Bagley. Improvements in apparatus for feeding coal to gas producers. April 17.

7651. W. Lawrence. Improvements in processes and apparatus for drying organic and inorganic substances, or in extracting the moisture from brewers' grains, earthy substances, or solutions of salts, and such like. April 17.

7893. J. T. Sands. Improvements in apparatus for bleaching smoke. Complete Specification. April 21.

8545. C. G. J. Möller and P. Pfeiffer. Improvements in and relating to drying and evaporating apparatus. April 30.

8643. C. L. W. Albach. Improvements relating to the lixiviation of dyewood, bark, hops, and other materials, and to apparatus therefor. May 1.

9049. E. A. Uehling and A. Steinbart. Improvements in and relating to processes and apparatus for analysing gases and gaseous mixtures. Complete Specification. May 7.

9186. W. Cliff and M. Mannaberg. Improvements in apparatus for calcining lime, dolomite, and the like. May 9.

9198. B. G. Davis-Cooke. Improvements in the method of manufacturing ganister for the lining and repair of cupolas, furnaces, and other similar purposes, and the manufacture of bricks and other fireclay goods from the same. May 9.

9210. E. Cramer. Improvements in kilns for burning cement and other minerals. Complete Specification. May 9.

9395. T. E. Pye and C. E. Seull. Improvements in valves or regulators for gas and chemical works.

### COMPLETE SPECIFICATIONS ACCEPTED.\*

#### 1893.

10,467. E. Latham and E. G. Guyot. Improvements in apparatus for evaporating liquids by means of steam. May 16.

10,870. J. Fischer. Improvements in apparatus for cooling, heating, drying, or similar purposes. May 9.

12,018. J. Clarkson and S. Mortimer. Improvements in apparatus for supplying air and steam to furnaces so as to effect a more complete combustion of fuel and prevent the formation of black smoke. May 9.

12,774. F. W. Scott, E. G. Scott, and F. W. Scott, jun. Improvements in evaporating brine and other liquors, and apparatus therefor. May 9.

12,972. A. Horn. New or improved cooling apparatus. May 9.

13,146. F. Spence. A new or improved hydrostatic apparatus for automatically mixing with water or other liquid disinfectants, softening or purifying substances in definite proportions. May 16.

18,652. C. Latarche. An improved actinometric pyrometer. May 9.

#### 1894.

1377. C. Morel and A. Heimpel. Improvements in apparatus for crushing dry or wet materials, such as cements, lime, plaster, alabaster, kaolin, quartz, phosphates, minerals, and the like. April 25.

5262. W. Henneberg. Improvements in apparatus for condensing vapours and heating or vaporising liquids respectively. May 16.

### II.—FUEL, GAS, AND LIGHT.

#### APPLICATIONS.

7527. C. Barker. An improved fire-fighter for lighting coal or coke fires. Complete Specification. April 16.

7599. J. A. A. Nyström. An improved fire-lighter. April 17.

7772. G. S. Cory and C. Cory. Improvements in the manufacture of artificial fuel. April 19.

7827. C. E. Jolly and H. J. Reid. Improvements in machinery or apparatus for the manufacture of briquettes from fuel material. April 20.

7911. R. Middleton. Improvements in machinery for manufacturing blocks or briquettes of fuel or materials for smelting and analogous purposes. April 21.

8998. L. Dardel and H. Bécoulet. An improved solid combustible and process of manufacturing the same. April 24.

8117. J. Posno. Improvements relating to the treatment of sewage and town refuse for transforming the same into fuel. April 24.

8598. H. J. P. Sprengel. Improvements in obtaining oxygen. April 30.

8588. A. G. Glasgow. Improvements in water-gas apparatus. Complete Specification. May 1.

8670. B. H. Thwaite. Improved method of utilising the waste gases from iron-smelting blast-furnaces. May 2.

8851. J. H. Hilton. Improvements for carburising coal and other gases. May 4.

8974. T. G. McEwen. Improvements in the manufacture of gas. May 5.

9197. H. Hawkins and S. H. Hawkins. Improvements in the production of hydrocarbon gas and metal sulphates as by-products, and in apparatus therefor. May 9.

9274. E. Stauber. Improvements in the process of and apparatus for producing peat briquettes and the like. May 10.

9387. C. Schmitz. Improvements in or relating to the combustion of fuel. May 11.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

8499. E. Hedges. Improvements in the manufacture of fire-lighters. May 2.

10,554. The Manchester Oxygen (Brin's Patent) Co. and W. M. Jackson. Improvements in and in the mode of utilising the pressure in cylinders or reservoirs of compressed gases, in the application or employment of such gases. May 16.

11,961. H. W. Crowther. Improvements in the treatment of the waste from gas-liquor to obtain useful products therefrom. April 25.

12,247. A. Norreys. Improved means or process for producing block fuel. May 9.

22,576. F. D. Marshall. Improvements in apparatus for washing and purifying gas. May 9.

1894.

1690. B. H. Thwaite and G. Threlfall. Improvements in methods and apparatus for generating gaseous fuel from solid hydrocarbonaceous or carbonaceous material. April 25.

#### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

##### APPLICATIONS.

7862. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the purification of anthracene. April 20.

8371. J. Bryson, J. Jones, and W. Fraser. Improvements in or relating to retorts for the distillation of shale and other bituminous substances. April 27.

#### IV.—COLOURING MATTERS AND DYES.

##### APPLICATIONS.

7540. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of dyestuffs. April 16.

7786. H. H. Lake.—From Wirth and Co., Agents for A. Leonhardt and Co., Germany. Improvements in the manufacture of colouring matters. April 19.

7859. C. D. Abel.—From The Actien-Gesellschaft für Anilin-Fabrikation, Germany. Manufacture or production of colouring matters and materials therefor. April 20.

8381. C. D. Abel.—From The Actien-Gesellschaft für Anilin-Fabrikation, Germany. Manufacture of a new naphthylendiamine mono sulpho acid, and of colouring matters derived therefrom. April 27.

8536. E. Petersen-Müller. Manufacture of colouring matters, dyeing un mordanted cotton, and their salts and sulpho-derivatives. Complete Specification. April 30.

8541. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of phenol alcohols. April 30.

8564. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. The manufacture and production of new azo colouring matters. April 30.

8867. J. H. Lütkehermölle. New bases from benzidine, zolidine, &c. May 1.

9291. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in the manufacture of artificial indigo-white and of bodies related to the indigo group. May 10.

9441. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of dyestuffs. May 12.

9443. J. Imray.—From R. Vidal and La Société Anonyme des Matières Colorantes et produits Chimiques de St. Denis, France. Manufacture of colouring matters applicable to un mordanted fibres or fabrics. May 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

8093. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of dyestuffs. March 28.

10,996. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik. The manufacture and production and application of new colouring matters or dyes from naphthalene. May 2.

11,892. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of dyestuffs. April 25.

13,394. H. E. Newton.—From the Farbenfabriken vormals F. Bayer and Co. The manufacture of new leuco compounds and the production of colouring matters therefrom. May 16.

13,395. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of colouring matters by reduction of tetranitroanthrachry-one and dinitroanthrachrynone-disulphonic acid. May 16.

13,402. H. H. Lake.—From K. Ochler. Improvements relating to the production of amidophenol and amidoeresol-sulpho acids, and to the manufacture of colouring matters therefrom. May 16.

13,419. A. Ashworth and J. Burger. Improvements relating to the production of colouring matters. May 16.

1894.

5143. R. Korff. Manufacture of new amido bases and of azo colouring matters therefrom. May 2.



5870. J. Grime. A new or improved method of oxidising jigo fibres and the like, and apparatus employed therefor. April 23.

6004. F. Muhlert. Manufacture of yellow and orange colouring matters from *B-resorcylic acid*. May 2.

## V.—TEXTILES, COTTON, WOOL, SILK, Etc.

### APPLICATIONS.

7642. A. V. Newton.—From A. Nobel, France. Improvements in the manufacture of artificial silk and substitutes for vegetable fibre. April 17.

7690. T. Bedford and F. Bedford. An improved process of preparing rhea or ramie fibre, and in the manufacture of ropes and twines therefrom. April 18.

7907. W. Pickstone and A. Greenhalgh. Improvements in and relating to the manufacture of crinkled textile fabrics. April 21.

7975. O. Reichenbach. New or improved apparatus for use in degreasing or otherwise cleansing wool or other fibres. April 23.

8038. D. E. Radclyffe. Improved apparatus or method or system for preparing china, grass, or rhea fibre, or hemp, or similar fibres, for combing or spinning. April 24.

8893. E. Cadoret and A. Jost. An improved process for treating vegetable substances suitable for the manufacture of woven fabrics, by converting them into soft and silky fibres adapted for spinning and dyeing. May 4.

### COMPLETE SPECIFICATIONS ACCEPTED.

1891.

3484. O. Reichenbach. An improved process and apparatus for cleaning wool and other textile products. May 2.

4396. J. S. Farmer, H. L. Storey, and I. H. Storey. Improvements in the manufacture of solid-colour floorcloths, and in machinery employed in such manufacture. May 15.

5718. H. Rous and G. N. West. A new or improved process for rendering textile materials waterproof and flameproof. April 25.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

### APPLICATIONS.

7650. F. G. Armfield. See Class XIV.

7885. J. S. Lord and G. S. Lord. Improvements in machines for washing, scouring, mordanting, bleaching, sizing, and dyeing yarn in the hank. April 21.

8278. A. Whittall. Improvements in apparatus for dyeing yarns. April 26.

8558. E. Cadoret and A. Vost. Improvements in the process of and apparatus for bleaching and treating fibrous materials and fabrics. April 30.

8767. F. Barraclough. Improvements in machines for scouring and dyeing textile warp or other yarns. May 3.

8964. J. W. Abom. Method of bleaching paper pulp, or other flecky substances. Complete Specification. May 5.

8969. G. N. Midwood and C. Edmeston. Improvements in dyeing and otherwise treating cotton and other piece goods, and in machinery or apparatus to be used for the purpose. May 5.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

9371. C. D. Abel.—From The Farhwerke vormals Meister, Luthius, and Brauning. A process for dyeing wool with fast colours. May 9.

11,891. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The production of colours on fibres. May 16.

11,929. J. Riley and The Thornliebank Company, Limited. Improvements in the bleaching or cleansing of vegetable yarns and fabrics. April 25.

12,076. J. Grime. Improvements in vats for dyeing indigo-blues and the like. April 25.

12,367. L. Frakenburg and C. O. Weber. Improvements in or connected with the producing or printing of designs upon waterproof fabrics. May 2.

15,083. H. H. Lake.—From E. Michaelis and Co. An improved process for dyeing textile materials. May 2.

1894.

7641. W. H. Hughes and S. F. Hughes. Improved process for scouring, cleansing, bleaching, and disinfecting wool and other fibres or fibrous materials. May 16.

## VII.—ACIDS, ALKALIS, AND SALTS.

### APPLICATIONS.

7554. G. T. Beilby. The manufacture of cyanides. April 17.

7578. F. Hamer. The production of nitric acid  $\text{HNO}_3$ , and the nitrates of soda, of potash, and of lime. April 17.

7603. C. J. Hammerton. See Class XI.

7636. A. Shearer and R. R. Clapp. Improvements in the manufacture or production of phosphorus and compounds thereof. April 17.

7671. C. M. Pielsticker. Improvements in the production of sodium and potassium. April 18.

8048. R. Hodgson. A new or improved process and apparatus for the manufacture of salt. April 24.

8305. H. W. Crowther, E. C. Rossiter, J. J. Hood, and G. S. Albright. Improvements in the manufacture of ferrocyanides of potassium and sodium from their sulphocyanides, and apparatus relating thereto. April 26.

8652. R. H. Leaker. Improvements in the manufacture of vinegar. May 2.

8690. H. Fielder. Improvements in apparatus for and process in the manufacture of chlorate of potassium. May 2.

8889. O. Imray.—From Goerlich and Wichmann, Germany. An improvement in the manufacture of cyanides of the alkaline metals. May 4.

9004. J. F. Hutcheson. Improvements in the manufacture of acetate of lead. May 7.

9197. H. Hawkins and S. H. Hawkins. See Class II.

9250. R. Hodgson. Improvements in machinery or apparatus for obtaining salt from brine. May 10.

9275. H. W. Crowther, E. C. Rossiter, and G. S. Albright. Improvements in the manufacture of cyanides. May 10.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

9521. W. V. Wilson and J. J. Lundy. Improvements in the manufacture of sulphate of lead. May 16.

12,572. J. Addie, J. Cunninghame, and W. Macfarlane. Improvements in the production of cyanides. May 9.

13,655. F. Maxwell Lyte and G. Lunge. Improvements in the production of caustic alkali and chloride of lead for conversion into chlorine and refined lead. May 16.

13,656. G. Lunge and C. H. Maxwell Lyte. An improved process for producing basic lead salts and obtaining certain by-products. May 16.

13,842. T. Chaudelon. New process for the preparation of bieleite phosphate soluble in ammoniac citrate. April 25.  
13,927. A. Müller. An apparatus for obtaining carbonic acid. May 9.

15,475. R. H. Leaker. Improvements in the manufacture of vinegar. May 2.

19,705. B. K. Rigby, F. A. Remington, and A. C. Carr. See Class IX.

1894.

5808. J. D. Darling and H. C. Forrest. Improvements in producing nitric acid and metals from nitrates. May 2.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

### APPLICATIONS.

8076. D. Jones, W. W. Wardle, F. C. Wardle, and A. W. Beech. Improvements in ornamenting china, earthenware, and other brittle articles. April 21.

8523. A. Mason and H. E. Mason. Improvements in the manufacture of fireclay tuyères and apparatus for effecting same. April 30.

8575. J. Hughes. An improved method of decorating on pottery. May 1.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

8555. A. D. Brogan.—From The Glasgow Plate Glass Co. Improvements in apparatus for rolling glass. May 2.

11,098. H. Tugby. Improvements in the manufacture and production of stoneware sanitary pipes and the like. May 9.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### APPLICATIONS.

8468. A. W. Keim. An improved material for wall-plastering, priming, casting, or the like, and process for producing the same. April 28.

8175. J. L. Kerr. Artificial plaster of Paris, made from the lime residue resulting from the reduction of sulphate of ammonia. April 28.

8549. R. Stone. Improvements in the manufacture and treatment of fire-proof plastic material for cement. Complete Specification. April 30.

8977. G. Beekert. Improvements in building blocks and slabs. May 5.

9455. R. Hauers. Improvements in the preservation or treatment of buildings, structures, building materials, and the like from dirt or decay. May 12.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

9550. R. Haddan.—From G. R. Anderson, A. H. Shotton, W. Koehler, and C. H. Dover. An improved artificial asphaltum or pitch, and process of making same. May 16.

12,468. Goegg, Fruh, and Co. Improved decorative material on surface in imitation of marble. May 2.

13,598. J. Fisher. Improvements in and connected with concrete and similar pavements and floorings. May 16.

19,705. B. K. Rigby, F. A. Remington, and A. C. Carr. Improvements in the manufacture of cement from Chance or Le Blanc alkali waste or the like. April 25.

1894.

2582. C. H. Slicer. Improvements in and relating to the production of asphaltic composition in the form of a powder or of a mastic. May 2.

4223. P. Baumert and A. Meck. Improvements in grinding or pulverising machines. May 2.

5595. C. A. Ropes and J. C. Sellars. Improvements in or connected with cements or cement work. April 25.

## X.—METALLURGY, MINING, ETC.

### APPLICATIONS.

7672. C. M. Pielsticker. Improvements in the production of metal alloys. April 18.

7854. W. A. Thoms and W. H. Burgum. An improvement in the coating of metals with a metallic alloy. April 20.

8085. P. C. Choate. Improvements in the art of producing metallic zinc. Complete Specification. April 24.

8087. A. A. Ackerman. An improved process for the manufacture of hard-face armour plate. Complete Specification. April 24.

8136. H. W. Gablett-Fairfax. Improvements relating to methods of and apparatus for hardening or tempering steel plates and other articles. April 24.

8183. W. P. Thompson, From L. R. Bonehill and E. T. Gantier, Belgium. Improved process and apparatus for extracting metals from their ores. April 25.

8184. J. S. MacArthur. Improvements in precipitating precious metals from cyanide or similar solutions. April 25.

8306. C. T. J. Vautin. An improved process for the extraction of metals from oxides and sulphides. April 26.

8370. B. P. Stockman. Improvements in the manufacture of steel and iron. April 27.

8401. H. Ramage. Improvements in or connected with the preparation or coating of aluminium surfaces, and in alloys or metallic mixtures therefor, and for soldering. April 27.

8405. H. L. Salzman. Improvements in or relating to the treatment of ores. April 27.

8586. C. M. Allen. Processes of and converters for smelting and refining ores. Complete Specification. May 1.

8712. J. Brock and J. F. Allen. Improvements in the manufacture and production of alloys of copper and manganese. May 2.

8790. M. T. Brown.—From J. C. Preston, Australia. A method of and apparatus for the extraction of gold and silver from their ores or tailings. May 3.

8899. H. F. Taylor. Improvements in apparatus for coating with tin, lead, or other metals or alloys of same. May 4.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

2639. J. A. Mays. Process of and apparatus for separating metals and similar substances. May 16.

10,608. D. W. Sugg. An improved metallic alloy. May 2.

11,709. J. B. Cochrane and T. H. Taylor. Improvements in the purification of iron for casting and general foundry purposes, as also in the production of steel and wrought iron. May 16.

12,034. G. Bischof. Improvements in the treatment of lead fumes. May 16.

1894.

5530. E. Singer, F. Donat, and P. Kirchseisen. Process for soldering aluminium. May 9.

5808. J. D. Darling and H. C. Forrest. See Class VII.

## VI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### APPLICATIONS.

7603. C. J. Hammerton. Application of electricity to the manufacture of cyanides and ferrocyanides and improvement in same. Preservation of vessels from corrosion. April 17.

7801. C. Kellner. Improvements in or relating to electrolytical apparatus. April 19.

7853. W. A. Thoms and W. H. Burgum. An improvement in the electro-deposition of heavy metals with a base metal alloy. April 20.

7865. D. G. Fitzgerald and A. W. Southey. Improvements in the construction and working of galvanic batteries. April 20.

7950. H. Guthrie. Improvements in apparatus for electrolytical purposes. April 21.

7985. T. Drake. An improved method, means, or process of producing caustic soda and chlorine gas by the electrolysis of common salt in solution. Complete Specification. April 23.

7994. E. de Pass.—From H. Thofehn, France. An improved electrolytic trough or cell. April 23.

8061. H. Carmichael. Improved apparatus and process for electro-chemical decomposition. Complete Specification. April 24.

8350. H. H. Leigh.—From Vohwinkel, Austria. Improvements in electric batteries. April 27.

8480. T. H. Rushton. Improvements in and relating to galvanic batteries. April 28.

8731. T. Froggatt. Improvements in electric batteries. May 2.

8808. R. McLauchlan McDonald and A. McDonald. Improvement in electric batteries. May 3.

8815. L. Mond and R. L. Mond. Improvements in electrolytic cells.

8895. F. King. Improvements in or connected with secondary or electric storage batteries. May 4.

8917. H. Alexander. Improvements in or in connection with apparatus for the electro-deposition of metals. Complete Specification. May 4.

8972. F. Griffin. Improvements in or relating to secondary batteries. May 5.

9129. H. Blumenberg, jun. Improvements in electrolysis. May 8.

9285. C. Kellner. Improvements in or relating to electrodes and method of manufacturing the same. May 10.

9292. E. A. Allen. Improvements in or connected with electrolytic cells. May 10.

9328. B. Hooker. An improvement in and relating to apparatus for electrolysis. May 11.

9363. F. Brain. Improvements in electric fuses. May 11.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

7906. D. G. Fitzgerald. Improvements in the manufacture of elements or electrodes for voltaic batteries and electrolytic decomposition cells. May 16.

12,857. J. C. Richardson. Improvements in and connected with tanks for the electrical decomposition of chloride of sodium or potassium in solution. May 9.

13,318. The Lithanode and General Electric Co., Lim., and J. T. Niblett. Improvements in plates or elements for secondary electric batteries. May 16.

13,274. C. Rousseau. Improvements in the manufacture of plates for electric accumulators. May 16.

13,339. H. H. Lake.—From A. C. Girard and E. A. G. Street. An improved manufacture of carbon for electrical purposes. May 9.

13,568. C. T. J. Vantou. An improved dry electrolytical process for the production of an alloy of lead and sodium or potassium. May 16.

22,912. E. H. Wheeler. Improvements in electrodes and cells for secondary batteries or accumulators. April 25.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

### APPLICATIONS.

7519. F. Boek and E. Wipperf. Improved soap. April 16.

7520. F. Boek and E. Wipperf. Improved soap. April 16.

7548. J. Y. Johnson.—From L. Rivière, France. Improvements in the manufacture and use of fatty acids. April 16.

7846. W. B. Leachman. Improvements in or connected with apparatus for purifying or clarifying oils, fats, and other similar materials. April 20.

8022. J. H. Iredale and J. W. W. Huddleston. Improvements in soap. April 23.

8399. A. G. Spilbury. Improvements in the manufacture of paraffin wax. April 27.

8669. J. F. Bennett, A. Firth, and F. H. Firth. Improvements in lubricants and method of applying same for gas engines, oil engines, and other cylinders. May 2.

8676. W. H. Horton. An improvement in what is commonly called dry soap or soap powder. Complete Specification. May 2.

8965. B. Weigel and J. Hands. Improvements in the manufacture of detergents. May 5.

9179. A. Wendtland. An improved process for removing the green colour from paraffin, vaseline, and lubricating oils, which have had that colour imparted to them by bodies containing oxygen. Complete Specification. May 9.

9200. M. Zingler and W. Chattaway. See Class XV.

9364. D. Young.—From J. Marini, Italy. An improved lubricant, chiefly designed for use on the collectors of dynamo electric machines. May 11.

### COMPLETE SPECIFICATION ACCEPTED.

1894.

5821. F. J. Machalski. Improvements in apparatus for extracting fat and glue. May 9.

## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

### APPLICATIONS.

5856a. H. P. Scott. Improvements in anti-corrosive compositions or paints for coating iron, steel, or metal structures. Filed April 26, date claimed 21 March 1894.

5856b. H. P. Scott. Improvements in anti-corrosive compositions or paints applicable for coating iron, steel, or other metal surfaces exposed to the action of petroleum, turpentine, or like liquors. Filed April 26, date claimed 21 March 1894.

8005. E. C. Marks. A compound for a liquid preparation for reproducing pictures from illustrated papers on writing or drawing paper in black or colours. April 23.

8112. C. W. Kriens. Improvements in the manufacture of animal charcoal. April 24.

8178. C. M. Pielsticker. Improvements in the production of copper colours. April 25.

8213. W. C. Horne. Improvements in the manufacture of zinc-white paints. April 25.

8297. A. S. Caine and H. Gillmer.—From C. L. Bartlett, United States. An improved paint compound or pigment. April 26.

8298. A. S. Caine and H. Gillmer.—From C. L. Bartlett, United States. An improved paint compound or pigment. April 26.

8806. G. W. Thomas. Improvements in compositions for use in waterproofing absorbent matters and materials and for preserving metals from oxidation. May 3.

9194. W. H. Hyatt. Improvements in the manufacture of paint. May 9.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

9930. J. Renison. A new material for making varnish and the like. May 9.

12,791. E. Edwards. — From J. W. Overton. An improved paint. May 16.

1894.

1301. J. W. Blake and S. J. Blake. Improvements in compositions or paints for preserving ships' bottoms and other structures. May 9.

#### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

##### APPLICATIONS.

7650. F. G. Armfield. A new and improved process for staining any kinds of tanned leather in a variety of shades of colour on each single skin. April 17.

8894. J. Rees. Improvements in the treatment of gelatine and the manufacture of articles therefrom. May 4.

9090. R. Combret. Improvements in the manufacture of substances suitable for finishing, polishing, preserving, or otherwise treating leather skins, hides, and the like. May 8.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

12,849. C. Heinzerling. Improvements in tanning. May 2.

1894.

5821. F. J. Machalski. See Class XII.

#### XV.—AGRICULTURE AND MANURES.

##### APPLICATIONS.

8887. F. W. Saatmann. Improvements in the manufacture of superphosphates. May 4.

9200. M. Ziegler and W. Chattaway. Improvement of manufacturing guano from the refuse of fish and other animal matter, and the extraction of oil or fat therefrom. May 9.

#### COMPLETE SPECIFICATION ACCEPTED.

1893.

11,804. H. E. Newton.—From C. Seybold and F. Heeder. Improvements in the manufacture of manures and new processes for obtaining the same. April 25.

#### XVI.—SUGARS, STARCHES, GUMS, Etc.

##### APPLICATIONS.

8463. B. Laeh and H. Benies. Process for purifying and decolourising saccharine juices and solutions. April 28.

9054. H. H. Lake.—From A. L. Todesco, France. Improvements relating to the manufacture of sugar, syrups, alcohol, and beer. May 7.

9436. E. Hubner. Improvements in the manufacture of white sugar and the like from molasses. Complete Specification. May 12.

#### XVII.—BREWING, WINES, SPIRITS, Etc.

##### APPLICATIONS.

8212. F. W. M. Karsten.—From E. Karsten, Germany. Improved means or method of treating barley and other grain for its conversion to malt for brewing, distilling, or other purpose, and in the construction of apparatus connected therewith. April 25.

9054. H. H. Lake.—From A. L. Todesco, France. See Class XVI.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

10,914. E. O. Greening and E. W. Greening. An improved method of utilising spent hops for food and litter. May 9.

11,936. F. R. Stone. An aerator and rouser for wort or liquor. May 9.

23,998. C. Zimmer. Improvements in the process and apparatus used in brewing. May 9.

1894.

3642. J. C. Pennington. Improved process for the manufacture of beer. May 2.

#### XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

##### APPLICATIONS.

##### A.—Chemistry of Foods.

7543. E. O. Tatlin. An improved composition for use as food, and method of preparing the same. Complete Specification. April 16.

7612. R. G. Nash. Improvements in or relating to the "aeration of milk." April 17.

8744. F. Goracci and A. Anselmi. A new or improved compound for use as an ingredient for soup or other food and method of manufacturing the same. May 2.

8814. A. C. Sterry. Improvements in the preservation of eggs. May 3.

##### B.—Sanitary Chemistry.

7574. G. C. Purvis. New or improved method of sewage purification. April 17.

7656. F. W. Stoddart. Improved apparatus for the treatment of sewage and similar liquids. April 18.

7992. R. Schneider. Improvements in and apparatus for the utilisation of refuse. April 23.

8117. J. Posno. See Class II.

9338. O. Schmidt. An improved process for purifying sewage and waste water of a putrescible character. May 11.

#### COMPLETE SPECIFICATIONS ACCEPTED.

##### A.—Chemistry of Foods.

1893.

6662. J. Miles. Improvements in the manufacture from vegetable substances of products suitable for use as cattle food, for making boiler compositions or fluids, and for other purposes. May 2.

15,714 J. Y. Johnson.—From C. F. Boehringer and Soehne. Improvements in the manufacture of compounds for alimentary and medicinal purposes. May 16.

*B.—Sanitary Chemistry.*

1894.

1169. A. J. Boulton.—From F. S. Salberg and M. Neumann. Improvements in or relating to the disinfection of house or other refuse and apparatus therefor. May 16.

*C.—Disinfectants.*

1893.

9359. G. Whalley. A new compound or mixture for purifying sewage and foul water or liquid. May 2.

**XIX.—PAPER, PASTEBOARD, Etc.**

*APPLICATION.*

8766. J. P. Cornett. Improvements in or relating to the manufacture of paper. May 3.

*COMPLETE SPECIFICATION ACCEPTED.*

1893.

13,175. G. Sinclair. Improvements in or connected with boilers for boiling or chemically treating and for washing esparto grass and other grasses, straw, wood, rags, and other fibrous materials for paper-making. May 9.

**XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.**

*COMPLETE SPECIFICATIONS ACCEPTED.*

1893.

16,589. B. Willeox.—From C. F. Boehringer and Soehne. Process for obtaining an iron albumen derivative from animal organs. May 9.

1894.

4018. W. Mallmann. An improved manufacture of artificial musk. April 25.

6190. F. Krafft and A. Roos. Manufacture of esters, phenol, and other ethers by means of aromatic sulphonic acids. May 16.

**XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.**

*APPLICATIONS.*

7973. J. M. Gillies, R. M. Inglis, E. E. F. d'Albi, and W. C. Nixon. Improvements in colour photography. April 23.

8016. B. J. Edwards. Improvements in the manufacture of photographic films and plates, and in apparatus for use therein. April 23.

*COMPLETE SPECIFICATIONS ACCEPTED.*

1893.

8956. C. E. Pettritt. Improvements in the manufacture of sensitive plates and films for photographic purposes. May 2.

10,685. W. Jones and H. Jones. Improvements in or connected with the production of photographs on slate. May 2.

11,796. F. Steinberg. An improvement in photography. April 25.

11,872. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation. Improvements in photographic developers. April 25.

20,342. F. J. M. Gerland, W. C. Hesse, and M. Levy. Improvements in half-tone negatives for photo-mechanical processes. April 25.

21,379. A. G. Adamson. Improvements in the application of electric light for photographic purposes and apparatus therefor. May 2.

**XXII.—EXPLOSIVES, MATCHES, Etc.**

*APPLICATIONS.*

7944. L. de Stein and J. Joscht. Improvements in the manufacture of matches and of rubbing surfaces therefor. April 21.

8805. G. W. Thomas. Improvements in matches, fuses, and the like. May 3.

9017. H. J. P. Sprengel. Improvements in the preparation of explosive compounds. May 7.

9149. T. Tallyn. Railway detonating signal and apparatus. May 9.

*COMPLETE SPECIFICATIONS ACCEPTED.*

1893.

9688. J. McNab and J. Dickson. Improvements in gun-powder cartridges for mining and blasting, and the apparatus employed therein. May 16.

1894.

1846. G. E. Cassel and D. Kempe. An improved process of preparing pine or analogous wood for the manufacture of matches. May 9.

4626. A. Maurette. An explosive composition. May 2.

5939. H. Priester. An improved compound for use in the manufacture of matches. April 25.

*PATENT UNCLASSIFIABLE.*

*COMPLETE SPECIFICATION ACCEPTED.*

1893.

10,979. F. M. Grumbacher. Improvements in and relating to the treatment of vegetable substances and the like for preserving and vulcanising the same and for obtaining products of distillation therefrom. April 25.

# THE JOURNAL OF THE Society of Chemical Industry:

## A MONTHLY RECORD

FOR ALL INTERESTED IN CHEMICAL MANUFACTURES.

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### NOTICES.

In accordance with the provisions of Rule 18 of the By-Laws, notice is hereby given that those Members of Council whose names are placed in italics in the annexed list, will retire from their respective offices at the forthcoming Annual General Meeting.

Dr. T. E. Thorpe, F.R.S., has been nominated to the office of President; and Mr. E. C. C. Stanford has been nominated Vice-President under Rule 11.

Mr. John Calderwood, Dr. C. Dreyfus, Dr. A. Dupré, F.R.S., and Prof. W. A. Tilden, F.R.S., have been nominated Vice-Presidents under Rule 8; and Mr. Wm. Thorp has been nominated an Ordinary Member of Council under Rule 17, in the place of Dr. C. Dreyfus, nominated a Vice-President.

Mr. C. A. Leysitt has been nominated an Ordinary Member of Council under Rule 24, and the Treasurer and Foreign Secretary have been nominated for re-election to their respective offices.

Mr. David Howard, Mr. George N. Stoker, and Mr. Frank Wilson have been nominated under Rule 18, and Prof. A. K. Huntington has been nominated under Rule 19, to fill four vacancies among the Ordinary Members of Council.

A Ballot List and Member's Ticket are enclosed herewith.

#### ANNUAL GENERAL MEETING.

The Annual General Meeting will be held in Edinburgh on the 17th, 18th, and 20th July next. The following is a synopsis of the arrangements:—

##### PROGRAMME.

*Wednesday, July 18th.*

9.30 a.m. COUNCIL MEETING—Queen Street Hall.

A. 10.0 a.m. GENERAL MEETING—Queen Street Hall.

##### BUSINESS:

1. Reports of Council and Treasurer.

2. Election of Office-bearers.

3. Presidential Address.

4. Miscellaneous Business.

5. Place of next Annual Meeting.

12.0 noon. ALTERNATIVE EXCURSIONS.

B. Coaches will start from the Waterloo Hotel for Rosslyn Chapel, thence *via* Hawthornden to Dalkeith Palace. By the kind permission of His Grace the Duke of Buccleuch, Dalkeith Palace and Grounds will be open for the Society's inspection.  
(Ticket, 2s.)

C. Train from Waverley Station for Penicuik, where the following paper mills will be visited:—

Messrs. Alex. Cowan and Sons, Lim., Valleyfield—Bag and Esparto.

Messrs. James Brown and Co., Eskmills—Esparto and Remedial Measures.

(Ticket, 1s. 6d.)

D. WALKS IN OLD EDINBURGH.—Baillie Dunlop and Messrs. Davidson and Ross have kindly consented to conduct small parties of Members to places of architectural and antiquarian interest in Edinburgh. Proposed excursions will be posted from time to time in the offices at headquarters.

E. 4.30 p.m. GARDEN PARTY, by invitation of Sir JAMES A. RUSSELL, Bt., the Honourable the Lord Provost of Edinburgh, at his residence, Woodville, Canaan Lane.

F. 8.30 p.m. Reception and Conversazione, by invitation of the Honourable the Lord Provost, Magistrates, and Council of Edinburgh, in the Museum of Science and Art.

*Thursday, July 19th.*

Excursions to the following groups of works have been arranged:—

*Note.*—As the time available for the inspection of these works extends only from 9 a.m. till 1 p.m., it will not be possible for any party to visit more than one group from 9.0 to M. (Tickets for each group, 1s.)

G. Floor Cloth and Linoleum Manufacture.—Messrs. John Barry, Oslere, and Co., Lim., Kirkcaldy.

H. Printing, Colour Printing, and Bookbinding.—Messrs. Thomas Nelson and Sons, Parkside Works, Edinburgh.

Machinery Manufacture.—Messrs. Bertrams, Limited, St. Katharine's Works, Sciennes, Edinburgh.

Brewing.—Messrs. James and Thomas Usher, Park Brewery, St. Leonard's Hill, Edinburgh.

I. Brewing.—Messrs. Wm. McEwan and Co., Limited, Fountain Bridge, Edinburgh.

Distilling.—The North British Distillery Co., Limited, Gorgie Road, Edinburgh.

J. Collieries, Brick and Fireclay Manufacture.—The Lothian Coal Co., Lim., Rosewell.

K. Paraffin Oil and Shale Distillation.—The Broxburn Oil Co., Lim., Broxburn.

L. Flint Glass Manufacture.—Messrs. John Ford and Co., Holyrood Glass Works, Edinburgh.

Brewing.—Messrs. William Younger and Co., Lim., Abbey Brewery, Edinburgh.

Gas Manufacture.—Edinburgh Gas Works, Canon-gate, Edinburgh.

M. Ornamental Glass Manufacture.—Mr. A. D. Jenkinson, Abbeyhill, Edinburgh.

Disposal of Refuse.—Edinburgh Refuse Destructor, Powderhall, Edinburgh.

Shipbuilding.—Messrs. Ramage and Ferguson, Lim., Leith.

Docks.—Leith Docks. Peter Whyte, Esq., C.E., Superintendent.

N. 1.0 p.m. A steamer will leave the West Pier, Leith, for the Forth Bridge, sailing thence to Granton, where, by invitation of Messrs. A. B. Fleming and Co., a Garden Party will be held at Royston. The Society will have an opportunity of inspecting the manufacture of Lamp Black, Printing Inks, and Solidified Oils, at Caroline Park. The Marine Station will be open for inspection. The Members will return to Edinburgh by special train at 6 p.m.  
(Ticket, 2s. 6d.)

O. 7.30 p.m. The Annual Dinner will take place at the Waterloo Hotel, Edinburgh. Ticket (including light wines), 10s. 6d.

*Friday, July 20th.*

Excursions will leave the Waverley Station for—

P. Aberdeen, crossing the Forth and Tay Bridges.  
(Ticket, 10s.)

Q. Perth and St. Andrews. The Members will be conveyed, *via* the Forth Bridge, to St. Andrews, thence, *via* the Tay Bridge, to Dundee and by Steamer to Perth; returning from Perth by rail. (Ticket, 10s.)

R. 9.0 p.m.—Smoking Concert in the Waterloo Hotel.

The Headquarters of the Society during the Meeting will be in the Waterloo Hotel, where an Inquiry and Information Bureau will be open on the 17th, 18th, 19th, and 20th of July, for the issue of tickets and full particulars relating to the various items upon the programme. Payment for excursion tickets, &c., will only be received at the Treasurer's Office, Waterloo Hotel. Ladies will be able to take part in nearly all the above excursions and parties.

#### BANKERS' ORDERS.

For the convenience of Members, the Treasurer has arranged with the Bankers of the Society that they shall collect subscriptions from Bankers in town and country; and Members, who have not already done so, are invited to fill up and sign the Banker's Order enclosed with the December number of the Journal, which should then be sent to the Honorary Treasurer, Mr. E. Rider Cook, East London Soap Works, Bow, E.

Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 41 of the bye-laws, the Society has the right of priority of publication for three months of all such papers. Infringement of this bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.



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SESSION 1893-94.

*Meeting held Monday, May 21st, 1894.*

MR. WILLIAM THORP IN THE CHAIR.

## THE MANUFACTURE OF SMOKELESS POWDER.

BY OSCAR GUTMANN, ASSOC. M. INST. C.E., F.I.C.

The general interest created by the appearance of the so-called smokeless powder for military use, and their adoption by nearly every army in the world, together with the comparative ignorance in which the civilian public finds itself with regard to the nature and production of these powders, made me think that a brief summary of the most striking peculiarities of this rapidly increasing industry would be of interest.

Most of you will be aware that in 1888, the world was first startled by the appearance of newspaper reports about a new and smokeless powder invented by a chemist of the French Government, and that very soon after this the German Army had a similar one. It has since been stated that the first experiments in connexion with the discovery of smokeless powders were made in 1881, by Mr. Vieille, the well-known chemist of the French Government Gunpowder Factories. What this powder at that stage was is not quite clear, but it is not improbable that it consisted of a mixture of collodion cotton and picric acid, similar to the original basis for the much talked of melinite. This composition, however, seems to have been abandoned after a short period, and that kind of smokeless powder which is now very largely used in other countries as well as in France, to have been adopted.

In 1889, Alfred Nobel took out a patent for the manufacture of "Ballistite," which is an ingenious modification of his blasting gelatine. This, and the above-mentioned French powder are the two types upon which most of modern smokeless powders are based.

The first approach to a powder giving no smoke on combustion, and which at the same time, was not composed of the usual saltpetre, sulphur, and charcoal mixture, was, apart from the gun cotton used over 30 years ago, the Schultze powder, which has been used as a sporting powder in this country for more than 20 years, and which consisted of nitrated wood, prepared by the treatment of wood

with nitric acid, mixed with saltpetre or a similar substance. Although this powder has been brought to great perfection for sporting purposes, it has failed to be practicable for military use, since it can hardly be such a uniform material as is required for this purpose.

A much nearer approach to the modern smokeless powders, and, in fact, in some way indicative of them, was the E.C. powder, invented by Mr. Walter F. Reid, and patented in 1882 by Messrs. Reid and Johnson. Generally speaking, Reid formed grains of nitrocellulose by putting powdered nitro cotton into a barrel, sprinkling it over with water and revolving the barrel, whereby, through agglomeration, grains of various sizes were formed. These were dried and then moistened with ether-alcohol, which had the effect of gelatinizing the surface of the grains. A small addition of aurine gave the powder an orange colour. After being again dried, the grains were then put through a sieve in order to separate them, since they adhered slightly to each other through the gelatinizing process.

In a similar way Max. von Forster made cubical gun cotton powder by cutting cubes out of compressed gun cotton and dipping them into a solution of acetic ether which coated them externally with a thin skin of collodion. This was only used for filling shells. Later on Messrs. Judson and Horland made a smokeless powder called the J.H. powder, by a process similar to the E.C. powder process, the only difference being that the gun cotton grains were treated with a solution of camphor in benzoline, which on being evaporated, left some camphor behind. This powder did not remain long in the market.

It is a pity that Mr. Reid stopped at this stage of the manufacture, because he was very near making that class of smokeless powders which are now known as pure gun cotton powders; but in extenuation it may be said that at that time, the want of such a powder had not been clearly expressed, since there was then neither rifle nor projectile in existence which would have been suitable for the use of powders whose pressures and velocities are so much higher than those of the ordinary black powder.

It is due to the success of the long continued experiments of two Swiss experts, Major Rubin and Professor Hebler, who have advocated for more than 10 years the adoption of the small calibre rifle, that powder manufacturers have been forced to find powders suitable for the use of such weapons.

I remember very well how in the beginning of 1886, Professor Hebler showed me an experimental cartridge case made for his small calibre rifle, and asked me whether I could give him a pellet of compressed gun cotton which could be loaded into such a cartridge case, and which would be likely to give to his long cylindrical projectile the required velocity. I then pointed out to him that such a charge would be impossible on account of the sudden combustion and the very high pressure it would develop, and I offered to make him a small piece of blasting gelatine which would burn more in layers, and, therefore, probably suit his purpose better. The very suggestion of using blasting gelatine in a rifle was so much against all recognised ideas, that the matter was not further proceeded with; but after all, that rather hazardous suggestion of mine has turned out to be an idea in the right direction, although I had no further part in its development.

Modern smokeless powders can be divided into three classes. First those in which only gun cotton is used, whether it be the so-called insoluble or the so-called soluble variety. Secondly, those in which nitroglycerin is used in connection with soluble or insoluble nitrocellulose. Thirdly, those which contain nitrocellulose in connection with a nitro-derivative of some aromatic hydrocarbon.

There have also been devised some other smokeless powders containing nitrocellulose in connection with oxygen carriers and also some which consist of merely mechanical mixtures of oxygen carriers and carbonaceous matters, but none of them have yet found favour for service use.

I will just briefly indicate the composition of those smokeless powders which have hitherto been devised. In the first place, come the pure nitrocellulose powders, where the nitrocellulose is simply dissolved in some solvent and then made into flakes or grains. Such powders are the

French B. powders, the German smokeless powder, the Wettstein, the Wal-rode, von Forster's, and various others. The French Government, von Forster, and a few others are using a mixture of ether and alcohol as a solvent; the German factories, acetone. The nitrocellulose used is as a rule guncotton, although in some cases, wood nitrocellulose has been tried.

With the pure nitrocellulose powders can be classed the E.C. and the J.B. powders. The E.C. powder, now sold as No. 2, contains some camphor and is soaked throughout in ether-alcohol, whereby a harder grain results. To the second class belong powders made of nitroglycerin and nitrocellulose. There is first of all the Ballistite of Mr. Alfred Nobel, consisting of equal parts of nitroglycerin and collodion cotton, with an addition of 1 to 2 per cent. of aniline or diphenylamine. This Ballistite has with some modifications, been adopted in Italy, Austria, and for some guns in Germany. In Italy when made into cords, it is called Filite. To the same class belongs the Cordite, adopted by the British Government. This consists of 58 parts of nitroglycerin, 37 parts of the highest nitrated guncotton, and 5 parts of vaseline, which ingredients are dissolved in 19.2 parts of acetone.

Curtis and André made a powder consisting of 44 parts of tri-nitrocellulose, 12 parts of di-nitrocellulose, and 40 parts of nitroglycerin, with an addition of solid paraffin and shellac solution, which is formed into grains by means of a mixture of ether-alcohol. This powder is sold under the name of Amherite.

M. E. Leonard, of Manchester, in the United States, makes a powder of 150 parts of nitroglycerine, 50 parts of guncotton, 10 parts of lycopodium, and 4 parts of powdered urea crystals dissolved in acetone.

To the third class, namely, those containing nitrated aromatic hydrocarbons, belong the following powders:—The Indurite of Professor Charles Monroe, which is made from insoluble nitrocellulose and nitrobenzin. The Dupont powder of the Dupont Powder Company at Wilmington, U.S.A., which also consists of nitrocellulose and nitrobenzin granulated by a peculiar process.

There are also a large number of powders brought out by the Smokeless Powder Company of Warwick, under the name of Rifleite, S.S., S.P., S.K., S.V., and S.B. They are not patented and their composition is kept secret, but from information received from various sources, I believe that the Rifleite consists of soluble wood nitrocellulose dissolved in acetone and mixed with nitrobenzin and saltpetre, and granulated in a similar manner as the E.C. powder.

A very remarkable powder of this class is made by Hermann Guttler of Reichenstein, in Germany, which is made by dissolving nitrated wood cellulose in molten di-nitrotolual.

To the miscellaneous class of smokeless powders belong really only two powders which are both sold by the French Government for sporting purposes. One kind is called Poudre Pyroxylée, and it is composed as follows:—

Soluble guncotton	-	28 parts.
Insoluble guncotton	-	37 parts.
Barum nitrate	-	29 parts.
Potassium nitrate	-	6 parts.

Ether is used as a solvent with this powder. The other more recent one which has substituted for the Poudre Pyroxylée, is the so-called J. Powder. This is due to the Engineer Brunau, and consists of 83 parts of guncotton, and 17 parts of ammonium bichromate. The Nobel Company of Austria also proposed to make a smokeless powder consisting of 70 to 99 parts of nitro-starch, and 30 to 1 part of di-nitro-benzine, but it does not appear to have been adopted yet.

There were also various other powders proposed, such as that of Kaliwoda von Falkenstein, and that of Koli and others, but these propositions have apparently been made by people not sufficiently conversant with the requirements of a good service powder and they need not therefore be considered here.

One of the most important conditions in the preparation of smokeless powder, is the proper selection of prime materials. I do not intend to give an opinion as to the

relative value of the various powders and consequently of the constituents. Generally nitrocellulose has been selected as a chief ingredient, and from the many nitrocelluloses available, the nitrocotton, or guncotton, has been most favoured. There are besides the nitrocellulose, many nitro compounds known, which possess explosive properties, and give off no fumes, or very few, in burning, but nitrocellulose seems to have been selected, because it can be readily dissolved, and is well known, a solution can be mixed much more easily than a mechanical compound, and also because, after the solvent is evaporated, the nitrocellulose remaining can be shaped into various forms by easy mechanical means and without any danger.

The wood used for making nitrated wood cellulose was formerly, like the Scholtze powder, cut up into thin squares. In modern smokeless powders, the wood pulp from the sulphite cellulose, or soda processes of cellulose manufacture, such as is supplied for paper making has been used. This kind of cellulose is generally supplied from the factories in thin sheets, which are not very porous, have rather a glazed surface, and would have to be again reduced to pulp before it could be nitrated. A more convenient form, and at the same time, a very pure kind of cellulose has been made by the chemical factory of Waldhof. This cellulose resembles tissue paper, the difference being, that it is of looser structure, more like gauze, very porous and can be easily torn into small pieces by hand, so that it can be used direct for nitration. The wood cellulose has not yet been adopted by many factories for the reason that it does not seem to give such a tough powder as guncotton.

I believe that it is unnecessary nowadays to refer at length to the well known differences between soluble and insoluble guncotton. Suffice it to say that it is generally recognised that the term soluble nitrocellulose means that kind of nitrocellulose which is soluble in ether-alcohol, but that it is not always of the same composition, since the amount of nitrogen contained in the soluble nitrocellulose may vary up to 12.78 per cent., and also the insoluble nitrocellulose may contain from 12.78 up to 14.14 per cent. of nitrogen. This does not mean that the soluble nitrocellulose contains an admixture of what is known as hexa-, or insoluble, nitrocellulose. It may be a mixture of various kinds of soluble nitrocellulose, that is to say, of intermediate stages of nitration between mono and penta-nitrocellulose, but the whole of it must be soluble in ether-alcohol. At the same time, it is necessary that the nitrocellulose should comply with certain requirements laid down to adapt the powder made therefrom for the special purposes it is intended for.

Thus, for instance, certain powders will be made of a soluble nitrocellulose containing less nitrogen, and others from such containing the highest possible amount of nitrogen consistent with perfect solubility. As regards those powders, where only the highest or hexa-nitrocellulose enters into the composition, it is apparent to all who are acquainted with the manufacture of nitrocellulose, that it has been hitherto impossible to obtain nitrocellulose containing 14.14 per cent. of nitrogen, that is consisting entirely of hexa-nitrocellulose. Generally the guncotton, which is the most used form of hexa-nitrocellulose, contains about 12 per cent. soluble nitrocotton, but guncotton containing only 2 per cent. has been made by me on a large scale. In using hexa-nitrocellulose, one has therefore to be careful to regulate the amount of soluble nitrocellulose, which can be done either by blending or by using special means during the manufacture.

It was for sometime known and recently proved by Messrs. Nobel and Macnab, that by treatment at a temperature much below the freezing point of mercury, the so called insoluble nitrocellulose is soluble in ether-alcohol, but these are conditions which are only obtained quite exceptionally. It has also been found by Professor Odling, that by making special mixtures of nitric and sulphuric acids, it is quite possible to make two kinds of guncotton, both containing about the same amount of nitrogen, yet the one is soluble and the other insoluble in ether-alcohol. This has no other bearing upon the practical manufacture, than that of showing the manufacturer how to avoid obtaining such results, which would be contrary to his

intentions. What is generally aimed at, and what is nowadays quite possible, is to obtain nitrocellulose containing a definite amount of nitrogen and a suitable amount of solubility or insolubility.

Most military powders contain only the highest nitrocellulose, generally dissolved in acetie ether or acetone. In sporting powders, where less rapid action is desirable, soluble nitrocellulose is used, sometimes dissolved in a mixture of ether and alcohol.

Of the nitroglycerine used for the manufacture of smokeless powder, very little need be said, since nowadays there is no difficulty in producing a perfectly stable and in every respect suitable article. Of course it is not so easy as it would seem from the indication given in text books. It can only be done in factories conducted on sound scientific principles, and having large experience at their disposal.

A very important feature as to the final composition of the powder is the solvent used. Although its complete evaporation is almost invariably aimed at, yet small traces of it and especially such impurities as exist in the solvent, will remain in the powder. The nature of the solvent bears largely upon the structure and appearance of the dough prepared, and consequently, the finished powder may have a different density and a different surface, and thereby a different rate of combustion.

It is known that ether by absorption of moisture, becomes acid in time, and although on evaporation, the powder does not seem to be technically acid, yet if proper care is not taken, it may not stand the heat test as well as it ought to. Acetone is a comparatively new solvent, and of that made on a commercial scale, very little was known. For use with smokeless powders, it has to comply with a severe specification. A good serviceable acetone should be quite clear and miscible in all proportions with distilled water, without any precipitate forming. It should not have more than 0.005 per cent. of acidity, nor more than 0.1 per cent. of aldehyde. With Krammer's iometric test (transformation into iodoform by an excess of iodine solution in the presence of soda solution) it should show at least 98 per cent. of pure acetone and when treated with a 0.1 per cent. solution of permanganate of potash, it should retain its coloration for more than two minutes.

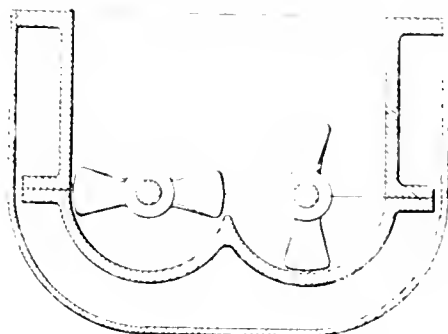
I have here a sample of acetone such as is used in very large quantities for military purposes. It has a specific gravity of 0.7965. 98 per cent. of this acetone distils over between 56.2° and 56.4° C. It stands the permanganate test for nine minutes and shows 0.00225 per cent. of acidity. An addition of alcohol to acetone has been tried and it seems to make the powder burn a little slower, but this can also be accomplished in other ways.

The nitrocellulose is of course dried at a temperature not exceeding 40° C. This is usually done in specially constructed drying houses to which I have referred in a former paper before this Society. I only wish to mention this on account of a peculiar process which was used in Austria as early as 1891 at least, but which was patented in this country in 1892. It consists in what they call the alcoholisation of the nitrocellulose, namely, adding alcohol of high percentage to the wet nitrocellulose, diluting thereby the alcohol, and then evaporating the diluted spirit. Since the boiling points of dissolved alcohol and moderately diluted alcohol are very nearly the same, and since both are very much lower than the boiling point of water, it is clear that the removal of the moisture is done much more rapidly in this way. In order to utilize the alcohol more fully, one can, as it has been suggested in France, use the alcohol in separate stages, that is to say, using first, diluted alcohol from former treatment, and after evaporating it, using the stronger one, which would have to deal with a smaller amount of water, and so on three or four times, whereby a considerable saving in alcohol would be effected.

I believe that in France, flat ebonite vessels were originally used, in which the guncotton was spread out in a thin layer, and the solvent poured over it. These vessels were then put under glass covers and allowed to stand until the solution was completed. Then a current of air heated to 55° C. was passed over it to evaporate the ether, and this was condensed in separate apparatus.

Nearly all the powders are made up by very simple processes. The solution of nitrocellulose in the solvent is effected by means of kneading machines such as are used by bakers for preparing their dough, and which have long been used in the manufacture of blasting gelatine. Those of Werner and Pfleiderer are almost exclusively used.

Fig. 1.



Their construction is shown in Fig. 1. It consists of a trough, composed of two halves of a cylinder joined together and surmounted by a square box, consequently the bottom is about the form of a *w*. In each of these cylinder segments is a shaft which carries a helical blade. The blades revolve in opposite directions, and the one makes about half the number of revolutions of the other. The blades very nearly touch the bottom of the trough, and the consequence is, that any material brought into the machine is divided into two parts, then kneaded against the bottom of the cylinders, then pushed along the blade, and by the next half revolution, turned over by the other part of the helix; and since the velocity of the two blades is different, there will be with every revolution, a different part of the dough submitted to the kneading operation.

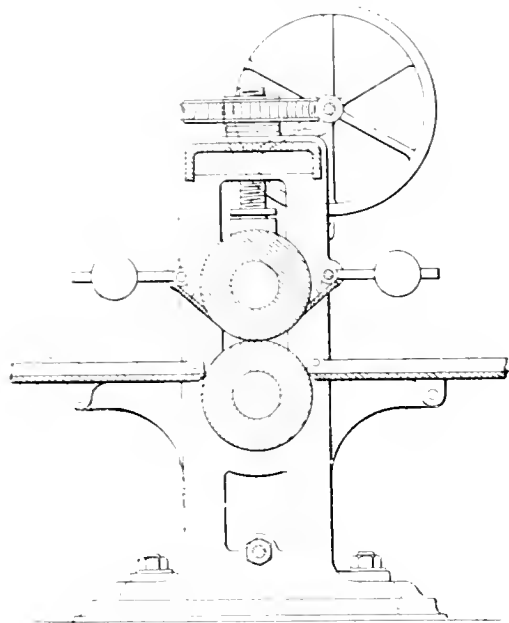
As a rule, the mixture of smokeless powder, when once the solvent is introduced, ceases to be liable to explosion, and is only combustible, so that these kneading machines are usually made from iron only. Since guncotton has a very high absorbing power, the amount of solvent used is about weight by weight the same, but this varies according to the length of time given to the kneading operation. It is quite possible to work with a small amount of solvent provided the kneading of the mixture is prolonged sufficiently. The question as to whether it is more advisable to use a minimum amount of solvent, or to take a minimum length of time over the operation, has to be decided from economic consideration, since hitherto it has been found impossible to recover the solvent economically. When the dough leaves the machine, in which it has been kneaded from three to ten hours, it has a perfectly uniform and translucent appearance, and has about the consistency of soft india-rubber. It then undergoes a further treatment according to the form which the finished powder is ultimately to have.

Some of the pure guncotton powders, like the Walsrode, are formed into grains by placing the mixture in hot water and blowing steam into it, which causes the dough to break up and become granular. Some are pressed through dies into cords, like the Cordite, of which mention will be made later on, but as a rule, for military purposes, the dough is passed between heated rollers and rolled out into thin sheets, the solvent being simultaneously driven off by the heat from the rollers. The general principle of such rollers is shown in Fig. 2. They consist simply of a pair of hollow highly polished hard cast-iron or steel rollers, the lower one of which rotates in a fixed bearing, whilst the upper one can be elevated by means of gearing actuated by a hand wheel. Two scrapers are placed against the rollers to prevent the rolled out sheet from doubling up against the rollers and being carried round by them.

The temperature maintained in these rollers depends upon the boiling point of the solvent used, but it does

not exceed, as a rule,  $60^{\circ}\text{C}$ . These rollers effect at the same time the thorough mixing and solution of any particles of nitrocellulose that may have escaped solution in the kneading operation. During the rolling there are occa-

Fig. 2.



sionally small detonations heard, which were by some attributed to the bursting of air bubbles in the sheet, but are most likely due to some particles of gun-cotton exploding by combined heat, friction, and pressure, which is proved by local burning marks. Such explosions do not spread and are harmless. When the rolling out of the dough into a thin sheet of the required thickness has been effected, it is taken to a cutting machine, which cuts it up into small squares or flakes of the required diameter. This cutting machine is shown in fig. 3. It consists of a number of circular steel knives set on a shaft at a suitable distance apart, by means of distance washers; two such shafts stand opposite each other in such a position that the knives overlap slightly. There is a comb or grating fixed between

Fig. 3.

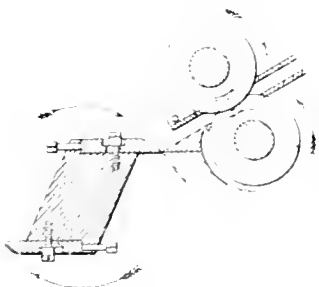


Fig. 4.



Fig. 5.



the two sets of knives so as to take out the strip from the knife immediately it has been cut, and to prevent it being carried round and choking the cutter. By putting a sheet of powder between the two sets of knives, a number of strips are cut, which when they leave the circular cutters pass over a fixed longitudinal knife-edge, in front of which are two or four longitudinal knives, carried on a revolving shaft, which cut up the strips into flakes. The length of a flake depends upon the velocity with which the strips

pass from the knives and on the rate at which the chipping knives revolve. The velocity is regulated by means of cog-wheels, and as a rule, the chipping knife is driven direct from the belt, and the cutting knives from this. The knives used for cutting strips were originally made, as shown in Fig. 4 with a small bevelled edge, but this was found very inconvenient on account of the machine being easily choked. Nowadays, the form of the edge is generally that of a *Λ* as shown in Fig. 5, a form of knives which has long been in use for the purpose of cutting up Bristol board for playing cards.

In case the powder should have the form of cubes instead of thin flakes, the former are made by cementing together several sheets of powder. It would not do to make from the outset a thick sheet, because it would contain then a too large quantity of solvent, a great many air bubbles, and the mixture would probably not be thoroughly made. It is therefore better to conduct the whole operation by rolling out the mixture into thin sheets, and as there is a sufficient amount of the solvent left in them, cementing them into a thicker sheet by simply running several sheets together through the corresponding wider spaced rolls. Such cubes are perfectly translucent, and if cut normally to the surface, do not show the way how they are made, but if they are cut in an angle, the lines of division can be plainly seen. The powder after the rolling and cutting operations still contains a small amount of the solvent, which on account of the homogeneous and tough consistency of the powder, takes a good deal of time to entirely evaporate. It is therefore treated in drying houses, sometimes for more than a week, unless it is specially desired to retain a certain small percentage of the solvent. In very rare cases, it has been thought desirable to make round discs instead of square ones, and the machine adapted to that purpose, is similar to that used for making certain kinds of pastry, namely, passing the dough through a die and allowing a knife to revolve rapidly against the cord issuing from the die, thereby cutting it into fine discs.

With Nobel's Ballistite, which contains a mixture of nitroglycerine and soluble gun-cotton, it was originally intended to absorb the nitroglycerine by the collodion cotton in a vacuum vessel, then to press out the excess of nitroglycerine, and to then heat the remainder of the mixture in order to dissolve the collodion-cotton.

Later on, Messrs. Lundholm and Sayers devised a process by which the solution of nitroglycerine and nitrocellulose can be readily made without such complicated means. It is based upon the curious fact that, although gun-cotton containing a small amount of water is soluble only with difficulty in nitroglycerine, such gun-cotton is readily soluble when suspended with the nitroglycerine in a large quantity of water. For this purpose the nitroglycerine and collodion cotton are put into a vessel containing hot water and stirred by means of air, or steam, whereby the incorporation of nitroglycerine and collodion cotton takes place; but it is also sufficient to maintain the water at a temperature of about  $60^{\circ}$ , and to let the mixture stand for several days, stirring it from time to time. When the gelatinisation is completed the mixture is first submitted to pressure in order to remove the largest part of the water, and then formed into sheets under heated rollers, and finally cut and dried in the usual way. In Italy they form cords like those of Cordite, and call the powder Filite.

As it is known that nitroglycerine alone will not dissolve the highest nitrated cellulose by ordinary means, collodion cotton is used in the manufacture of Ballistite. Sir Frederick Abel and Professor James Dewar found that they could make a perfect combination of gun-cotton and nitroglycerine by dissolving them both in a common solvent. The peculiarity in this process is, that although one would imagine that on evaporating the solution the two constituents would separate, since the one is not soluble in the other under ordinary circumstances, yet the two remain in a perfect combination, which has quite the appearance of the solution effected in the case of nitroglycerine and collodion cotton. It has been claimed by Abel and Dewar that they are not in solution as a matter of fact, but are existing side by side.

In the manufacture of Cordite the gun-cotton and nitro-glycerine, together with a suitable amount of acetone, which is used as a solvent, are placed into the kneading machine and worked for  $3\frac{1}{2}$  hours, when the mass has a perfect

dough-like appearance. At this stage a small quantity of vaseline is added and the dough worked another  $3\frac{1}{2}$  hours, when the combination is considered to be perfect. During the kneading operation care is taken to prevent the escape

Fig. 6.

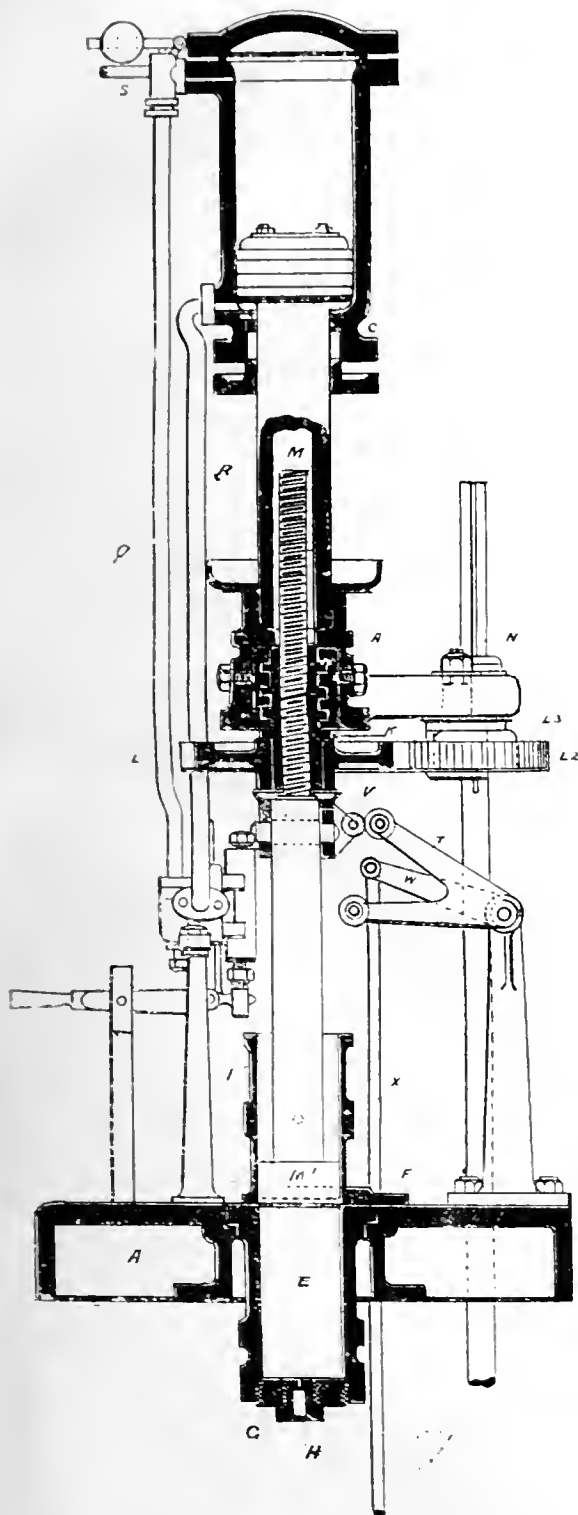
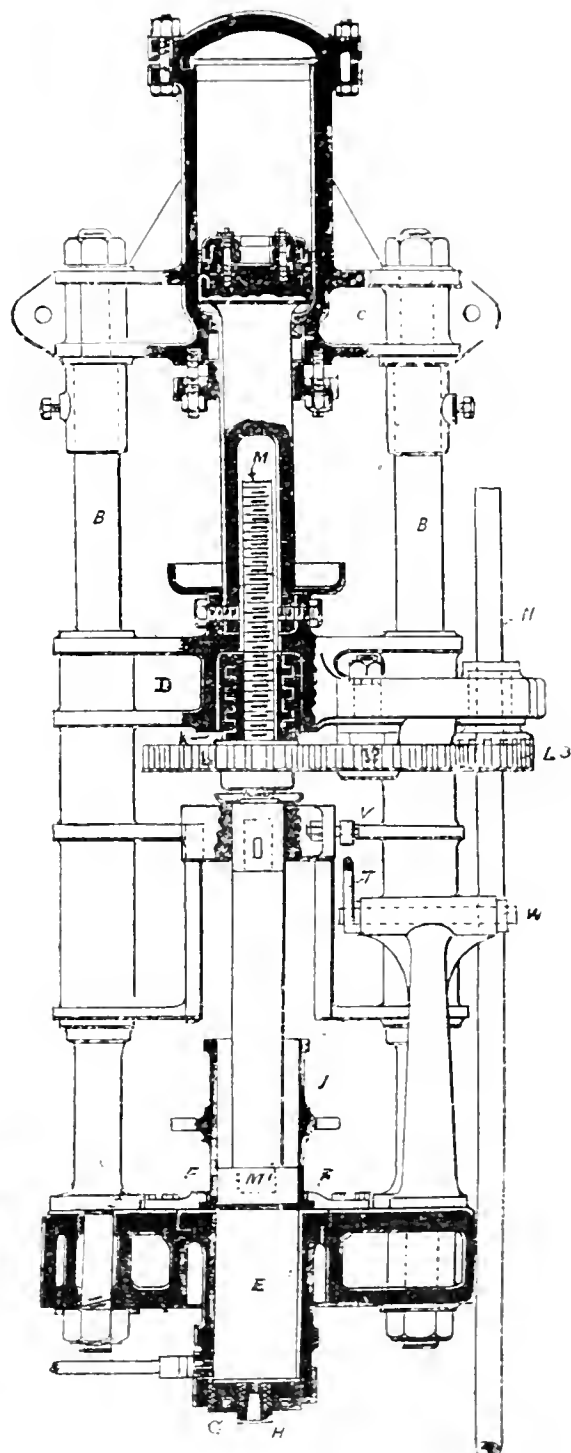


Fig. 7.





of the solvent, and by means of a water-cooling jacket the heat generated during the kneading is reduced so as to prevent the evaporation of the solvent. The dough is then brought into machines which squirt it through dies into the form of threads or cords.

One of the machines is shown in Figs. 6 and 7. It consists of a base-plate A and two columns BB, connected by a head C; between the two columns a cross-head D slides up and down. The mould E is contained in the base-plate A, and held in tight by clamps F; at the bottom of the mould E is a cover G, with a nozzle H made to correspond to the thickness of the cord required. Another mould I, which contains the dough, is put on the top of the mould E. In the cross head there is a nut K which can turn, and which is fixed into the base of the cogwheel L. Into this goes a screw M, having fixed to it, by means of a cutter, the piston N. The cogwheel is made to turn by means of an intermediate cogwheel P, 2 and a driving-wheel Q. 3. On the shaft of the cogwheel L 3 there is a long feather key N, and if, therefore, the cross head rises for some reason or the other, the whole mechanism slides up on the feather without ceasing to revolve. It will be seen, therefore, that on being rotated by the cogwheel, the nut, the screw, and with it the piston, must gradually descend into the mould and press out the mass through the nozzle in the form of a thin cord. In order to prevent the pressure in the mould becoming too great from any cause—such as the die getting blocked by any foreign matter—there is an hydraulic arrangement provided. The cross-head is screwed fast with a hydraulic piston, in which the screw can work up and down freely; this piston works in a cylinder which forms parts of the head of the press. Hydraulic pressure is applied on both sides of the piston by means of the pipes Q and R. Since the free surface of the piston is smaller on the lower part than on the upper one there is always a pressure acting on the piston corresponding to the difference of the two areas. The amount of pressure is regulated by means of the safety-valve S, and should it exceed a certain limit the water flows out through the safety-valve, and the cross-head and piston ascend, when the whole of the contents of the mould are pressed out; the lever T is caught by a tappet V. With this lever is connected another lever W, and as T is moved, the lever W either lifts or presses down, by means of a rod X, a counter weight, with the result that one driving-belt is thrown on to the loose pulley and the other on to the fast pulley, and the motion of the piston reversed; in the same way the machine can be brought to a standstill altogether.

The cord issuing from the press is reeled, in a similar manner to cotton spools, on to drums made of sheet metal stampings. A number of these reels are re-reeled on to a large drum, and several large drums on a larger one, so as to obtain a uniform blending. The cord is then brought to a machine, where it is pushed several strands at a time into the cartridge and cut off at the proper length. With Cordite of a larger diameter, the cord is cut immediately after leaving the die into lengths of about 12 inches.

In the case of Amherite, the manufacture is somewhat different. First of all, grains are formed from the nitrocellulose, probably in a similar way as it is done with the E.C. powder, and are subsequently treated with a solvent which dissolves the soluble nitrocellulose only, for instance, with sulphuric ether and alcohol. In this way, there is a kind of cement formed within the mass of insoluble guncotton, which binds the grains thoroughly together and yet leaves the guncotton unaltered. There is in this case, therefore, not only the hardening of the surface as in the case of the E.C. powder, but the formation of a kind of conglomerate throughout. This, of course, diminishes the rapidity of combustion of the powder a good deal. In order to arrive at a proper proportion of insoluble and soluble nitrocellulose, the patentees add the required proportion of soluble guncotton to the ordinary guncotton of commerce, which usually contains already about 12 per cent. of the soluble variety.

In the case of the Leonard powder, the materials are simply mixed with acetone and left standing for 12 hours in tightly closed pots, after which the solvent is evaporated, and the mass granulated in the usual way.

Professor C. E. Munroe proposes for his smokeless powder, which he calls Indurite, to use very pure guncotton, which he obtains by treating the ordinary guncotton in a kind of extracting apparatus with repeated infusions of methylic alcohol, until all the soluble nitrocellulose is eliminated. The remaining hexa-nitrocellulose is dissolved in nitrobenzin. He then rolls the mass out into sheets and cuts it into grains, and the resulting rather soft flakes are treated with hot water and steam as is the case with the Waisrode powder, whereby the grains harden externally. He calls this latter process the induration. I believe that as nitrobenzin is highly volatile at comparatively low temperatures, all that takes place in this induration, is simply a partial evaporation of nitrobenzin on the surface, which has the effect of leaving the guncotton bare and hard.

The Dupont powder, in which nitrobenzin and nitrocellulose also enter into combination, is made by a somewhat similar process to that of Lundholm and Sayers. The only difference is that a special apparatus with rotating paddles is used, whereby the mass is first made into a dough, and then on continued stirring, breaks up into grains. By admitting steam into the apparatus, the grains, which at first have a soft, putty-like appearance, become hard and consistent. When the grains are finished, they are placed in a rotating barrel, into which steam and water are admitted. This has the effect of making the grains round, and carrying away any excess of solvent.

The French Poudre Pyroxylée is produced by a rather complicated process. The materials are first mixed by hand, and then in incorporating mills for 45 minutes, where an addition of 40 per cent. of water is made. After this, they are rubbed through a sieve of about  $\frac{1}{16}$  of an inch mesh, dried up to 1 per cent. of moisture, and 65 per cent. of ether added to the mixture. The putty so obtained is rubbed through a perforated zinc sieve with holes about  $\frac{1}{16}$  of an inch diameter. The mixtures of grains and dust thus produced is revolved in a wooden drum for 45 minutes, then moistened with 50 per cent. of water and afterwards dried. The grains are sorted. Those between 1.6 and 1 mm. diameter being moistened with 15 per cent. of ether and polished in a copper drum. The powder is then again rubbed through a sieve of about  $\frac{1}{16}$ -inch mesh, and again sorted into grains between 1.6 and 1 mm. The residue is treated in a similar way in order to again get grains of suitable size, and the final powder consists of one part of grains of the original make, and two parts of those coming from the working up of the residue. The Poudre J is probably made in a similar way.

The properties of smokeless powders vary very much according to the composition, the mode of manufacture, and also according to the object for which they are to be used. For large guns, a powder is required which burns very slowly, in order that the force should be developed gradually so that the energy imparted to the projectile should reach its maximum as the projectile is just about to leave the gun. For sporting powders on the other hand, a quick combustion is desirable in order to impart to the shot sufficient penetration and little spreading, or what is called, a good pattern. At the same time, the powder should develop as small a gas pressure as possible and a very high muzzle velocity. For military purposes it is desirable that there should be as much force as possible given off by unit weight of the powder, because this enables rifles of small calibre to be used, and it also allows the soldier to carry a large number of cartridges. For sporting powder on the other hand, it is not possible to change the type of the guns in use as rapidly as it can be done by the aid of a national exchequer with Service rifles, and the sportsman prefers a powder that he can use in the same cartridge as black powder. Considering then that an average charge of black powder for a 0.500 express rifle, is about 138 grains of Curtis and Harvey's No. 6 black powder, whereas of a good smokeless powder, only 47 grains are needed, it is easy to see that with the smokeless powders, a large space of the cartridge will remain empty, although the volumetric density of the smokeless powder is much smaller than that of black powder. For sporting purposes, therefore, such a powder whose volumetric density is the smallest,

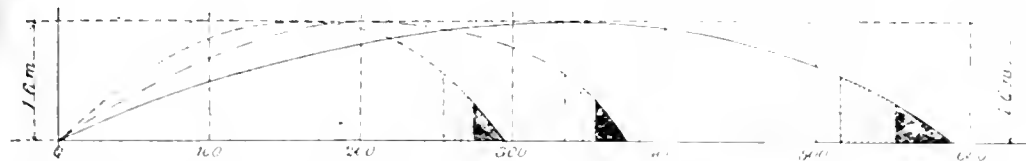
and which is consequently most likely to fill the whole of the cartridge case, possesses some advantage.

This brings forward another question which has for some time given a good deal of trouble with smokeless powders, namely, the loading of cartridges. With black powder, a slight increase of the charge meant much less than with smokeless powders. The latter are very liable to set up extraordinary pressures when the charge increases, and with some of them it has been found impossible to take more than  $1\frac{1}{2}$  times the charge for fear of bursting the gun. On account of the flaky nature of some of the powders, the ordinary machines for loading cartridges, where the powder

runs through a funnel in a measured vessel, could not well be used, because the powder settles differently and occasionally there is an increase in the weight of as much as 10 per cent. These obstacles have, however, been overcome with most of the powders.

One of the peculiarities of a small bore rifle is, that the smaller the diameter of the projectile becomes, the straighter will the flight of the shot be, because of the diminution of the air resistance. This of course has led to the adoption of very long projectiles, and in judging a powder, the straightness of course of the projectile, will have to be considered. An idea of the diminution of the height of flight can be

Fig. 8.



gathered from Fig. 8, in which there are represented the path of projectiles from the old Prussian 13 mm. needle gun of 1862, the French 11 mm. Gras rifle of 1874, and the 6.5 mm. Mannlicher rifle of 1892. It will be seen that a man of average height, say 1.6 m., or about 5 ft. 3 in., is liable to be hit at any distance up to 295 m. only with a needle gun, up to 375 m. with a Gras rifle, and up to 530 m. with the Mannlicher rifle. There is a corresponding increase for lower lying objects, as will be seen by the plain and lined parts of the diagram which represent objects of 0.4 and 0.8 m. height.

The absence of smoke in a powder has become a great consideration now, but it can be said that almost every smokeless powder produces only steam and gas without colour. The sole difference is to be found in the constitution of the gas, which in some powders may contain free nitrous acid, whereas with others, a perfect combustion takes place. At one time it was thought that armies could not long stand the fire of smokeless powder on a battle field, because of the penetrating "chemical" smell evolved, but this has been almost entirely overcome now. The marked difference in the colour of the products of combustion from ordinary black powder, and those from the new smokeless powders I shall endeavour to show to you by a method suggested by my assistant, Mr. Pollitt. It consists in burning small portions of the various powders on a tray and throwing shadows of their products of combustion on to a lantern screen.

Very important is the question of residue and heat developed. Some powders only leave a slightly imperceptible residue which is cleaned out automatically by the next shot. The heat evolved in connection with the friction caused by the residue erodes the rifling of the gun, and how delicate a mechanism rifling is, will be understood when I say that the depth of the rifling in the Lee-Metford rifle is only 0.004 inch.

A good powder should furthermore be perfectly stable under varying temperatures and should not suffer by storage in moist climates or damp stores. At the same time it is highly desirable that the powder should not be liable to explosion when struck by bullets, and that loaded cartridges should not explode "en masse" when carried in an ammunition cart.

From all this, it will be seen, that the manufacture of a good smokeless powder requires a large amount of skill and experience, and that however well devised the composition of a powder may be, it is liable to be unsuitable on account of its not fulfilling one or the other of the numerous conditions imposed upon it.

The form of smokeless powder now used is either that of grains, small flakes, cubes, or of cords. The grains vary in size. In grained powders there are between two thousand and three thousand grains per gramme. The flakes for military purposes are  $1\frac{1}{2}$  m. square, and 0.3 mm. thick. For sporting powder the thickness is reduced to 0.1 mm. The thinner the powder flakes are, the quicker they will burn

For large guns the flakes are made 3 mm. square by 0.7 mm. and upwards, or they are made in cubes of 2, 5, 10 and 15 mm. side.

Cordite and Filite are made in various thicknesses. That for the service rifle is 3.80 (0.0375) inch thick, and its diameter increases with the size of the gun for which it is used.

The colour of smokeless powder is, as a rule, in consequence of the action of the solvent, rather a dirty grey or yellow; with nitroglycerin powders, from light to dark brown. Very often the powders are polished with black-lead in order to fill up the pores and to give them a smooth surface, in which case, they have a silvery grey or black appearance.

The absorption of moisture by a good smokeless powder is very small, and if it should become damp, it can be easily dried again. An experiment made by exposing Ballistite in open saucers for a whole year has shown that most of the powders contained less moisture at the end of than before exposure.

The volumetric density of smokeless powders varies between 0.55 and 0.40. The absolute specific gravity is about 1.60 for most of them.

In this country and in most others, smokeless powders are tested for their stability against heat in a similar way to gun-cotton. But they are also tested as to the increase of gas pressure resulting from an increase of temperature in the barrel. According to an experiment made by Sir Andrew Noble with a 4.7 inch quick firing gun, Ballistite developed a mean pressure of 14.3 tons per square inch, or 2.180 atmospheres. Cordite, 14.3 tons per square inch, or 2.127 atmospheres, whereas the ordinary service pebble powder gave rise to 15.9 tons per square inch, or 2.024 atmospheres. The muzzle velocity was 2.140 ft.-seconds for ballistite; 2.140 ft.-seconds for the cordite, and 1.839 ft.-seconds for the pebble powder.

Smokeless powders are, as a rule, not explosive in the ordinary sense of the word. They burn with some difficulty and they are also fairly insensible to blows or the impact of bullets. The consequence is that they require caps of great strength, and developing much heat. They generally give off permanent gases, mostly carbonic acid and steam, but there are also some nitrogen-oxygen gases formed, which impart a slight yellow tinge and a little pungent smell to the smoke. On account of these acid compounds developed, there are always traces of acid residue left in the barrel, and unless these are cleaned out after ceasing to fire, the barrel becomes rusty.

The chemical analysis of smokeless powders is done on similar lines as that of blasting gelatine, but it is necessary to grind them in a mill in order to reduce the very hard grains to a fine powder. This grinding sometimes causes particles of iron and steel to fall into the ground powder, which have to be taken out by means of a magnet.

What I have said here, must of necessity have been of a somewhat superficial character, because on the one hand, the various Governments and manufacturers do not as a rule willingly divulge the details of their manufacture, and the knowledge to be acquired is therefore somewhat circumstantial. It is thus quite possible that not everything I told you is absolutely accurate, yet I have given you what I believe to be correct. Of course, I have not said all I could say on the subject, because this is not desirable for various reasons. All I wanted to do was to give you a general idea, to show how smokeless powders are constituted and made, and to enable you to follow this question a little more closely in the future. One thing I wish to impress upon you is, that with a comparatively new manufacture dating back only six years, it is quite impossible to have acquired all the experience necessary for the production of a powder faultless in every respect. There must, therefore, be of necessity a number of complaints at the beginning, and a large number of defects have been found in each powder in each country. In this empire especially, which extends almost from the north pole to the south pole, where there are extreme varieties of climate, temperature and moisture, the manufacture of a powder fully up to the requirements of the service, is extremely difficult, yet up to the present, it has been found possible to meet most of those defects as they showed themselves in course of time, and I have no doubt, that if the necessity for any further improvements become evident, it will be quite possible to effect them.

#### DISCUSSION.

Mr. W. F. REID said that the whole manufacture of powder for the army had become a chemical process, and on listening to the formulae that had been mentioned, they were almost transported into the benzene ring. With regard to Schultze's powder not being adapted to military purposes, that was partly due to the nature of the ash. Both that and the E.C. powder left a hard ash, whilst ordinary black powder left an ash to a certain extent soft, which afforded some lubrication to the succeeding bullets. With a hard ash the rifling was soon obstructed, the bullets stripped, and all accuracy was lost. The author had referred to him in connection with the E.C. powder, which he described as being the first powder which, if not quite smokeless, yet approached that condition, and also to his having been the first to harden the grain, by means of the action of a solvent upon the gun-cotton contained in the grain. That was the theory he had before him in working at that powder. Mr. Guttman said it was a pity he had stopped at that stage, but as a matter of fact he did not stop there, for he made a number of further experiments on gun-cotton powders, but there was a very good reason for not issuing them at that time. The use of powder for sporting guns was limited entirely to the cartridges then in use as it still was practically, and for military purposes it was quite impossible then, and still was for any English inventor to introduce any powder into the army in this country. The usual way was to bring them before a foreign government, and then that acted on public opinion, public opinion acted on the Government, and then something was done. That was the case with the smokeless powder. In 1882 he had occasion to show some French Military Attachés some of those experiments, and two years afterwards they were in full swing, experimenting on the same subject in France. The English Government took somewhat longer to get to work on the subject. In 1882 he took some specimens of smokeless powder to Waltham Abbey, and they were tried, and two points in particular were raised as being great objections, though there were others also; one was that the trajectory was so much flatter that the sights of all the rifles would have to be altered. Another was that the amount of power which could be concentrated in a given space would necessitate an alteration in the cartridges; this would involve an alteration in the whole of the rifles, and of course nothing was done. Then when the French Government took up smokeless powders, the question was brought in a very prominent way before Parliament, and before the Military Authorities. One of

the most important points in the manufacture of smokeless powders alluded to in the paper was the alleged impossibility of recovering the solvent economically, and it was said that hitherto that had not been done on a large scale. It was done on the commercial scale with the E.C. powder, in fact it was one of the first questions which occupied his attention. The solvent, which was a composition of ether-alcohol, was very expensive, and a great number of experiments were made with the object of recovering it. This was finally done in a simple manner. The apparatus consisted of a revolving drum heated externally, and a current of warm air passed through it. A very slight current was necessary, and then the air containing the solvent was cooled down, and the solvent recovered the greater portion of it, at any rate. That could be re-distilled, and used over again. As a matter of fact, this point was of great commercial importance in the manufacture of these powders, and he had no doubt that in most cases the solvent used could be recovered at a nominal cost. With regard to acetone, there was a good opening for any member of the Society who could find some means of producing it cheaply. At present it was a very expensive substance, but the demand for it was increasing rapidly. Theoretically it was possible to produce it from a variety of substances, such as sugar, and he had made some experiments in that direction, but it was not a practical process at present. He was quite sure there was a great future for chemical industry in that direction. Another point which he did not think had been sufficiently emphasised in the paper, though it must have impressed itself upon all who were present, was the great safety with which these powders could be handled. As a matter of fact they were extremely safe. If the largest cube on the table were set fire to it would not burn more violently than a small quantity of, say, petroleum, and it would not explode as black powder did. That was of great importance in handling these explosives. With regard to the general use of these powders, so far as his present knowledge went, those which contained nitro-glycerine were being rapidly superseded by those which consisted of nitrocellulose alone. So far smokeless powders had proved successful, and, as the author had remarked, they had reached a certain stage, but they hoped to do better. He thought the process of the survival of the fittest would result in the use of nitrocellulose alone. With regard to the lengthening of the projectile it was only possible to elongate it and to diminish the diameter within certain limits. Beyond a certain point such an amount of friction was produced in the rifle that the pressures became too great, and there were other theoretical reasons with regard to the flight of the projectile which prevented elongation beyond a certain extent. He believed also that doctors rather objected to a long narrow projectile. He heard of one military surgeon having stated that it was like sending a knitting needle through a man; it did not dislodge him at the moment, but it was a very difficult wound to cure. These small-bore bullets would also go through about a dozen men, one after the other. The amount of smoke developed could not be quite gauged by the appearance when the powders were ignited in an open space; it was much less when ignited in a confined space, because substances which in the open air were condensed in the atmosphere as soon as they touched cooler air became volatilised in a hot confined chamber, and did not show. These powders had a great advantage over the old black powders in respect of storage. They could be stored under water, and when taken up and dried were as efficient as ever. In 1882 he sent a sample of E.C. powder to a yachtman who was out wild fowl shooting, his boat sank, and he related with great enthusiasm that he got up the powder, boiled it, and then baked it, and it was much better than it was before; he even advised baking all his powder because he said he got so much better force with it. That showed the great advantages which modern military men had, that they need no longer keep their powder dry according to the old Cromwellian maxim. The climax of propulsive power he thought had almost been reached with smokeless powders. Theoretically the maximum effect would be produced if one could get solid hydrogen in connection with solid oxygen and detonate them together. At

the point which had now been reached they were obliged to take in some inert nitrogen also, but if they could eliminate the nitrogen and leave the other substances in a state so that they could at once be converted into gas, of course matters would be improved, but theoretically he did not think there were any much greater advances possible in this direction. The progress which would yet be made no doubt was probably in their shape or the form of the grain. One method of propulsion, though one could hardly call it a smokeless powder, was that by means of liquified or concentrated gas, and some experiments were made not long ago by M. Giffard which were not quite successful from a practical point of view, but they showed a way in which progress might in future be made. The method of cutting up the sheets of nitrocellulose after they had been hardened to form a powder was, he thought, open to very grave objection theoretically. If you could get a globular form of grain you would certainly have a better powder theoretically in every way. With flat grains there was the disadvantage of their setting or caking together, and you could never be quite sure that you had exactly the same spaces between the grains. Next to the globular form, no doubt the cylindrical was the best, but that also had a disadvantage from the point of view of ignition. On the question of ignition again the caps were of very great importance, and in this respect these powders were very different from the ordinary black powders. They required a certain definite amount of ignition to produce a definite effect, and unless the caps were very regular you got irregular results with powder of exactly the same composition, and used in the same quantity. He discovered that in a very startling way by some foreign cases containing very strong caps which were introduced for nitro-compounds for sporting purposes. The result was a number of burst-guns and accidents to sportsmen. It was very important that the method of ignition should be uniform, and in the ordinary caps this was not so. With regard to the manufacture of these powders, unfortunately as they had seen recently in the Government factories many accidents had happened, but it was rather curious that those accidents did not happen to such an extent in the private factories. There were many things which caused danger in a new explosive of this kind, and he might mention one which he found himself of very great importance, and in erecting smokeless powder factories he had always provided for it. That was electricity. One effect of covering the grains with graphite or plumbago was that it formed a conducting surface. If you shook these nitrocellulose sheets together, if they were not blacklead, they became highly electric and adhered to each other, and to everything. In fact they would often adhere to your fingers after having shaken them up for a bit. If they were faced with graphite that did not happen. Now in the Government factories it was the rule to exclude all metal from the interior of the drying sheds. But you were bound to have metal in some form in the shape of pipes and fittings, and if you covered these, which was often done, what you really produced was a Leyden jar which became charged with electricity in a warm house when the powder was moved or a current of air set in motion, in fact such a drying house was frequently a laden Leyden jar, and a spark might be produced which might cause a serious accident. The practice he followed was always to have metallic connection everywhere. Even in the treatment of dry nitroglycerine a considerable quantity of electricity was produced which he had been able to detect.

Mr. W. P. BLOXAM said the author had not made any reference to the use of smokeless powder in Her Majesty's navy, which he thought was a slight oversight, as they were greatly interested at present in the issue of this smokeless powder and its stability. Of course it would be very difficult to obtain any official utterances to the stability of cordite, and such like materials after exposure to high temperatures, but there was very distinct evidence of the high temperature of the magazines in many modern warships, and the seamen gunners and the gunnery officers were considerably exercised in their minds as to what would be the effect of the prolonged storage of cordite in the magazines of some of these ships. Again, if he followed the paper correctly,

the author did not refer to carbon monoxide, but he believed from certain statements that there was as much as 38 per cent. of carbon monoxide recognised in the products of combustion, and the seaman gunner again was not looking forward with any great pleasure to the use in closed turrets, of a material which developed such a quantity of this gas. Comparatively little, from a physiological point of view, was known of the toxic effect of this gas, but it was stated variously that from 0.1 to 1 per cent. proved rapidly fatal. He wished to know, therefore, if they were likely to have 38 per cent. It was very difficult, as Mr. Gottmann said, to get much information. They could not expect the powder to grow up in six years, and do everything it ought to do, and they must not be impatient in trying to force the hands of those gentlemen who were carrying on patient researches towards perfection, but still though the sailor was a confident man, he would not swallow everything. It was not so much a question of the strength of poor Jack as the persistent nature of carbon monoxide. Of course everything could not be stated in so short a period, but he was bound to say, being associated with naval gunnery officers, that it was of great importance to hear such an able and concise summary classification of smokeless powders, so lucidly presented.

Mr. W. MEXAN said he only had one or two remarks to make, not so much on the manufacture of smokeless powder as on one or two points which had been raised incidentally. With regard to the explosive, rifleite, he said that he had never found any alkaline or alkaline earthy nitrates in specimens he had examined, the components being nitrocellulose and nitrobenzene. With regard to the production of thick sheets of smokeless powder, no doubt it was quite correct when made with acetone or similar solvents, in order to make a thick sheet, to run several thinner sheets together, but in making the ordinary ballistite, consisting of nitroglycerin and nitrocellulose, in which no facilitating solvent is used, the sheets could be readily rolled up to about 1½ inch at once. With regard to what the last speaker said about the gas produced by the explosion, he could entirely confirm his opinion that there was carbon monoxide produced from cordite, ballistite, or, in fact, from nitrocellulose powders, to a large extent, sometimes amounting to 30 per cent. or 40 per cent. of the permanent gas. The more nitroglycerin there was present the less was the percentage of carbonic oxide, and the larger the percentage of the less harmful carbonic acid. One point which might be of some little interest was with regard to the effect of gelatinisation on nitrocellulose. He had examined nitrocellulose (gun cotton) as to the amount of heat evolved on explosion by a good sample of it containing 13.30 per cent. of nitrogen, and the result was that 1,061 calories were developed, but the same after gelatinisation, and being cut into grains yielded 922.

Mr. GETTMANN, in reply, said Mr. Reid said that the Schultze and E.C. powder gave a very hard ash, which was one of the chief reasons they could not be used for military purposes. No doubt that was one reason, but there was also the other reason that he gave in the paper, that they could not be made of such a degree of uniformity as to fulfil the very strict requirements of military purposes. As to the recovery of the solvent he simply stated that it was found impossible to recover it economically. Mr. Reid told them that they tried to do so in a revolving drum, passing heated air through it, then collecting the gas and condensing it. That was precisely the way in which it was tried by various governments, and in various countries, but found impracticable for the simple reason that when you had to deal with very large quantities, much larger than the E.C. Company had, you wanted such a large amount of drying drums and such a large quantity of air to pass over the powder in order to evaporate the solvent perfectly and absolutely that the amount of acetone contained in the air was too small to be worth recovering. This at least was the experience of various governments with which he was acquainted. Mr. Reid also said that there would be a good opening for a chemist to produce acetone cheaply. He believed there would be some opening, although he believed the quantities that were used by each government were much smaller than was generally supposed. Still a good deal of saving might be effected in that direction, and as a matter of fact he

(Mr. Guttman) had erected an acetone plant for the Government at Waltham Abbey. With regard to powders containing nitroglycerine, being rapidly superseded by those containing only nitrocellulose, as he had said in his paper he did not wish to express any opinion as to the merits of the various powders. He believed it would be wiser to abstain from so doing. Everybody had a right to his own opinion, and Mr. Reid would allow him to have his. Then Mr. Reid said the projectiles could not be elongated beyond a certain point, which was quite right. He only said that with the smaller bore the projectiles had to be lengthened. To what extent that was possible was rather a question for military men, still it had been found that a rifle with five mm. bore, or about 0.2 of an inch was quite practicable. It had been tried, and it was quite on the cards that it might be introduced somewhere. As far as the rifling of such a small bore went, there was no difficulty in making even so fine a rifling as required for that. Mr. Reid was not quite right when he said the wound produced by such a small projectile was so small that a man might go on fighting, and then afterwards find himself incapacitated. Experiments had been made with small bore bullets for a very long time, and a very elaborate work on the subject had been issued by the firm of Lorenz, of Carlruhe, who first made compound bullets. Quite recently in Austria, he was sorry to say, there had been riots, and the small arm rifle had played havoc with some of the miners, and when examined it was found that the bones were shattered more than with a large bore. They made almost incurable wounds. With regard to the amount of smoke developed, what Mr. Reid stated held good to a certain extent only. There were certain products of combustion which if you burnt a powder in the open air would simply escape by force of the combustion, that is to say, solid particles not being burnt, and that would not take place in a confined space to such extent; but when you burnt a small quantity of powder which flashed up in a comparatively long time when there was plenty of time for combustion to take place, the difference between the smoke as regarded a confined and unconfined space was not so very large. The use of liquified gas for the propulsion of bullets had been mentioned, and the experiments of M. Giffard of the sliding railway fame in the Paris Exhibition. He believed both M. Giffard and Mr. Edison, who wanted to decompose water and form gas and explode it, were on the wrong tack as far as practicality went. He quite agreed that theoretically the combustion of solid oxygen and hydrogen were probably the best things in the world, but to introduce them into practice required a genius, and he feared that manufacturers were not always geniuses. Then it was said that globular powder was better than flat powder, especially that flat powders caked together, and the flame could not be propagated so fast. He had no interest in advocating either one or the other, but all he could say was that he had on the table samples of smokeless powder of various descriptions, and every one examining Von Förster's powder would find that it consisted of a peculiar form of flakes which made them fill up an ordinary black cartridge, although the weight was only one-third, and there was plenty of air space and probably more than with globes. With a globular grain if you made it small you would have plenty of surface in relation to bulk, but if you had it too great the bulk would be much larger than the surface presented, and this might not be the case in the flake powder. For large powder like ballistite it would be impossible to make globular powder of that size. Globular powder might have its advantages as against cubical powder, but it would take too long to go into full explanation of these details; it would be sufficient to say that those who selected cubical powder had very good reasons for their choice, those who selected flakes, and those who selected the globular had also a reason for what they did. Every one tried to work in the right direction, and, as Mr. Reid said, in the result the fittest would be found to survive. With Von Förster's smokeless powder a peculiar mode of drying was adopted, which caused the flakes to crumple up, and consequently leave a great amount of air space. Reference had been made to the accidents at Waltham Abbey, and it was said that such accidents did not happen at private shops. He was happy to say that he had not yet had a serious

accident in his 20 years' experience, but he believed he was an exception. He was sorry to say that most of the manufacturers had found in starting their works, and in the beginning when they did not quite know how to work or exactly the nature of the stuff with which they were working, that there were sometimes explosions. Not so very long ago there was a very severe conflagration, not an explosion, in a ballistite factory in Italy, and there had been a number of accidents in connexion with nitroglycerine all over the world, although much less than in gunpowder works, so that they need not be surprised that there had been some at Waltham Abbey. There must always be some experiments made, but he believed they could be done with proper care. He could not speak any further on that subject as he might have to give evidence on the subject. But he thought it would be found eventually that the explosions at Waltham Abbey, except the last one, were not of such a serious character as was thought. With regard to electricity in connection with nitrocellulose powder Mr. Reid would remember that he dealt with that very fully in a paper before this Society last year, where he acknowledged his work in construction of drying houses so as to make them nonelectric. He did not think the black-leading of nitrocellulose was done with that object generally, though it had that effect; and he quite agreed with Mr. Reid that the electricity should be carried away with such powders. Unfortunately, every manufacturer did not see it in that light, and he feared they would have many gun-cotton drying-houses on fire before they realised the importance of this matter. The black-leading was generally done for the purpose of filling up the pores of the powder, and giving it a smooth surface. Mr. Bloxam had found fault with him for not mentioning powder for the Navy, but as a matter of fact he did not know of any general difference between powder for the Navy and powder for the Army, for the same effect was desired, the same class of powder would be used, and the exigencies for all of them were nearly the same. With reference to the high temperature of the magazines he thought he recollected seeing in some paper a letter asking why did the constructors of ships set their magazines so near the boilers so as to over-heat them; surely they might put them somewhere else, and if for other reasons a powder is preferred which does not stand heat so well as black powder the removal of the magazine was not an insurmountable difficulty. Then there was an answer to that, of course, that they could not alter the ships, the magazine was in the most handy place, and so forth, but even then there were means known by which such magazines could be cooled down if necessary. But in general the danger of over-heating such powder was not so great as people imagined. It would stand a good deal of heat before it decomposed or went off. Mr. Maenab had corrected him with regard to rifleite not containing potassium nitrate. He also said ballistite could be made in sheets up to 1½ ins. in thickness. No doubt it could; he believed it might be made thicker, but he did not believe they would be good sheets, or that they would work well in a gun. In making such thick sheets the difficulty of removing the air-bubbles and the solvent would be very considerable.

Mr. W. MAENAB said he referred to powder made with nitroglycerin.

Mr. GUTTMANN said he understood that, but even with these the air-bubbles would remain. Mr. Maenab had answered Mr. Bloxam with regard to carbon monoxide, and therefore he need not say much with regard to it. There would be carbon monoxide wherever there was imperfect combustion, but their aim was to make a powder which had perfect combustion; ballistite, cordite, and nitroglycerine powders in general gave perfect combustion, provided they were properly made. Nitroglycerine having an excess of oxygen in burning would always produce gases containing less carbon monoxide than others. His remarks as to heating, and so forth, were highly interesting, and he understood Mr. Maenab had done a great deal to investigate the heat developed by various powders.

Meeting held Monday, 4th June 1894.

MR. WM. THORP IN THE CHAIR.

## AN ACCOUNT OF SOME EXPERIMENTS ON THE STRENGTH OF LEATHER.

BY WALTER G. McMILLAN.

IN course of his work in the Indian Ordnance Department the author was asked by the Director-General of Ordnance in India to undertake a series of mechanical tests with the object of ascertaining the tensile strength of harness-leather made in the Government Harness and Saddlery Factory at Cawnpur, and to compare it with that of samples made in private tanneries in Madras and in England. Several hundred tests of various kinds were made, and it is proposed, with the sanction of General Walker, to summarise the more useful of these results in this short paper. Visits to the Cawnpur factory have enabled the writer to give a sketch of the system of tannage by which the leather was made, and thus to give additional interest to the research.

The factory is worked by the Ordnance Department of India, under the immediate superintendence of officers of the Royal Artillery. The foreman is an experienced English tanner, and he is assisted by several staff-sergeants instructed by him in such details as are required to enable them to supervise the native workmen in their own departments. To Colonel Baddeley, the superintendent, and to Mr. Wilsforth, the foreman tanner, I am very greatly indebted for information in regard to the details of Cawnpur tannage. The tannery is of no mean size, there being usually from 35,000 to 40,000 hides under treatment in the tanyard at a time; the currying and the subsequent fashioning of the leather into harness and saddlery components are conducted in the same establishment.

### THE SYSTEM OF TANNAGE USED IN THE CAWNPUR MANUFACTURE.

THE FACTORY is situated on the banks of the Ganges, close to the point at which it is joined by the Ganges Canal, and it is from this latter source that the water supply is drawn. The following numbers are the results of an analysis of water from this canal made by Dr. Compigné in the autumn of 1867, and recorded in a Government publication\* :—

Degrees of total hardness .....	4.7
“ permanent hardness .....	2.8
Grains of oxygen required to oxidise the readily oxidisable organic matter in 1,000 grains of water..	0.00065
Solids in 70,000 grains of filtered water .....	8.26
Volatile matters .....	0.7
Mineral .....	7.56
Earthy salts, &c. insoluble in water .....	5.07
Lime as carbonate .....	2.6
Soluble salts .....	2.5
Sodium chloride .....	1.5

With the exception of the somewhat large percentage of suspended matter, a difficulty met by allowing the supply to stand in settling tanks before use, the water appears to be suited for the work of the place.

THE HIDES are for the most part buffalo and cow hides, which come to the factory from different parts of India very lightly salted, while a few are obtained green from the Cawnpur butchers and require immediate treatment. They are said to be fairly well flayed as a rule, with but few flesh cuts and little fat. Occasionally, careless handling prior to salting is found to have produced local putrefaction, which greatly deteriorates or destroys the skin, and is made apparent in the lime pits, even if it have not declared itself previously.

THE TANNING MATERIAL used has generally been Babul (*Acacia Arabica*) bark, with a small proportion of myrabolans; within the last few months valonia has been

substituted for the latter, but all the Cawnpur leather referred to in this paper was made with the addition of myrabolans. A few experiments were tried with a view to introducing the use of Sál (*Shorea Robusta*) bark in place of Babul, but the leather so prepared was somewhat hard and dark-coloured, and gave a distinctly inferior test, as the numbers quoted hereafter will show. Babul bark, which is abundantly obtainable from local contractors, has been found by Christy to yield 18.95 per cent. of a good cream-coloured tannin; a sample given to the author on the occasion of his visit to Cawnpur in 1893 gave 21 per cent. of tannin, while a specimen of Sál bark yielded only 9 per cent. The bark is said to be of fairly constant strength, showing only a slight loss of tannin (by exposure) during the rainy season. The exhaustion of the bark is effected in latches, which are worked in series of eight, giving different solutions, with a range of from 2 to 50 barkometer. The latches are of brick with wooden false bottoms, and are of three different sizes in different sheds. The sizes and weights of material employed are as follows:—

Size of Latch.	Charge of Bark.	Charge of Myrabolans.
12' x 19' x 8	Cwt. 51	Cwt. 15
16' x 10' x 8	50	15
10' x 9' x 6	35	10

The tan liquors are returned to be refreshed, to one or other of the latches according to the strength indicated.

THE TANNING PROCESS is conducted as follows:—

The hides are soaked in pits, in which the water is changed whenever, from its appearance, it is judged to require renewal; they are then stocked for about half an hour in a gentle stream of running water.

They are next limed in a series of six pits, each fresher than the last. The lime pits are, as usual, worked in rotation, being made up originally with 5 cwt. of lime to the pit; this liquid lasts for about a month without further addition of lime, and treats about 700 or 800 hides. Each hide may remain in the limes for from 15 to 18 days, but in the hottest weather a somewhat shorter soaking suffices.

They are now unlimed and fleshed, and are then bated in a liquor made up by extracting seeds from the pods of the Babul tree with water. This bate has originally about 2 cwt. of the seed to each pit, and lasts about three weeks; a gentle fermentation is initially set up by the addition of a few buckets of tan-liquor. For light leather a bran bate is generally substituted. The process demands careful attention at all times, but particularly in the hot season.

The hides are now at once transferred to handlers containing weak liquors (7° to 8°) for two or three days, being handled every two or three hours during the first day. The spent liquors from this vat are allowed to run to waste. After this, the skins are handled daily for about a month in stronger liquor (15°); then for a month in “floaters” in liquor of about 20°; next they are transferred to “dusters” working at a strength of 30° and containing a small proportion of fresh bark; here they are handled daily for from one to two months, and are then put away for about six months in layers, the liquor-strength in which varies from 35° to 50°, but averages 45°. Each layer-pit contains, in addition to the liquor, 4 cwt. of bark and 2 cwt. of myrabolans, and treats 100 hides.

CURRYING.—The hides are now transferred to the curriers' shops, where they are successively shaved to the required thickness, scoured on flesh and grain, passed rapidly through weak sumach liquors, oiled with cod-oil, sammed, and struck out, re-shaved or flatted, stuffed with a mixture of cod-oil and tallow (in equal parts in the cold weather, but containing 50 per cent. of tallow in the hot season), and are finally dried out and finished by the removal of surplus grease.

In all the processes here described, the average treatment has been given; but this must of necessity be greatly modified at different seasons in a climate which is equivalent

\* “Statistical, Descriptive, and Historical Account of the North-West Provinces of India,” Vol. VI., p. 223.



to an English summer in the cold weather, where the thermometer may daily rise for several months to over 100° F., when a very small proportion of atmospheric moisture exists, and where for two or three months the atmosphere may be saturated with water vapour with a thermometer indication of over 90°.

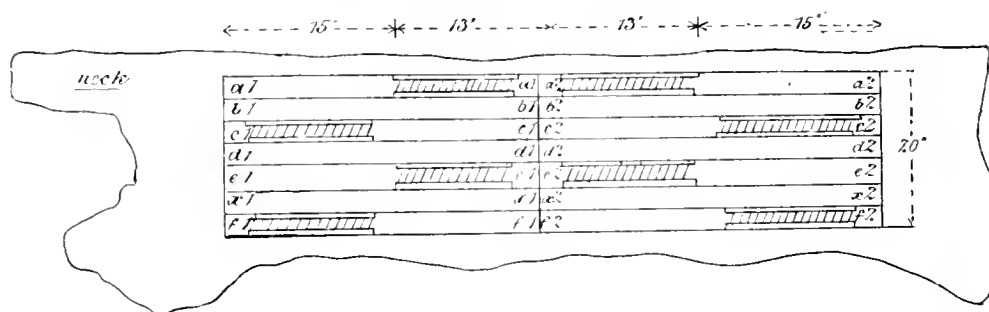
Concerning the processes used in the preparation of the Madras and of the English leather, the author has no information. The latter, however, was taken from a large number of hides supplied to the Government by a good firm of English tanners.

The tests to be described were made chiefly with new leather. Half hides were supplied to the author's Department and were cut, under his direction, in the manner detailed below. A certain number of the Cawnpur (Babul- and Sâl-

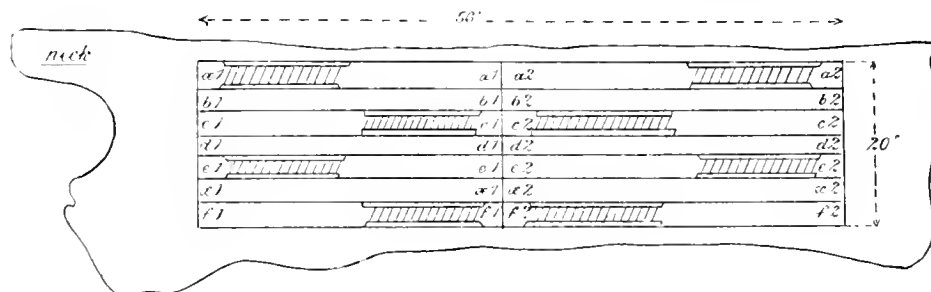
tanned) English hides were made up at Cawnpur into harness components, were issued to a battery of artillery, and, after six months' service, were returned for test, and were then cut up into straps of suitable shape and size for the testing-machine. The testing-machine was one of Greenwood and Batley's lever pattern, and although intended and generally used to test metallic specimens up to a 100-ton load, was yet equally adaptable to the measurement of any stress exceeding 500 lbs.

#### TESTS OF NEW LEATHER.

The half skins being submitted, straps measuring in the test-portion 10 inches in length by 2 inches in width, were cut from different positions in the hide, as indicated in the annexed diagrams.



Method A. of cutting: with test-portions in "means" of straps a and e.



Method B. of cutting: with test-portions in "extremes" of straps a and e.

N.B.—The shading (10" x 2") indicates the actual portion of the strap on which the test is made and measured.

TABLE I.  
SHOWING THE AVERAGE STRENGTH OF THE VARIOUS LEATHER SAMPLES.

Hide.	Neck Half.								Rump Half.							
	No. of Hides Tested.	No. of Straps Tested.	Thick-ness at Point of Rupture in Ins.	Average Breaking Stress in Lbs.		Average Stretching per Cent.		No. of Straps Tested.	Thick-ness at Point of Rupture in Ins.	Average Breaking Stress in Lbs.		Average Stretching per Cent.				
				Actual.	Per Sq In.	Under Maximum Load.	Perma-nent after Rupture.			Actual.	Per Sq. In.	Under Maxi-mum Load.	Perma-nent after Rup-ture.			
Cawnpur, heavy, <i>mean</i> ....	3	12	0.158	1,650	5,190	26.0	6.3	12	0.194	1,870	4,780	26.0	6.6			
"    light, <i>mean</i> .....	3	12	0.110	1,200	5,740	21.0	6.0	12	0.175	1,840	5,220	27.0	5.8			
English, heavy, <i>mean</i> .....	6	24	0.174	1,490	4,330	29.0	6.4	24	0.246	1,810	3,960	29.0	7.1			
"    light, <i>mean</i> .....	6	24	0.163	1,040	5,080	28.0	6.5	24	0.142	1,270	4,540	27.0	5.7			
Madras, <i>mean</i> .....	3	12	0.141	1,480	5,360	28.0	6.4	12	0.212	1,890	4,900	29.0	6.8			



Half of the hides were cut according to method A, half according to method B; only the straps *a*, *c*, *e*, and *f* were tested in the usual way, the intervening strips *b*, *d*, and *x* being reserved for special tests. All pieces marked *1* are considered to have come from the neck half, those marked *2* from the rump half of the hide.

The test portion was always of the size quoted ( $10'' \times 2''$ ), and the edges and corners were carefully and evenly cut. The thickness was measured by means of a micrometer gauge at 2-inch intervals of length, so that the thickness at the point of rupture might be accurately known. The short end of the piece was gripped directly by the jaws of the testing-machine, and the long end was turned over on itself, and stitched on to form a loop which passed over a large pin, placed in the moving cross-head of the machine. The 10-inch length, forming the test portion, was thus freely suspended between the supports of the machine. To

ensure rupture occurring in the desired part, the test portion was cut  $\frac{1}{2}$  inch narrower than the ends, which were left  $2\frac{1}{2}$  inches wide.

The tests of six Cawnpur (Bahul), twelve English, and three Madras hides are dealt with in Table 1.

For the sake of showing the variation in strength at different parts of the hide, the above results have been worked up into the following diagram, which may to some extent be regarded as representing the distribution of strength in typical hides, every number quoted being the mean of the breaking loads of all the straps from similar positions in hides of the same class. The straps marked *a* were nearest to the back; those marked *f* were within a short distance of the belly edge of the skin, the letters corresponding to those given in the diagrams sketched above.

TABLE 2.  
SHOWING DISTRIBUTION OF STRENGTH IN THE HIDES.

Method of Cutting.		Heavy Hides.				No. of Hides contributing to Mean.	Light Hides.							
		Neck.		Rump.			Neck.		Rump.					
CAWNPUR:														
Method A.....	a.....	2020	5830	1800	4303	..	..	1200	5103	190	5700	..		
	c.....	1470	5330	..	..	2000	4803	1150	6070	..	..	2050	4900	
	e.....	..	1810	5330	2160	4950	..	..	1500	5850	130	5150	..	
	f.....	1370	5030	..	..	1350	4250	1500	5200	..	..	1450	5000	
Method B.....	a.....	1300	4200	..	..	2200	5550	700	3550	..	..	2000	5100	
	c.....	..	2110	5503	2490	5603	..	..	1400	5750	2550	6 00	..	
	e.....	1700	5400	..	..	1700	4450	1500	6450	..	..	1400	4300	
	f.....	..	1500	4350	1400	5450	..	..	..	..	1400	5550	..	
ENGLISH:														
Method A.....	a.....	..	1300	3250	1500	3350	..	..	1100	4300	1200	4350	..	
	c.....	1400	5150	..	..	1720	3300	950	5000	..	..	1110	3500	
	e.....	..	1810	4700	2080	4150	..	3	..	1100	5600	1500	5103	..
	f.....	1290	4820	..	..	1700	3800	670	4800	..	..	1200	4750	
Method B.....	a.....	1280	3550	..	..	1450	3000	910	4630	..	..	1080	4250	
	c.....	..	1600	3950	1910	3300	..	3	..	1130	5003	1300	4650	..
	e.....	1340	4550	..	..	1600	2950	1010	5203	..	..	1050	3650	
	f.....	..	1850	4700	2200	4800	..	..	..	..	1500	5750	..	

NOTE.—The ordinary figures represent the actual breaking load in pounds; the antique figures *a* to *f* the stress in pounds per square inch of sectional area.

A few tests were next made to determine the relative values of the leather samples when saturated with water. In Table 3 will be found recorded the strength of the wet straps, together with the difference per cent. of this result as compared with that of a normal (dry) test of a strap cut from a position immediately adjacent in the same hide. The straps were allowed to soak in pure water for 18 hours prior to testing, and were transferred to the testing-machine directly they were withdrawn from the water. The excess water became rapidly wrung out of the leather as stress was applied and stretching set in, and at the conclusion of the test the strap was only damp. It does not appear that the wetting has appreciably altered the strength of the leather, as the results varied on both sides of the normal, and the mean difference of 5 per cent. is scarcely sufficient to indicate a serious diminution based on so few results.

In order, if possible, to ascertain how far statements of test results reported on the square inch of cross section were reliable for samples which had not undergone perfectly uniform treatment under the fletcher's knife, a few tests were made with specially shaved hides. The straps were thinned evenly down with a sharp knife by men accustomed to working with leather, until about half of the substance

had been removed from the flesh side, leaving the grain side intact. The results of the tests are given in Table 4, and a comparison is made with normal tests from adjacent straps. It will be seen that a reduction of thickness equal to 50 per cent. reduces the strength, on the average, by about 15 per cent., while the extensibility under stress is considerably increased. I may here mention that in some earlier tests not reported in this paper, a deep flesh cut which occurred across the centre of one strap appeared to have no appreciable influence on the result.

Finally, a tearing test was made by passing a round steel hook  $\frac{1}{4}$  inch in diameter through a clean-cut hole in the leather, and applying tension through this hook, noting the point at which the hook began to tear through the hide, and that at which complete rupture resulted.

In Table 6 is given the average thickness and weight, both per sq. in. of surface and per cu. in. of substance, of the hides examined, as found from the measurements made of the various straps cut from each.

\* The number of hides tested is of course insufficient to give a good average.

TABLE 3.  
SHOWING THE RESULTS OF WET TESTS.

Hide.	Thickness in Inches.	Breaking Stress in Lbs.				Extension per Cent.		
		Actual.	Per Square Inch.	Difference from Normal.	Under Stress.	Difference from Normal.	After Rupture.	Difference from Normal.
Cawnpur, heavy, No. 2 .....	0.160	1,610	5,031	-12.6	29	+ 7	11.0	+ 53
"    light, No. 1 .....	0.125	1,500	4,900	-12.0	26	+ 4	11.5	+ 63
English, heavy, No. 1 .....	0.256	1,800	3,529	-27.5	33	0	12.0	+ 56
"    "    4 .....	0.194	1,500	3,879	- 4.9	36	+20	13.0	+136
Madras, No. 1 .....	0.210	1,700	4,050	- 4.2	38	+ 9	15.0	+ 23

Rump End.

Hide.	Thickness in Inches.	Breaking Stress in Lbs.				Extension per Cent.		
		Actual.	Per Square Inch.	Difference from Normal.	Under Stress.	Difference from Normal.	After Rupture.	Difference from Normal.
Cawnpur, heavy, No. 2 .....	0.194	1,700	4,380	-13.6	29	- 7	11.0	+ 28
"    light, No. 1 .....	0.205	2,100	5,120	- 6.4	27	+ 8	10.8	+116
English, heavy, No. 1 .....	0.250	1,900	3,800	-11.8	29	- 9	12.0	+ 71
"    "    4 .....	0.214	1,800	3,330	-11.1	35	+24	13.5	+ 61
Madras, No. 1 .....	0.211	2,150	5,120	-20.5	35	- 3	12.5	+ 25

TABLE 4.  
SHOWING RESULTS OF TESTING SHAVED HIDES.

Hide.	Thickness in Inches as Tested.	Percentage Shaved away.	Breaking Stress, Lbs.	Breaking Stress per Sq. In.		Extension per Cent.			
				Lbs.	Difference from Normal.	Under Load.	Difference from Normal.	After Rupture.	Difference from Normal.
Cawnpur, heavy, No. 2 ..	0.086	41.1	804	4,650	-11.0	30	+ 7	7.5	+ 7
"    light, No. 1 ...	0.075	39.5	520	3,470	-20.5	30	+ 7	9.0	+38
English, heavy, No. 3 ...	0.088	52.9	700	3,980	-17.2	37	+16	12.4	+77
Madras, No. 3 .....	0.085	30.3	840	4,940	-13.9	31	+35	7.6	+33
Mean .....	..	41.0	..	..	-17.9	..	+16	..	+29

Rump End.

Hide.	Thickness in Inches.	Percentage Shaved away.	Breaking Stress, Lbs.	Breaking Stress per Sq. In.		Extension per Cent.			
				Lbs.	Difference from Normal.	Under Load.	Difference from Normal.	After Rupture.	Difference from Normal.
Cawnpur, heavy, No. 2 ..	0.087	50.2	950	5,570	+ 2.2	35	+13	11.6	+35
"    light, No. 1 ...	0.105	42.6	850	4,050	-16.0	37	+19	10.5	+45
English, heavy, No. 3 ...	0.110	57.0	800	3,640	-12.5	45	+25	19.0	+87
Madras, No. 3 .....	0.088	58.5	640	3,640	-30.5	37	+ 6	12.2	+22
Mean .....	..	51.5	..	..	-15.3	..	+16	..	+47

TABLE 5.  
SHOWING RESULTS OF TEARING TESTS.

Hide.	Neck End.			Rump End.				
	Thickness at Perforation.	Incipient Tearing Observed, Lb.	Actual Rupture at Lb.	Lb. for Strap 0'1 in. Thick.	Thickness at Perforation.	Incipient Tearing Observed, Lb.	Actual Rupture at Lb.	Lb. for Strap 0'1 in. Thick.
Cawnpur, heavy, No. 1 .....	0'190	500	550	290	0'216	500	550	250
"      No. 3 .....	0'193	450	500	250	0'219	500	550	260
Mean .....	0'191	475	525	275	0'213	500	550	255
English, heavy, No. 1 .....	0'235	550	600	250	0'274	500	550	200
"      No. 2 .....	0'215	?	500	250	0'258	500	550	210
Mean .....	0'225	..	550	240	0'266	500	550	205
Madras, heavy, No. 1 .....	0'195	350	400	210	0'237	450	470	200
"      No. 2 .....	0'193	?	250	150	0'221	350	400	180
Mean .....	0'181	..	325	180	0'220	385	435	190
Cawnpur, light, No. 1 .....	0'149	?	<500	?	0'202	?	500	250
"      No. 3 .....	0'154	300	350	230	0'190	450	550	240
Mean .....	0'151	..	..	230	0'196	..	525	270
English, light, No. 1 .....	0'140	?	250	?	0'121	?	220	180
"      No. 2 .....	0'125	?	200	160	0'136	?	250	180
Mean .....	0'132	..	225	160	0'128	..	235	180

TABLE 6.  
SHOWING THE AVERAGE WEIGHT AND SUBSTANCE OF EVERY HIDE EXAMINED.

Hide, Average of.	Mean Weight in Grains per Sq. In.			Mean Thickness in Ins.			Mean Weight in Grains per Cub. In.		
	Neck End.	Rump End.	Whole Hide.	Neck.	Rump.	Whole.	Neck.	Rump.	Whole.
3 Cawnpur heavy hides .....	39'2	45'5	42'3	0'174	0'204	0'187	226	227	226
3 " light hides .....	29'2	41'3	35'3	0'127	0'183	0'155	230	225	224
6 English heavy hides .....	46'2	61'0	53'6	0'185	0'252	0'218	249	242	246
6 " light hides .....	26'9	34'0	30'4	0'113	0'144	0'128	238	235	236
3 Madras hides .....	34'4	48'3	41'3	0'150	0'218	0'184	228	220	224

In order to throw further light upon the nature of the leather tested, its absorbing power in regard to water was taken, and also the specific gravity of the material when saturated with water, the results of this examination being recorded in Table 7. It will be noted that the most absorbent and least dense leather was that of Cawnpur manufacture, next the Madras, and last the English, which order represents also that of their respective tenacities.

Summing up the work, the highest and lowest normal tests, and the mean of each series, together with the results of the tests of the experimental salt-tanned specimens, are included in Table 8.

#### TESTS OF USED HARNESS-LEATHER.

The only tests which it was possible to make on the standard size of strap (10" x 2") were cut from the central portion of leather girths, and these results are included in Table 8 (above) for convenience in comparison with those obtained from unused leather. The size or shape of the other harness components would not admit of the cutting of so large a strap; all the remaining tests were therefore made upon the longest and widest pieces that could be cut in any given case.

TABLE 7.

SHOWING ABSORBING CAPACITIES AND SPECIFIC GRAVITY OF THE SPECIMENS.

Title.	Hygroscopic Water.		Maximum of Water absorbed on Soaking.		Specific Gravity of Wet-Leather Water = 1.
	Water in Leather as Supplied.	Percentage of Water absorbed from Air by perfectly Dried Leather.	By Leather as Supplied.	By Perfectly Dried Leather.	
	Per Cent.		Per Cent.	Per Cent.	
Cawnpur, heavy, No. 1.....	12.21	13.91	49.1	70.17	1.194
"    "    " 1 (R)*.....	12.46	14.23	51.9	70.92	1.192
"    "    " 2.....	11.97	13.53	53.8	81.54	1.181
"    "    " 2 (R)*.....	12.31	14.04	53.3	74.80	1.184
"    "    " 3.....	12.28	14.09	53.1	74.52	1.189
Mean.....	12.24	13.95	54.1	75.59	1.188
Cawnpur, light, No. 1.....	11.37	13.60	68.8	91.8	1.165
"    "    " 2.....	11.29	12.73	59.2	79.43	1.173
"    "    " 3.....	11.66	13.20	64.7	86.50	1.174
Mean.....	11.34	13.18	64.2	85.91	1.171
Mean of all Cawnpur samples.....	11.94	13.54	59.1	80.75	1.180
English, heavy, No. 1.....	11.41	12.88	35.77	53.26	1.205
"    "    " 2.....	11.42	12.90	32.88	50.63	1.205
"    "    " 3.....	11.41	12.88	35.51	52.97	1.200
"    "    " 3 (R)*.....	11.90	13.62	33.84	51.77	1.223
"    "    " 4.....	16.97	12.32	35.63	52.30	1.196
"    "    " 5.....	10.85	12.42	35.97	52.53	1.195
"    "    " 6.....	11.20	12.64	35.76	52.88	1.203
"    "    " 6 (R)*.....	11.65	13.19	36.56	54.80	1.199
Mean.....	11.56	12.81	35.24	52.57	1.203
English, light, No. 1.....	15.02	17.71	48.5	74.77	1.184
"    "    " 2.....	14.13	16.46	47.1	71.65	1.188
"    "    " 3.....	14.53	17.69	53.5	81.99	1.166
"    "    " 4.....	14.23	16.60	49.9	74.79	1.194
"    "    " 5.....	14.32	14.72	54.5	75.66	1.183
"    "    " 6.....	13.84	16.02	52.3	77.73	1.181
Mean.....	14.34	16.75	50.7	76.10	1.183
Mean of all English samples.....	12.85	14.78	42.98	64.33	1.193
Madras, No. 1.....	12.19	13.85	48.67	69.26	1.179
"    "    " 1 (R)*.....	12.96	14.94	49.73	72.03	1.188
"    "    " 2.....	12.07	13.99	51.57	72.32	1.156
"    "    " 2.....	11.56	13.97	46.89	66.08	1.165
"    "    " 3 (R)*.....	13.65	15.80	44.88	67.78	1.196
Mean of all Madras samples.....	12.19	14.26	48.35	69.49	1.177

\* In this table "R" attached to a mark indicates that the specimen was cut from the extreme rump end, whereas the remainder were cut from near the neck.

TABLE 8.  
SUMMARY OF RESULTS.

Hides.	No. of Straps Tested.	Ultimate Strength in Lbs.						Extension per Cent.					
		Actual.			Per Sq. In.			Under Maximum Load.			Permanent, after Rupture.		
		Highest.	Lowest.	Average.	Highest.	Lowest.	Average.	Highest.	Lowest.	Average.	Highest.	Lowest.	Average.
Cawnpur, heavy, Babul tannage.	24	2,410	1,100	1,760	6,110	3,180	4,990	33	15	26	10.0	3.0	6.6
Cawnpur, light, Babul tannage.	24	2,570	700	1,520	6,820	3,570	5,350	31	13	24	10.0	3.0	5.0
Cawnpur, experimental, Sal tannage.	24	2,020	850	1,390	5,250	2,850	4,190	34	10	27	11.5	4.0	8.0
Madras, heavy, .....	16	2,150	1,390	1,700	6,860	3,800	4,840	36	25	29	12.2	5.0	6.8
" light .....	8	2,220	1,300	1,650	6,340	3,730	5,070	35	23	25	10.0	3.0	6.1
English, heavy, .....	48	2,400	1,010	1,650	5,650	2,240	3,990	38	17	29	12.0	3.0	6.7
" light, .....	46	1,700	600	1,150	7,040	3,250	4,790	37	19	27	11.4	3.0	6.1
<i>Used Straps.</i>													
Girths, Cawnpur (Babul) .	32	1,950	1,010	1,480	5,740	2,680	4,440	25	15	21	8.0	3.0	7.0
" " (Sal) .....	18	1,500	900	1,235	4,300	2,900	3,615	Not taken.					
" English .....	15	1,740	800	1,160	5,370	2,280	3,480	25	16	20	5.0	2.0	3.0

NOTE.—The Sal tannage was conducted similarly to that with Babul bark, and the test straps had the same dimensions. The mean thickness at the point of rupture was 0.17 inch.

TABLE 9.  
TESTS OF USED LEATHER ON STRAPS 10 INCHES LONG BY 1.5 INCHES WIDE.

Straps.	No. of Straps Tested.	Ultimate Breaking Stress in Lbs.						Extension per Cent.					
		Actual.			Per Sq. In.			Under Maximum Load.			Permanent, after Rupture.		
		Highest.	Lowest.	Average.	Highest.	Lowest.	Average.	Highest.	Lowest.	Average.	Highest.	Lowest.	Average.
Breeching-strap, Cawnpur	5	1,900	1,300	1,750	6,400	4,330	5,820	33	23	28	10	5	7
" " English ..	5	1,440	1,000	1,240	4,800	3,250	4,030	33	28	31	8	5	7
Sureingle, Cawnpur, .....	15	1,900	900	1,230	7,700	4,430	5,870	32	25	28	10	4	7
" English .....	15	1,520	700	1,010	6,140	2,910	3,990	40	20	32	11	5	8

TABLE 10.  
TESTS OF USED LEATHER ON PLAIN AND PERFORATED STRAPS. 5 INCHES BY 1 INCH.

Straps.	Number of Straps Tested.	Ultimate Breaking Stress in Lbs.					
		Actual.			Per Square Inch.		
		Highest.	Lowest.	Average.	Highest.	Lowest.	Average.
Hip-straps, sound, Cawnpur, .....	15	1,300	850	1,140	6,350	3,590	5,020
" " English, .....	15	800	600	700	3,850	3,080	3,750
Flank-straps, perforated, Cawnpur ..	5	900	510	700	4,500	2,780	4,000
" " English ..	5	720	500	570	3,630	2,440	2,990

In Table 9 it will be noticed that the tests are higher than the corresponding tests of used straps 2 inches wide, recorded in Table 8. The author has had other evidence, however, confirmatory of the observation that a narrow strap is proportionately stronger than a wide one.

The special interest attaching to Table 10 is to be found in the indication which it gives of the extent of the weakening produced by perforations made to receive the tang of a buckle.

Finally, it may be observed that tests of the raw hide and of the semi-manufactured hide gave the following results—

	Actual Strength.	Strength per Square Inch.
Mean of 5 tests: 12 specimens (untested) ..	Lbs. 1,376	Lbs. 6,000
" " " " " " " " soaked hide, after soaking ..	1,300	10,226
" " " " " " " " stocking ..	1,096	8,420
" " " " " " " " bathing ..	750	5,310
" " " " " " " " dusting ..	970	5,080

The strips used for these tests were taken at random from the Cawnpur tanyard, and were tested on the machine in the author's laboratories at Cossipur, after an interval made as short as possible, in order to avoid deterioration by putrefactive action.

Finally, to bring to a focus a few of the points which appear to be indicated by the experiments quoted in the paper:—

A. In comparing the leathers produced by the two systems of tannage, that which had the lower specific gravity (wet or dry) was to a marked extent more absorbent than the other, and was at the same time stronger both actually and per square inch of section, but stretched less before rupture.

B. In leather produced by a given system of tannage—

1. Thin hides are stronger per unit of sectional area than thick hides, while the ultimate extension before rupture, although practically the same, is on the average slightly greater in the stout or specimens.

2. Thin hides are more absorbent, and have a lower specific gravity when wet than thick hides.

3. In any given hide, omitting from consideration the extreme neck portion of the back, which is altogether inferior, straps from the rump half should carry a higher actual load by reason of their greater thickness, but will nevertheless stand a lower stress per square inch than those from the neck half.

4. With average hides, a comparison of strength per unit of sectional area is permissible, but shaving or an exceptionally severe use of the fleshing knife may lower the strength per square inch of section, while it increases the extensibility.

5. Other things being equal, a narrow strap may be expected to be not only stronger than a wide one, both actually and per unit of sectional area, but also to stretch more under a given load.

#### DISCUSSION.

The CHAIRMAN said he hoped there were some specialists present acquainted with leather who would be able to discuss this paper. Mr. Macmillan had said he was rather sorry the English leather stood the tests worse than the Indian, which was perhaps a natural feeling, but it must be remembered that the Indian leather was tanned under English management, and perhaps climate had something to do with its superiority. As a simple matter of mechanics he was rather surprised to hear that a narrow strip should be stronger than a wide one. The idea of a thin strip being stronger than a thick one was more easily understood, because the greatest strength of leather was in the surface of the skin, and there would be more surface in proportion in thin leather than in thick. But it was perplexing that a narrow strip should be stronger than a wide one.

Dr. C. R. ALDER WRIGHT said he did not know whether Mr. Macmillan had any reason to suspect that any of the samples of English leather had been "dressed," which he believed was the euphemism for treating it with various materials with the object of giving it extra weight. Some few years ago there was a great deal of disturbance in the

minds of the officials at Woolwich and elsewhere with regard to the Government saddlery, and inquiries were made as to the quality of the leather supplied. He had then occasion to examine a considerable number of samples, some of which had, and some had not, been subjected to these weighting operations. One sometimes read in works of fiction of a man being wrecked in the Southern Pacific, and subsisting for a long period by sucking strips cut from his jack boots; but, as a matter of fact, some of the leathers in the market, if not absolutely competent to support human life for several weeks, had so much glucose and other nutritious materials in them that at any rate if boiled down, with the resources of a French *chef*, they would make a very nutritious soup indeed, as they would include these non-nitrogenous constituents as well as gelatinous matter. There was as much as 10, 15, or even 20 per cent. of glucose in some "dressed" leathers intended for saddlery, and he had examined a roll of leather where the average contained ran up to nearly 30 per cent.; he had obtained brandy, or spirit at any rate, in considerable quantity from the fermentation of the glucose extracted from such leather. He did not know how far a possibility of this sort might account for the inferiority of some of the English samples subjected to test. The object of this treatment was to add to the weight, but obviously it must add somewhat to the thickness, and perhaps to the rigidity; a sample treated in such a way containing 70 per cent. of actual leather, representing 100 per cent. as sold, would necessarily indicate a very much less degree of tenacity when reckoned on the 100 total material than if on the 70 parts of actual leather. Whether that were the case or not the difference between the breaking strain exhibited in different parts of the hide was certainly very valuable information, and the thanks of the Society were due to Mr. Macmillan for bringing these carefully-made observations before them.

Dr. S. RIDEAL said Dr. Wright had drawn attention to a point he was about to make in connection with this work of Mr. Macmillan. He was going to say that in leather tests of this sort it seemed very desirable that an analysis of the leather should be made. A determination of the nitrogen content of the leather would have given most valuable information as to the amount of tanning that had taken place on the different samples. Mr. Macmillan had given an elaborate description of the method adopted for tanning in Cawnpore, but whilst stating that the English samples came from an English tannery of good reputation, there was no information as to the process adopted, the length of tannage, or the materials used. If he had determined the nitrogen content one could have judged how far the tanning had proceeded. With regard to the tannage in Cawnpore, and the babul bark, he had never been in India, and knew very little about the botany of that country, but he believed that name was given to several trees of the acacia tribe, and that these acacias gave varying amounts of tannin, and tannin of different kinds, so that it was rather important that the exact kind of tree and not only the general name babul should be given. He presumed the *valonia* used was imported, and it would be interesting to know where it came from, or whether it was found in India. The bate was rather an interesting point, and he should like some further information with regard to the babul seeds and the fermentation set up. The bran fermentation was the common method in this country, and the bacteriology of the process had been studied. It would be interesting to know whether the same organisms are active in the babul-seed vats. The information as to the strength of strips of different sizes seemed to be very interesting and valuable, and he knew of no similar work having been done, so that the author was to be congratulated in bringing his results before this Society.

Mr. W. G. McMILLAN, in reply, said he was afraid he could not add much to the information that he had already given. With regard to the weighing of the leather, he had not observed any evidence of the addition of soluble organic matter, or of mineral substances remaining in the ash in the samples examined. He did not profess to be able to judge accurately of the quality of leather by inspection, but he had the opinion of the foreman tanner of

Cawnpore that the English samples were distinctly good, and he had placed on the table some of the leather for inspection after the meeting. He had not made nitrogen determinations owing to pressure of other work, which precluded him from undertaking anything beyond what was absolutely necessary; but if Dr. Rideal liked to have some specimens he should be glad to hand them over to him, or possibly he might be able to find time later on to make the determinations himself. The paper purported to deal primarily with the Cawnpore-tanned material, of which the tannage was described. The results of the English tests were, however, given for comparison. It is possible that the superior tenacity of the Indian leather may be due to the lighter salting which may have been given to a material to be used locally, than to one which was to be shipped and stored perhaps for months before tanning. With regard to the valonia, it had only recently been introduced, and was imported, but in all the experiments he had referred to myrobolans had been used; it was only within the last twelve months that valonia had been employed. Before he came away he saw a sample of local valonia which was to be tried, but he had not heard the result of the experiment. The bate was prepared by crushing the babul seeds and mixing the meal with water, the quantities used having been stated in the paper; a small quantity (about a pailful) of the old vat-liquor was added to set up the fermentation, which then went on quietly, regularly. He was told it lasted for about a month. Some authorities and Indian books stated that babul seeds were used as a tanstuff, but that was a mistake, which possibly arose through their having been used for bate; they contained practically no tannin.

## NOTE ON THE COLOUR OF BRASS.

BY WALTER G. McMILLAN.

So long as anything has been known as to the nature of the alloys of copper and zinc, certain distinctive colours have been associated with certain classes of brass. But the author is not aware that any attempt has been made to distinguish the minute gradations of shade corresponding with slight modifications in the percentage composition, or to apply these variations systematically to any practical use.

He therefore takes this, his first opportunity after returning to England, to exhibit two sets of glass tubes containing drillings of brass carrying varying percentages

of copper (71 to 63) which he collected in 1893 as the result of observations made upon alloys used in the manufacture of brass strip intended for the manufacture of cartridge cases.

In course of his ordinary duties he had made several hundreds of analyses of cartridge-metal, and a large increase in the volume of other work led him to enquire with what degree of accuracy the composition of the metal could be determined by inspection. A collection was made of all samples which had been analysed and were still in store, and these showed a range of colour from pale yellow in specimens containing 70 per cent. of copper, to a peculiar brownish shade of yellow in those having only 63 or 64 per cent. The general result was satisfactory, but the gradation from end to end of the range was not perfect. It was then remembered that the specimens were not all of identical origin; some of the drillings were from the cast ingot, others were from rolled strip; again, some drillings in each class were taken from a very pure alloy intended for the manufacture of the solid-drawn cartridge cases of the new magazine rifle, while the remainder were from brass which would be used as sheet or strip, and was less pure. A separation of the samples into the four groups mentioned, viz.:-

- A. Pure brass (i) cast and (ii) worked,
- B. A less pure alloy (i) cast and (ii) worked,

formed four series, each practically perfect in itself, but differing in colour to a slight but very appreciable extent from each of the corresponding series.

After the opening of the investigation, but before the grouping of the samples had been effected, a colour observation was made with each fresh specimen received for test whilst the gravimetric determination was in progress, but the standards and the new sample were examined in common German glass bottles, and these interfered with the accuracy of the readings. In this way 39 samples were were tested, first by inspection and then by analysis (electrolytically), and the two results were compared; out of these 39, 22 samples gave a higher reading by colour than by analysis, the variation ranging from 2.18 to 0.04 per cent., and averaging 0.76 per cent.; and 17 samples gave a lower reading, the average *minus* variation being 0.56, and the extreme 1.4 per cent. The two extreme samples (varying by + 2.18 and - 1.4 per cent. respectively) were the first two experimented with.

After the division into groups had been made, the standard specimens were sealed up in glass tubes, and, for comparison, the sample to be reported upon was introduced into a similar tube sealed at the one end and closed by means of a cork at the other. The actual results of the first 21 consecutive tests (and in fact the only tests) thus made were as follows:-

With Drillings from (rolled) Strip.

	Copper Percentage.		Variation.
	By Colour Estimation.	By Analysis.	
1	66.0	66.00	0
3	65.6	65.60	0
5	65.6	67.86	- 0.76
7	65.2	66.41	- 0.21
9	65.7	65.88	- 0.13
11	65.5	65.08	- 0.58
13	64.8	64.89	- 0.09
15	66.4	65.83	+ 0.57
17	67.1	67.23	- 0.13
18	66.0	66.34	- 0.34
21	65.0	65.35	- 0.35

With Drillings from (cast) Ingot.

	Copper Percentage.		Variation.
	By Colour Estimation.	By Analysis.	
2	65.3	65.03	+ 0.27
4	63.1	64.70	- 1.39
6	63.4	64.32	- 0.92
8	64.3	66.06	- 1.76
10	66.0	67.25	- 1.25
12	66.0	66.62	- 0.62
14	66.0	64.37	- 0.37
16	64.5	65.02	- 1.72
19	64.0	64.89	- 0.89
20	64.4	64.44	- 0.04



It will be seen that in the case of samples taken from ingots the highest variation was 1.76, and the mean variation 0.56 per cent., while with those from rolled strip the maximum difference between the actual and the estimated copper percentage was 0.76, and the mean difference only 0.21 per cent. The cause of the closer approximation in the case of the rolled, as compared with the cast, samples is undoubtedly due to the method of drilling the specimens by the native workmen in the engineering shops; those from ingot metal were obtained from small rectangular blocks about 4 in. by  $\frac{1}{2}$  in. by  $\frac{1}{2}$  in., and in drilling these the brass was often allowed to become so hot (owing to friction) that a perceptible film of tarnish resulted, and slightly altered the colour of the alloy. With the rolled strip, the samples were taken from thin sheet, which was drilled intermittently and therefore never became more than slightly warm. For this reason the possible accuracy of the process can be estimated fairly only from the numbers obtained with rolled strip. It is possible, too, that the observed difference in colour between cast and worked metal may be, in part, due to the slight film of tarnish which was so often present on the drillings from the former.

In the comparison of the samples, the general colour effect in the tube is produced by a combination of reflected light from the smooth cut faces of a few of the drillings with the more or less scattered rays from the rough surfaces on the under sides of the remainder; the difference between two specimens was, however, most accentuated on the rough faces.

In all the experiments recorded in the above table, the percentage of impurities in the brass was very constant, the lead ranging from 0.75 to 0.10 per cent., and the iron from 0.08 to 0.20 per cent., no other foreign substance being present in appreciable quantity. On one occasion, however, a sample was presented which had a peculiar greyish shade, and could not be fitted into any place in the series, although the percentage of copper was 65.9; the effect was produced by an abnormal (0.27 per cent.) proportion of lead. Any very marked variation in the percentage of impurities is always accompanied by a modification of the colour of the alloy.

It is believed, then, that the colour of brass drillings may be utilised to give rapid approximate indications of copper percentage, correct to within 0.5 per cent., or, with care, to within 0.25 per cent. But the following conditions must be observed to ensure accuracy:—

1. The samples and standards must be similar; this offers no difficulty if the same brands of copper and zinc are employed—and the same class of scrap—so that the only appreciable variables in the alloy are copper and zinc. In applying the test to alloys varying greatly in respect of purity, different sets of standards must be arranged to correspond with the metal under examination.

2. The method of drilling the samples should be uniform in regard to depth of cut and rapidity, and as far as practicable to the sharpness of the tool.

3. The formation of tarnish must be guarded against, whether it arise from heating during drilling or from subsequent exposure to the air. The samples should therefore be drilled slowly and protected from undue heating; while the standards should be preserved in sealed tubes, and the specimens under test should be examined as soon as possible after drilling; if delay be necessary they should be kept in well-closed tubes or bottles.

4. The specimen tubes should be of uniform size and colour.

The colour test is not therefore suitable for use in a general laboratory to which alloys from unknown sources are constantly supplied, and from which accurate results are expected. But it might be useful to the manufacturer who

is called upon to make cartridge metal or other brass to specification, if he use the same brands of raw materials, as the occasional analyses of the output may be supplemented by colour observations with any number of intermediate samples, inasmuch as the test is very rapid and requires no expenditure upon reagents. When the copper-zinc alloys are made in reverberatory furnaces, the application of the colour test would enable the melter to ascertain the composition of his bath within a few minutes, and thus to bring it to any desired composition by the addition of a known weight of either metal. To effect this, a fair average sample would be withdrawn from the bath, poured into a small open mould, cooled, drilled, and examined.

Only 60 trials in all (39 with bottles and 21 with tubes) have been made in this manner, and the results of all these are included in the above description. It is quite possible, therefore, that with due observation of the precautions laid down, a closer approximation than is here recorded could be secured.

#### DISCUSSION.

Dr. C. R. ALDER WRIGHT said Mr. McMillan had given the metallurgist a very valuable hint as regarded the possibility of getting rapid information as to the composition of alloys in the course of formation by physical tests such as the colour test of the drillings. In this particular case he believed the scientific basis of his proposition was that a definite compound of copper and zinc existed containing two atoms of copper and one of zinc, forming a definite molecular compound as distinct in its character as  $\text{SO}_3$ ; so that if you had a mixture of copper and zinc exactly in that proportion you had a single definite entity, but in actual practice this was a very exceptional occurrence; probably one or other of the two constituents was in excess, and there might be a small quantity of other impurities, such as lead, iron, or tin. But whether there were an excess of copper, or of zinc, or something else, the effect was more or less marked according to the circumstances not only in the physical property of the colour of the light reflected from the freshly-cut surface, but in various other respects. As he understood, the series of experiments were made on the one hand with an excess of zinc varying from 1 per cent. up to 2 or 3 or more, which gave a certain series of colour values, and on the other side with a corresponding excess of copper over and above the atomic compound which gave various other shades of tint in the opposite direction. It was by no means to be supposed *a priori* that corresponding quantities of other metal such as lead, iron, and tin, would produce exactly the same alteration in shade, and therefore, as the author had pointed out, a series of standards applicable to one set of alloys of a given character and standard of purity would not necessarily be applicable without certain alterations to metal containing other impurities. Still the figure he had given showed that a scale was possible of formation which might be of very great practical value to the metallurgist in general. It was noticeable in connection with these alloys that the yellow shade of brass entirely disappeared when the quantity of zinc present exceeded a certain amount. He had been working for some time past in connection with alloys, and in reference to this particular pair of metals had found that if you took zinc just melted at a temperature a little below that of boiling sulphur and stirred it up with fragments of copper which had been previously coated with zinc by immersion in an acid solution of zinc chloride, and then dipping into melted zinc, the zinc would only dissolved at that temperature a limited amount (a few per cents.) of copper, and you had a perfectly white alloy. At a slightly higher temperature the zinc would still remain white. In making a series of solubility determinations at various temperatures there were great practical difficulties in getting saturated solutions, whilst avoiding super-saturation; but assuming you could get the values fairly correct it was possible to plot out a curve

\* It has been suggested by Mr. Thorp that the colour of the brass might change during the first few hours after casting owing to a molecular alteration. I have therefore made up a sample, and examined it within 10 minutes of solidification—and again after a lapse of 24 hours, and have not been able to detect a change—although the colour varying a little from that of my standard specimens, comparison was not quite so easy as it would have been with the same brands of copper and zinc; the metals used for this test were less pure than those employed in India.

where the temperatures were shown by the abscissa, and the amount of metal dissolved the ordinates, and in that way it could be readily shown that at a comparatively low temperature the amount of copper taken into solution by the zinc was small, but gradually increased at an increasing rate until by and by you got perfect miscibility in all proportions of the two metals. This appeared to be not merely a property of copper and zinc, but one common to the majority of metals. On cooling down a saturated solution, however, you did not always get the reverse action taking place. There seemed to be a peculiar physical character inherent in most metals, tending to prevent an excess of matter which was dissolved at a given temperature from separating out on cooling in the same tolerably rapid way that a saturated brine at 100° would deposit the surplus of saline matter on cooling down to a lower temperature, and similarly with other watery solutions of salts. Metals seemed peculiarly prone to take into solution at a given temperature certain quantities of other metals which were not thrown rapidly out of solution on cooling. In point of fact the results he had arrived at so far led to the belief that the great bulk of alloys with which they had to do in ordinary life were more correctly described as solidified *supersaturated* solutions than anything else. He did not know that there was an exact analogue to that in watery solutions. You could obtain a supersaturated solution, say, of sulphate of sodium which, until particles acting as nuclei got into it, did not solidify; but when it did, it did not solidify *as a whole*, but separated into two things, one the solid particles of hydrated salt which came out of solution, and the other a watery solution saturated at the particular temperature obtaining at the time of observation. You did not get that sort of thing with a number of these metallic solutions or alloys. For example, if you took the alloys of copper and tin, at the temperature of melting tin, the tin would only dissolve a few per cents. of copper; at a higher temperature rather more; whilst at the fusing point of copper the two metals were miscible in all proportions. Now, taking a mixture of copper and tin, say, of equal weights, forming a saturated solution at the temperature of melting copper, if you cooled it down quickly you got a perfectly homogeneous mass, which to the microscope and all physical tests was absolutely uniform in structure. But if you cooled it slowly you had a separation into two materials; one a solid substance separating out which in all probability was a compound of copper and tin of a uniform definite atomic character analogous to the compound of copper and zinc in the case of the alloys examined by Mr. McMillan; the other remained fluid, and was a solution of copper in tin, which contained not only the amount of copper appropriate to the prevailing temperature, but also such an additional amount as the conditions might cause to remain in supersaturation. Similarly, in many, if not in most, analogous cases, as was well known to everyone who had to do with the cooling of mixed metals, you might get a mixture uniform or non-uniform in texture, according as the cooling was quick or slow. The fundamental principle at the bottom of all this was that there were certain circumstances, amongst which the presence of foreign matter was to be reckoned, which caused delay in the separating out of the matter which was in excess in solution at the moment of solidification, so that what you got was a material which, if it were to be compared with solution of sulphate of soda would be more akin to one of Guthrie's cryo-hydrates than to the mixture of crystals of sulphate of sodium, and the watery solution saturated with salt which one got in the ordinary experiment. This physical condition of supersaturation of solutions was a matter which it appeared to him was a considerable interest to all who had to deal with the alloys in question, and these particular experiments with reference to this special alloy had a very important bearing on the subject viewed from this point.

Mr. W. T. REID asked Mr. McMillan if he had made any experiments in comparing plane polished surfaces of metal. It seemed to him that the production of the shavings in the first instance might possibly produce a source of inaccuracy in unpractised hands. If it was all done by the same person no doubt the results were comparable, but it seemed rather a difficult thing to get shavings

of exactly the same character. Filings appeared to be a better form, and he should like to know whether experiments had been made with them because they could be produced of regular quality. With regard to the durability of the standard, he should like to know how long it had been in use, and also if such standards could be kept for any length of time so as to be available for future years. He had had occasion to make some experiments on metals and alloys of a darker colour than these, and found it very useful to take the streak, as was well known to goldsmiths. He first tried the streak on unglazed porcelain, but found that was not very satisfactory, and was about to give up the idea when it occurred to him that he might try a quartz surface, and on trying a surface of white quartz he found it extremely satisfactory. For some reason he could not explain this gave a colour clearer and plainer than unglazed porcelain. He then tried surfaces of dull ground glass of different colours, and was able to get very satisfactory indications, though he had not followed them up further. With regard to that interesting alloy, which possessed a different colour from the other, could Mr. McMillan tell them whether it was ductile or if it differed in that respect from the other brasses which were compared with it?

The CHAIRMAN said he was much interested in Mr. McMillan's papers when he first saw them, for he had been kind enough to send them to him some few days ago. In passing, he might venture to say that it would be well if the authors of all papers would send them to the chairman of the Section, whoever he might be. With regard to the latter paper especially, he was interested because the numbers obtained were exceedingly close. The author had carried out the experiments very fairly and carefully, making his sight comparisons before the analyses, so that there could be no kind of bias in the matter. Such approximate methods of examination were undeservedly neglected. In a large number of processes, with care to secure similar conditions, much might be done by careful inspection and comparison. In this case the author had very precisely laid down what those conditions must be. The materials must be of the same kind, and so forth; in fact the only variant should be the proportion between the two constituents of the brasses.

Mr. W. G. Mc MILLAN, in reply, said he had tried several experiments with a view to determining the best manner of observing the colour of the metal, and he distinctly preferred the result obtained with the mixed reflections, partly from the rough surface, and partly from the smooth. He had tried several alloys with polished surfaces, but although there were differences in colour, it was impossible to read them so accurately as in the case indicated. The fracture of the ingot would give a much better reading than the polished surface, but the best of all was obtained from the combination of the two. He had not tried many experiments with filings, but as far as he had observed, they were inferior to the borings taken by the ordinary boring tool. The samples exhibited were made over a year ago, and he did not apprehend they could change colour, unless from molecular alteration, because they were in sealed tubes. The alloy containing 1 per cent. of lead had a distinctly different character from that of the ordinary alloy; the turnings came off in finer flakes, which crumbled up readily on rubbing. With small quantities of lead the drillings were normal. In conclusion, it seemed to him that these experiments gave additional evidence of the effect of small quantities of metal in altering the physical characteristics of alloys, and indicated very clearly that if one wished to ascertain the true character of definite alloys, it was necessary to ascertain initially that the metals were absolutely pure and free from even traces of any impurity.

## THE DISTILLATION OF PEAT.

BY P. DVORKOVITZ, D.Sc.

It is with very great reluctance that I venture to approach the subject of peat treatment in face of the strong prejudice which exists against any method for its profitable utilisation; moreover, geologists and botanists having after careful research ignored its usefulness, it remains for the chemist to determine whether it can be turned to profitable account; hence with this object in view I have recently visited Ireland and, supported by considerate Government influence, have inspected the principal peat districts, procured reliable samples, and I propose to place before you as concisely as I can the result of my experiments.

Although peat has been known so long ago as B.C. it is only lately that it has excited a certain interest, it having been brought before your notice in very able addresses by two of your presidents; in 1889 by Mr. Ludwig Mond, and in 1892 by Professor Emerson Reynolds, and I think that as our Society has for its main object the furtherance and development of chemical industry in the United Kingdom, a few remarks on the development of the distillation of peat may interest you.

Before showing how peat could be treated, I will explain what it really is. Generally, opinions as to the origin of peat are diverse. First, that peat is mainly received by the decay of forests, and this view is supported by Mr. H. O'Hara in his paper read before the Royal Dublin Society in 1864. He says that abundant proofs exist that peat bogs in Ireland are mainly formed from forests, and that formerly Ireland was one vast forest. In times of warfare broad tracts were cleared to facilitate military operations, and extensive woods were consumed by fire. Immense quantities of timber were likewise consumed by the forges and iron furnaces, which at various times were in a state of great activity. The remains of these ancient bloomeries which are found in the counties of Antrim, Leitrim, Roscommon, Sligo, Tyrone, Killarney, Carlo, Tipperary, Limerick, Kerry, and Cork, prove that smelting of iron was very generally known in Ireland previous to the exhaustion of wood.

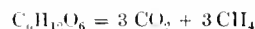
The destruction of the forests caused a considerable change in the climate and a still greater change in the soil; indeed the most remarkable fact has been the conversion of nearly one-seventh of Ireland into a swamp. This results from the greater exposure of the surface of the land to the moist winds of the Atlantic which are highly favourable to the growth of a species of moss known as *Sphagnum Palustre*. This moss thrives only in exposed situations and so favourable is the condition of Ireland to its propagation that if the existing arable and pasture lands which have a south-westerly exposure were abandoned to nature, most of them would be covered by peat moss to a depth of several inches in the course of a century.

Captain Portlock, in his account of the survey of Londonderry, is of opinion that sphagnum is acted upon by the superabundant moisture of the climate in inducing the formation, and this opinion is supported by Mr. Aher, who shows that trees are found generally six or seven feet above the bogs, standing as they grew, conclusively proving the foundation of peat to have been prior to the growth of the trees; a fact which in relation to fire may be verified in every bog in the parish of Donegal where turf exists from three to five feet underlying the layer of such trees.

Further Messrs. Nimmo and Griffiths in their bog report are of opinion that the strong resemblance to ancient water courses of the valleys and basins which now contain the bog, and the accumulation of marl and shells at the bottom of the moss naturally suggest the idea of shallow lakes. Such lakes may have originated in natural inequalities of the ground or been formed by the choking up of channels of transit by heaps of clay and gravel, or they may have been reduced to a condition of shallowness by the gradual wearing away of the obstacles which had blocked up and retained their water at a higher level. In all such cases the origin and formation of bogs would be as follows:—A shallow pool induces and favours the vegetation of aquatic plants which gradually creep in from the borders towards the

deeper centre, mud accumulate around their roots and stocks in a spongy semi-fluid mass specially conducive to the growth of moss, which latter, particularly sphagnum, begins to luxuriate, thus absorbing a large quantity of water and tending to shoot out new plants above, while the old ones are decaying, pressing the lower ones into a solid substance, and gradually replacing the water by a mass of vegetable matter. In this manner a marsh might be filled up, while the centre moisture portion continuing to excite a more rapid growth of the moss, would gradually rise above the edges until the whole surface had attained an elevation sufficient to discharge the surface water by existing channels. Springs existing under the bog would raise the surface so high as to cause it to flow over the retaining obstacle and flood the adjacent country.

The last theory as to the formation of bog from sphagnum only, I think is the most interesting, and this is corroborated by the experiments made by Mr. Joseph Boelm in the year 1875 about the fermentation of marsh and aquatic plants. He investigated the character of a large number of different plants and has come to the following conclusion. *Firstly*, that a large number of marsh plants, for example *Berula Angustifolia*, *Nasturum Officinale*, &c., if under water for any considerable time undergo a process of fermentation. *Secondly*, that aquatic plants and a large number of the marsh plants develop marsh gas. *Thirdly*, that the development of marsh gas from the decaying plants must be taken as a consequent result. This fermentation arises from unknown yet bacilli, which are very sensitive to high temperatures. *Fourthly*, the destruction of the cellulose of the plants by fermentation could be explained by the formation from one part of cellulose of three parts of carbonic acid, and three parts of marsh gas—



By this experiment the amount of carbonic acid has been less than it should be under the formula, and this is accounted for by the presence of ammonia with which a portion of the carbonic acid is combined, and *lastly*, and most important, that by a prolonged continuation of such fermentation a certain amount of turf is formed. Mr. Frey in 1879, in *Comptes Rendus* XXI., has expressed the opinion that plants have been first transformed into peat, and afterwards into coal, and that this process of transformation is the result of fermentation. I think that only upon this theory of the formation of peat from plants belonging to the family of sphagnum could be explained such facts as are known about the renovating of peat bogs. Already about 150 years ago, the Earl of Cromarty demonstrated that moss could be renovated if the bogs were cut down to the bottom and the moss peat from above filled in again, when in the course of years the bog will grow up again. Mr. De Loe who has had a very great experience at the beginning of this century in the surveying of peat bogs all over Europe has expressed his full belief in the possibility of growing up peat bogs in a comparatively short space of time.

Mr. Waggemann, in 1828, made experiments in the artificial production of peat from the plants of sphagnum, which he placed in brick tanks three feet high, filled with water, and carefully covered, leaving them to undergo fermentation, which resulted in dark brown peat the following spring.

As it is shown by the Table I, the area of the bogs which are spread over Ireland amounts to 2,830,000 acres. Of this quantity 1,816,642 acres lie in the mountains and hilly districts near the coast, and the remaining acres, viz., 1,013,358 extend across the great limestone plain, and contain almost an inexhaustible supply of peat.

In the Table I I have shown that the chief bogs are sufficiently high above the level of the nearest rivers and lakes, and therefore they could easily be drained.

One fourth of the entire superficial extent of Ireland between a line drawn from Wicklow Head to Galway, and another line drawn from Horoth Head to Sligo, comprised within it about six sevenths of the bogs of Ireland, exclusive of mountain bogs and bogs less than 500 acres. This division of Ireland from east to west is traversed by the river Shannon from north to south, and were the bogs to be divided into 20 parts, 17 of them would be found between

TABLE I.  
EXTENT OF THE BOGS OF IRELAND.

Districts.	Counties.	English Acres.	Depth.		Elevation above Sea at Low Water.		Rivers and Lakes into which the Bogs could be Drained.	Height of the Rivers and Lakes above Sea at Low Water.
			Greatest.	Average.	Greatest Height.	Lowest Height.		
Eastern part of Bog of Allen.....	Kildare .....	36,730	41	22-25	312	228	Troglit and other streams falling into the Barrow.	At Monastereven, 298 ft., 4 in.
Western part of Bog of Allen called District of the Barrow.....	King's and Queen's.....	41,075	41	22-25	329	263	"	At Ashy, 188 ft., 1 in.
The Ballyne .....	Meath and Westmeath .....	42,370	40	22-25	350	232	Streams discharging into Boyne..	At Edenderry, 291 ft., 7 in.
Bresna .....	King's.....	44,594	45	22-25	314	125	"	At Breauna, 153 ft., 7 in.
The Shannon .....	Westmeath, Longford.....	34,500	44	30	28	128	Blackwater, Shannon, Camlin and Lony.	At Fethlack, 111 ft., 4 in.
The Inny and Lough Ree.....	Longford and Westmeath.....	34,530	47	30-35	268	191	Streams discharging into Lough Corrib.	Lough Corrib, 175 ft.
Lough Gara .....	Roscommon, Sligo, and Mayo.....	83,680	45	20-30	415	204	Streams discharging into Lough Corrib.	Lough Corrib, 175 ft.
Between Rosreen and Killymule.....	Tipperary, Kilkenny, and Queen's.....	36,925	33	16-20	418	340	Streams discharging into Lough Corrib.	Lough Corrib, 175 ft.
Westward of Maryborough.....	Queen's.....	14,754	35	18-20	340	288	Streams discharging into Lough Corrib.	Lough Corrib, 175 ft.
Western extremity of county Clare.....	Clare.....	22,310	35	15-20	130	64	Dunbeg, Ac.	Lough Corrib, 175 ft.
Banks of the Barrow .....	Kildare and King's.....	7,459	36	20	257	227	Barrow.	Lough Corrib, 175 ft.
Lough Corrib.....	Galway and Mayo.....	86,724	45	15-20	564	45	Streams discharging into Lough Corrib.	Lough Corrib, 175 ft.
Three districts.....	Mayo and Sligo .....	161,962	51	8-16	188	51	Lough Mask, Lough Corrib, and Lough Corrib.	Lough Mask, 18 ft., 1 in.
Surrounding Lough Neagh and extending to the River Bann.....	Antrim, Down, Tyrone, Armagh, and Londonderry.....	61,865	..	..	200	163	Blackwater, Bann, and Lough Neagh.	Lough Neagh, 10 ft.
Keshore River.....	Kerry.....	64,567	25	5-12	250	75	Cashen, Ac.	Lough Neagh, 10 ft.
Lawman, Lower Maine .....	" .....	11,905	20	20	30	200	Blackwater, Ac.	Lough Neagh, 10 ft.
Upper Maine .....	" .....	17,900	20	6	160	25	Lakes of Killarney.....	Lakes of Killarney, 10 ft.
Slieve Donard.....	" .....	8,566	22	10-15	200	38	Gleeson and Lough.	Lough Neagh, 10 ft.
River Coshin.....	Kerry and Cork.....	32,902	32	6-12	700	250	Blackwater and Coshin.	Lough Neagh, 10 ft.
Lough Ree .....	Kerry, Northern part.....	31,517	32	12-20	140	75	Coshin.	Lough Neagh, 10 ft.
Southern extremity of River Suir.....	Longford, Leitrim, and Roscommon.....	26,630	43	20-30	220	141	Shannon.....	At Harbourn, 113 ft., 4 in.
Northern extremity of River Suir.....	Galway and Roscommon.....	78,818	39	20-25	284	126	Lough Corrib, Mask, Gara, Suir.	Lough Corrib, 16 ft., 1 in.
Mountain bogs and bogs less than 500 acres not included in the reports.	" .....	72,300	20	15	300	150	Suir.....	At Rathfriland, 116 ft.
	Kerry, Sligo, Galway, Wicklow, Ac.	1,053,358						
		1,815,642						
	Total.....	2,830,000						

those lines, 12 parts west of the Shannon, 5 east of the Shannon, and of the remaining three parts, two are south, and one north of the division.

If we take the average depth of the bogs of 20 feet, which, according to the Table III., contains in the average 64 lbs. per cubic feet. We will find that each acre contains 2,700 tons, or about 7,440,000,000 tons of very dry peat is on the bogs of Ireland.

Professor Reynolds is of opinion that the stores of peat are only an asset which may become valuable when the coal beds have been exhausted after 170 years. But certainly it is in the interest of the owners of the bogs, and all the Irish people to anticipate the consequent benefit, before the expiration of such period. It is also a matter of very great importance to the prosperity of that country to utilise so serious an amount of waste land.

We find that the British Government turned its attention to this question at the commencement of this century, a special commission of surveyors having been appointed, and about 50,000*l.* spent in surveying and preparing plans of the Irish peat bogs. I do not know for what reasons, but

the further investigation was abandoned, and no practical result obtained from the very valuable reports and maps now quiescent in the Government archives.

I show here five sections of peat bogs taken by the Commissioners appointed by the English Government; the first representing a section of turf bank exhibiting marl on bog. As is seen from the drawing, in the centre of the bog there is a layer of marl, the bottom being limestone gravel. The next represents marl on the surface of the bog with the root of a fir tree well preserved. The third represents a section of two growths of trees on bog. The fourth a section of a renovated pit, showing from the other side the method of cutting drains. Regarding renovating pits, sometimes old pits and turf hill are found filled with a new growth of moss, the surface appearing to have been sunk in the centre on being deprived of its water. This new growth consists of some of the varieties of the *hipnum* and *sphagnum* and seems perfectly distinct from the original formation with which it is in contact, and in some cases is not even the same variety of plant. No part of this new formation has yet undergone the process of decomposition,

Fig. 1.

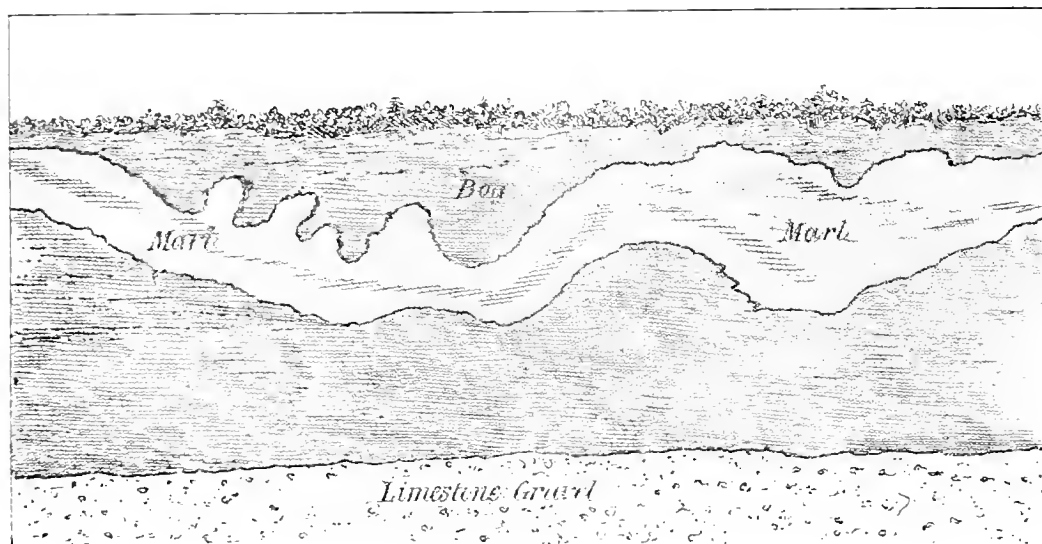
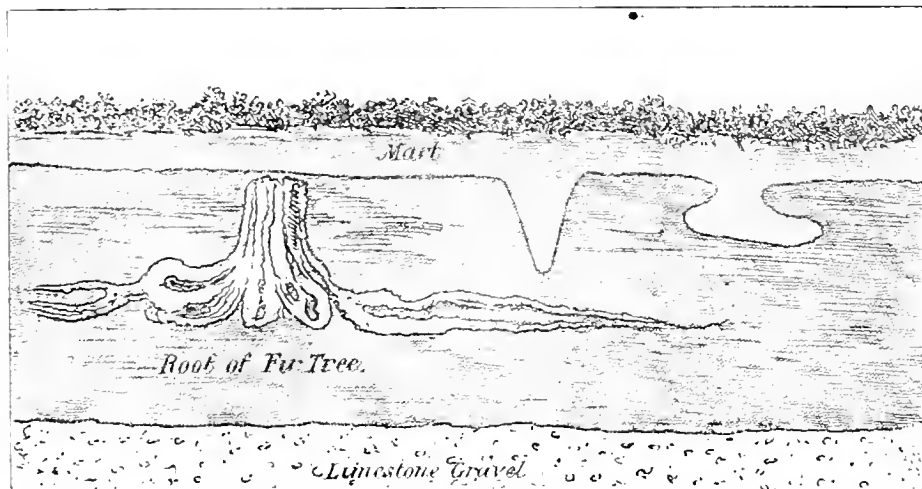


Fig. 2.



or even far advanced towards a state of decay. There are other pits in which the process of renovation appears to be going on, where the tender fibres of the convolvulus are spreading like a green film on the surface of the stagnant water. This, by interweaving with other aquatic plants may form a receptacle for the deposition of the light seeds or mosses which are blown about in abundance at the shedding season.

Figure 3 is a section of turf bank representing three distinct growths of trees. This bog is 12 feet high, and the lowest part represents the roots of trees based on limestone gravel. Above these roots is about 4 feet of compact black peat or strong turf. This constitutes the best and most durable fuel, is very hard, and has a high specific gravity. Above this black peat you will see roots of fir trees again well preserved. Beyond these we have another 4 feet deep

Fig. 3.

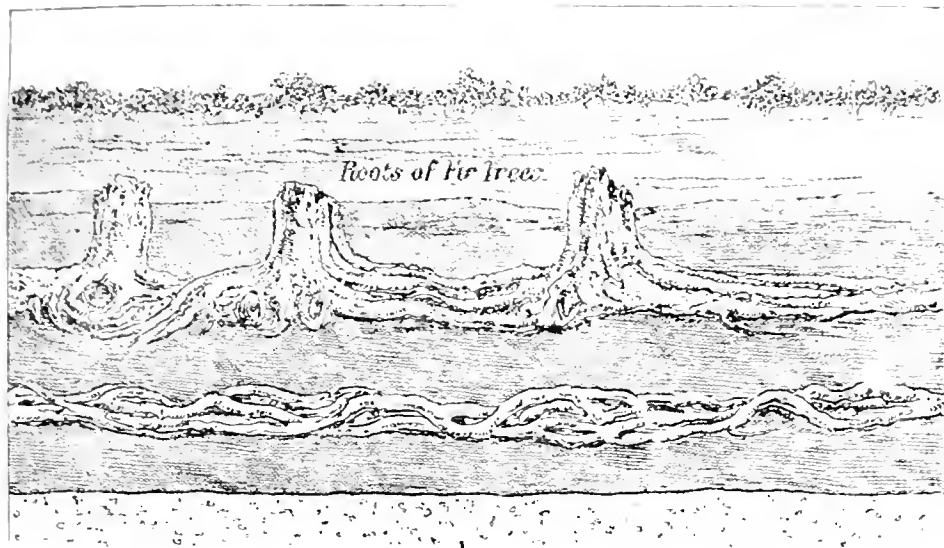
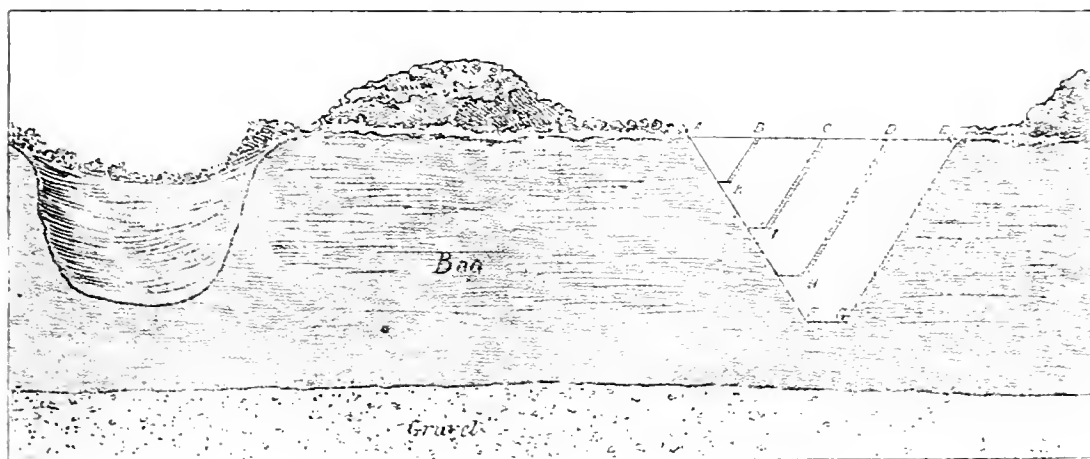


Fig. 4.



blackish peat or turf, and, further, roots of young fir trees, on the surface of which exists a layer of moss grasses. The fir roots and trunks possess a high degree of inflammability from the resin they contain, and when dried are used by the peasants in place of candles.

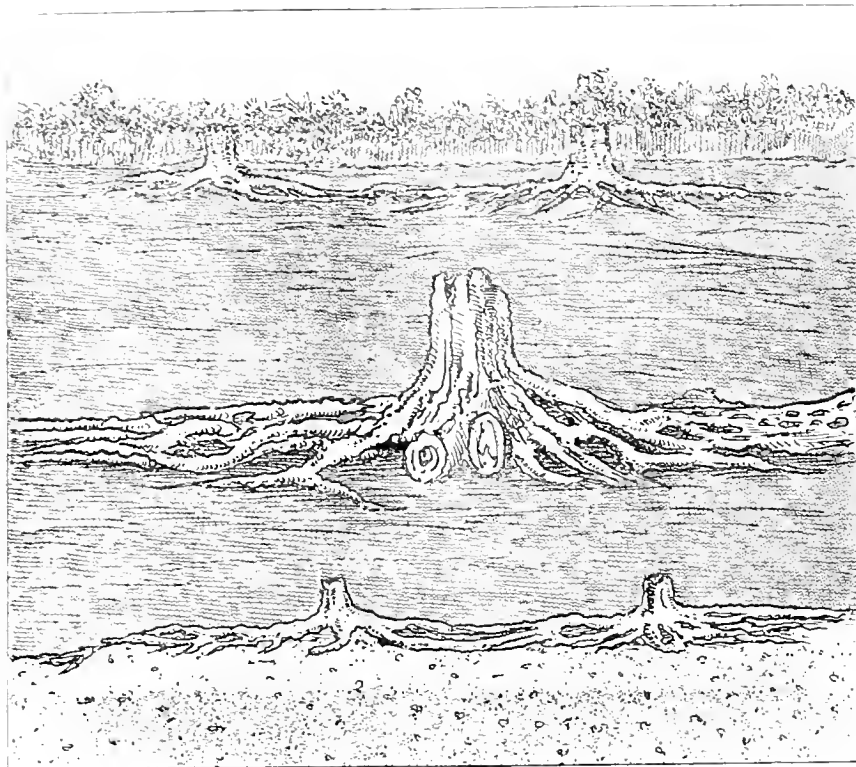
The question presents itself is peat advantageously convertible for industrial purposes? If we turn our attention to the development and use of peat in Europe, we discover that it is used in very great quantities in different industries. Already in 1856 in Germany the Aldenburg Iron Company was established and has consumed not less than 20,000 tons of peat per year, and notwithstanding coal existed in the immediate neighbourhood, and very profitable results

followed. Not far from the works of this company, in 1873, another company was established for steel manufacture by means of peat charcoal. Further, we find that in 1890, 27 glass works in Germany used peat fuel, one ton of glass consuming eight tons of peat. A Mr. Peuch at Berlin said that one ton of ready-made bottles (1,600 ordinary wine bottles) required only  $2\frac{1}{2}$  tons of peat dried in the air; or if we take 1,000 sods of peat as equal to  $3\frac{1}{2}$  tons, we find that one ton of bottles required 700 sods. A glass-melting stove with eight pots having a charge of 100 kilos, each, consumed  $4\frac{1}{2}$  tons of peat per day. In Bavaria about 60,000 tons of peat are used annually as fuel for railway locomotives.

In the report prepared by the Russian Government for the Exhibition of 1893 at Chicago, certain figures are given about the utilisation of peat for different manufacturing purposes; and we find for 1890 the following industries

have used peat as fuel, viz., the cotton manufacturers have consumed 537,000 tons; sugar manufacturers, alcohol manufacturers, confectioners, flour mills, and macaroni manufacturers, 70,000 tons; chemical manufacturers, 5,000

Fig. 5.



tons; candle, tallow, and leather manufacturers, 4,000 tons; wood-workers, 1,000 tons; tael manufacturers, 60,000 tons; glass manufacturers, 80,000 tons; paper manufacturers, 12,000 tons; miscellaneous manufacturers, 2,000 tons; aggregating approximately 772,000 tons. In addition, the Oural mines used 60,000 tons and the railway companies 15,000 tons, with prospectively an increased demand, proving conclusively the value of peat as fuel.

The use of peat for moss litter has greatly increased both in England and on the Continent during the last few years, and forms a most important branch of the industry. It is also largely used in Russia and on the Continent for earth closets and other sanitary purposes, for which, from its antiseptic properties, it is especially suited. These properties are also utilised for the preservation of fish, meat, and eggs in transit, and its non-conducting properties have rendered it useful for the preservation of ice. Lately attention has been paid to the utilisation of the fibres of the top peat for making paper-pulp, felt soles, and in substitution for shoddy in the manufacture of horse-clothing and other cheap cloths.

Professor Reynolds in his paper showed that peat in its ordinary condition is a very bulky fuel, occupying more than five times the space of an equal weight of coal; that it contains from 15 to 25 per cent. of water and seldom less than 10 per cent. of ash, and that at least 2½ lb. of Irish peat is required to perform the same work as 1 lb. of Staffordshire coal in an ordinary fireplace or furnace. All these disadvantages could be easily removed by more careful treatment of the peat itself. Reduction of the bulk and the removal of the water could be done by partial carbonisation.

Carbonisation of peat is a very old question, and generally speaking can be described under the following heads:—

*First*, carbonisation in heaps.

*Second*, in closed ovens of brick and iron in which the peat is lighted, and after it has sufficiently formed a good flame the oven is closed, air excluded, and the carbonisation goes on.

*Third*, the carbonisation in closed retorts heated from the outside.

*Fourth*, carbonisation by superheated steam.

*Fifth*, carbonisation by burnt and consumed gases.

*First*, carbonisation by heaps. As early as 1712 Mr. Carlowitz in his "Sylvicultura Economica" proposed to carbonise peat in heaps, but no important steps appear to have been taken until 1836, when Mr. Schmidt introduced it into the Lavariao State iron manufactories. This method was to pile up heaps of 2,500 cubic feet capacity; the quantity of peat carbonised was stated to be 13½ tons, and the amount of peat charcoal obtained 3 tons 8 cwt., or 25·2 per cent. The carbonisation of such heaps and the cooling down required about 12 to 14 days.

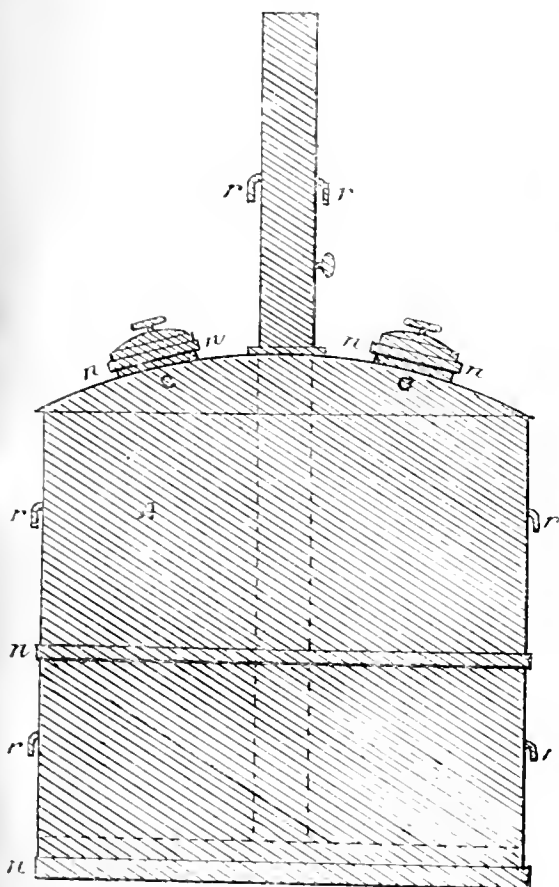
*Second*, in closed ovens. The oldest and best known oven has been constructed by Mr. Lange in 1745. As it is seen from the drawing, this oven consists of a square foundation (a a) on which an iron plate (b b) is placed, in which (b) a square hole of 15 inches is made; (c) is the grate, which is closed by a door. On the plate is based an iron cylinder (d), above (d) a second one (e), and above (e) a conical stone (f) is placed with an opening (g) 16 inches diameter.

In 1767 an oven of new construction was built on a round foundation of 17 feet diameter, and from 2 to 4 feet high,



on which has been placed a round brick cupola of 16 feet exterior diameter, and 4 feet thickness. In the centre of this cupola a cylinder of 6 inches diameter has been built up filled with sand and ashes. At a height of 13 feet, the oven finishes with an opening of 4 feet diameter. Both ovens, however, proved a failure. The peat lying above crumbled down the charcoal formed below it, and to avoid this Mr. Hahnemann proposed the following construction as seen by the drawing. A round cupola of 16 feet high and 7 feet diameter, with a thickness of the walls of 2 feet at the bottom and 10 inches at the top, on the foundation the grate (*f*) is placed, and connected with an opening (*k*) for taking out the charcoal. At the commencement of the charring, the opening (*k*) is closed, and the cupola is filled up with peat and fired from the top. When the peat has commenced to burn sufficiently, then the top of the opening of the cupola (*a b*) is closed, and thereby the gases produced are bound to pass down through the mass of the peat and through the opening (*f*) and pipe (*g*) from the retorts. The products

Fig. 6.



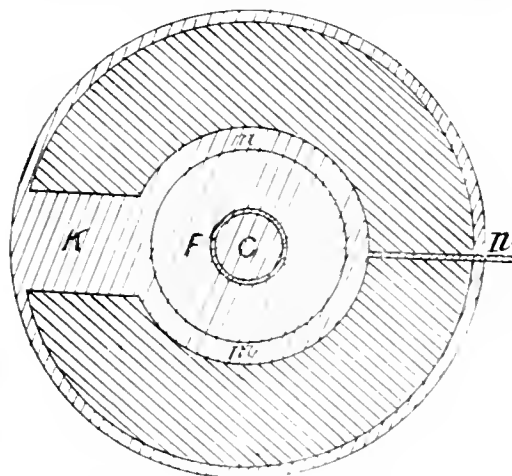
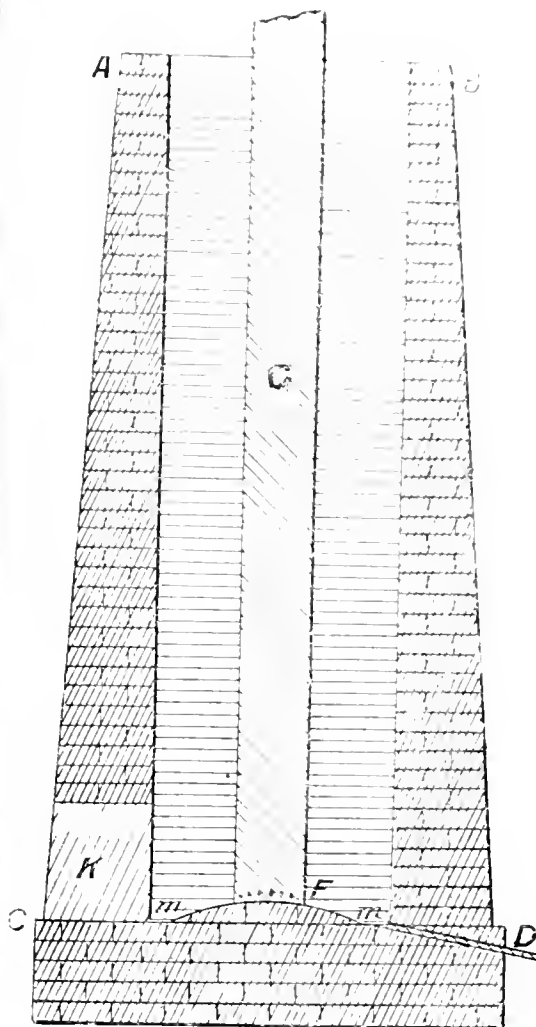
FIRST PEAT OVEN CONSTRUCTED BY LANGE, 1745.

of this distillation are condensed in the condenser. When the fire in the cupola has come down nearer to the opening (*f*) all the openings are closed and the stove is left to cool down.

On the same principle is based the stove of Moreau, Père and Fils, a model of which has been exhibited in the Paris Industry Exhibition in the year 1855. This apparatus consists of a cylinder of sheet iron eight feet in diameter and the same height, such stove carbonising in 24 hours three tons, and producing 40 per cent. of charcoal. In 1851 a special process was invented by Mr. Reece, in which process the British Government took very great interest, and appointed a special commission to investigate with a view

to establishing profitable peat industries in Ireland, and the Irish Peat Co. was then formed. A site was chosen in the

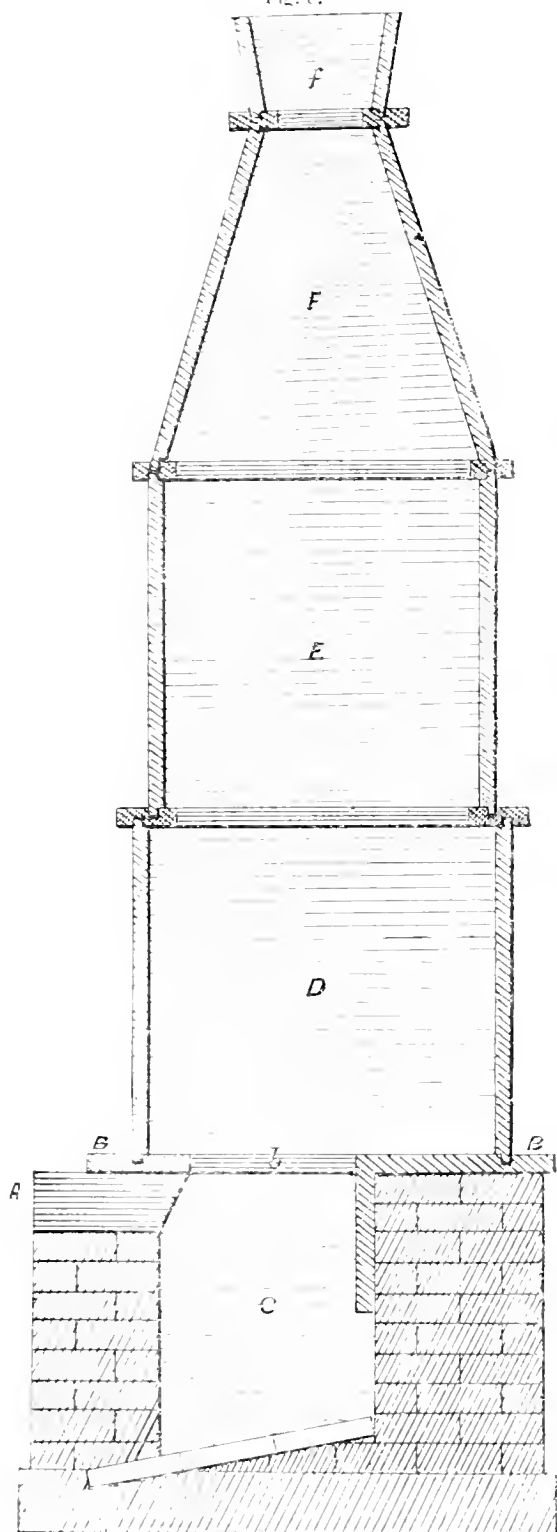
Fig. 7.



IMPROVED PEAT OVEN BY HAHNEMANN.

North of Ireland, called Kilberry, about four miles from the town of Arly in connection with the Carlo branch of the

Fig. 8.



PEAT OVEN BY MORLAU AND SON, EXHIBITED AT THE  
PARIS EXHIBITION OF 1855.

Great Southern and Western Railway, while on the other side runs the navigable river Barrow and the canal to Dublin and Waterford. 500 acres of very excellent bog land were taken on a lease for 100 years at a rent of 5s. per acre. Mr. Scanlon, chemist, was sent by the Company to make experiments. He commenced working the furnaces at low temperature, and distilling the products by steam. From the information supplied by the Company it seems that from 100 tons of peat were received—

	£	s.	d.
1 ton of sulphate of ammonia .....	12	0	0
½ ton of acetate of lime .....	9	10	0
50 gallons of naphtha .....	12	10	0
300 lbs. of paraffin .....	15	0	0
300 gallons of volatile oils .....	15	0	0
	61	0	0

these being current value at the time. Cost of production of peat, acids, labour, etc., 32*l*.

The last practical attempts, so far as I know, were made in the year 1880. A company was formed in the name of the West of England Compressed Peat Co., which constructed 12 ovens for making peat charcoal at Rattlebrook on the slope of one of the highest hills of Dartmoor, nearly 2,000 feet high. The process which was adopted by this company was the invention of Mr. Kidd. The principle of his invention was the introduction of a jet of superheated steam, but nothing came of the scheme. The Duke of Sutherland was the first to take up Kidd's process, and in 1874 an experimental plant was erected on his estate in Sutherlandshire. The peat here is of very dense quality like the Dartmoor peat, and contains a large amount of hydro-carbons.

From this short description of the processes introduced for the utilisation of peat in one way or the other, it will be seen that most of the inventors have only had one end in view, whereas to gain a real practical success, we must work in the same sensible manner as we do with all other products. Most of the inventors have striven to produce only peat-charcoal, not taking notice of by-products which could be and should be received in course of charring, or they have concentrated their attention on the by-products without regard to the charcoal.

TABLE II.

## ANALYSIS OF PEAT.

From Barrow-in-Furness.

No.	Peat.		Charcoal.		Liquor.		Gas.
	Grms.	Grms.	Per Cent.	Grms.	Per Cent.	Per Cent.	
1	115	40.5	35.2	52	45.2	19.6	
2	127	45	35.4	50	39.3	25.3	
3	120	45	37.5	47	39.2	23.3	
4	105	35.5	33.8	43	41	25.2	
5	115	43	37.4	48	41.7	20.9	
6	111	50	35.5	50	35.3	29	
7	120	50	41.7	48	40	18.3	
8	112	46.5	38.1	33.5	43.9	18	
9	106	39	36.8	43.5	41	22.2	
10	134.5	50	37.2	57	42.4	20.4	
11	139	45	34.6	55	43.1	22.3	
12	140	44	35.9	49	42.2	19.9	
13	129	49	38	49	38	24	
..	1580.5	Max.	41.7	Max.	45.2	29	
..	..	Min.	33.8	Min.	35.3	18	
..	..	Avg.	36.9	Avg.	40.9	22.2	

In my investigation I have first tried to avoid all the conditions under which any of the constituent parts of the peat would be destroyed. I have applied in this the same principle as I have adopted in the destructive gasification of oil and of coal. This means using a very low temperature, and gasifying in the presence of an inert gas which could not have any chemical influence on the substances received, but which would have the mechanical effect of extracting all the by-products and leaving only pure charcoal in the retorts.

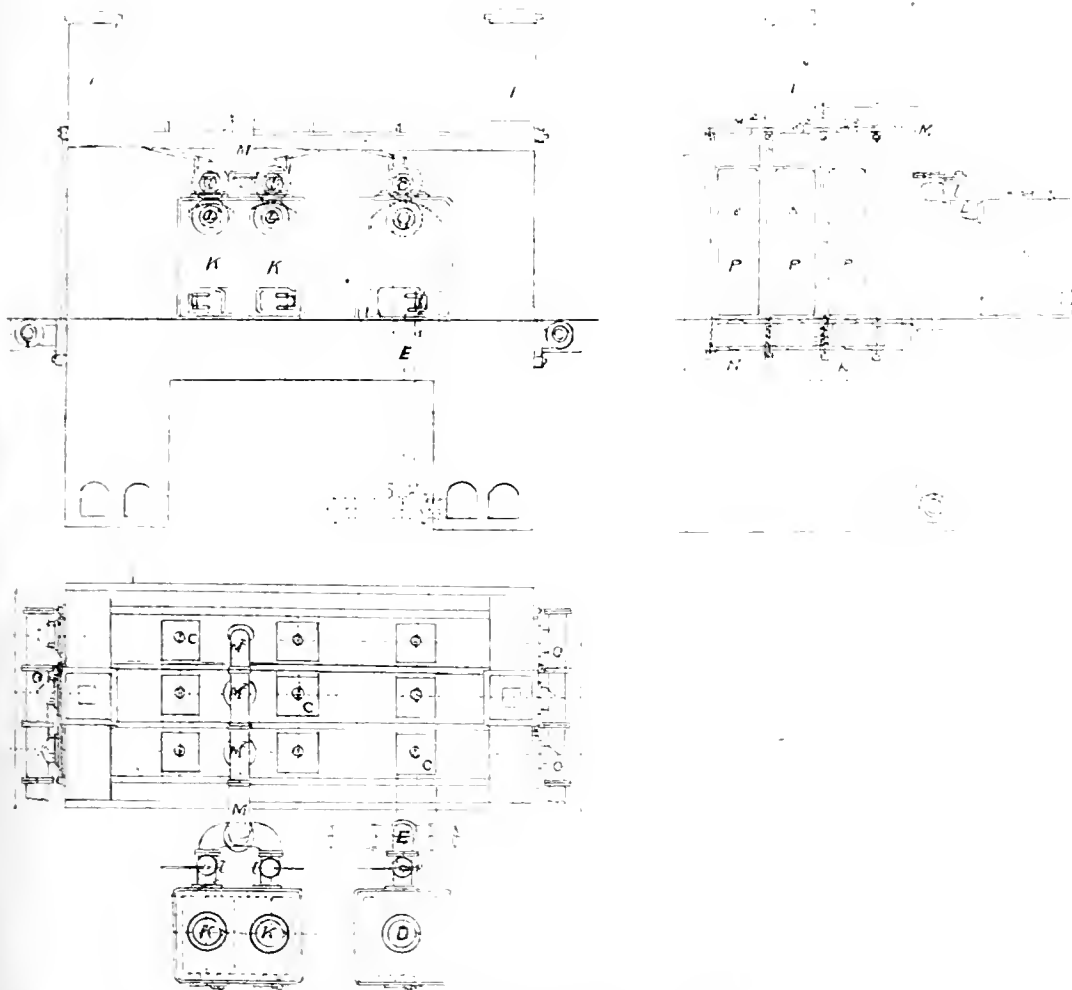
The apparatus with which I propose to treat the peat, consists, as you will see from the drawing, of two or more chambers or retorts, in which the peat is placed. The chambers or retorts are heated externally by generator gas from a furnace (*d*). The inside of the retorts or chambers is heated by water gas produced in a furnace (*k*) and passing to the upper part of such retorts. This water gas, which is admitted into the retorts at a temperature above the initial heat of the interior of the retorts, mingles with the matter under treatment, and rapidly separates the volatile constituents, which are conveyed from the passage (*m*) through the pipe (*o*) and then into the condensers in which the liquid by-products are condensed.

The charcoal contains 4.23 per cent. of ash. The liquor contains an average of 5.89 per cent. of tar, and 35.7 per cent. of water solution. The specific gravity of the liquor

is 1.0135. 100 cc. of the solution contains 0.2344 grms. of ammonia and 2.16 grms. of acetic acid.

From the Table 2, which is a result of 13 analyses made by my assistant Dr. Fuerst (to whom I now take the opportunity of expressing my thanks) I have received such results as applied in practical form will give the possibility of developing a large chemical industry in Ireland. The peat used for this analysis was sent down to me from Barrow in Furness, and was sufficiently dry. But to obtain a fairer sample and a practical knowledge of the peat bogs themselves I made a special journey to Ireland in April last, and after careful investigations took samples representing both lowland and mountain bogs. The first range of samples which I took (lowland) were from the estate of the Earl of Longford. This estate is situated at Killuenn, with the Royal Canal on one side and the railway on the other, and contains about 11,683 acres, which mostly consist of peat bogs. On this estate are about 340 tenants, who cut the peat for fuel, and afterwards reclaim the land. I also take here the opportunity of thanking the manager of this estate, Colonel Clark, for his kindness in showing me such a well conducted estate. He has been for 19 years fighting hard with the object of reclaiming the peat land for agricultural purposes, with splendid results, of which he has reason to be proud. From the view shown to you you will see that a large number of trees have been planted by him

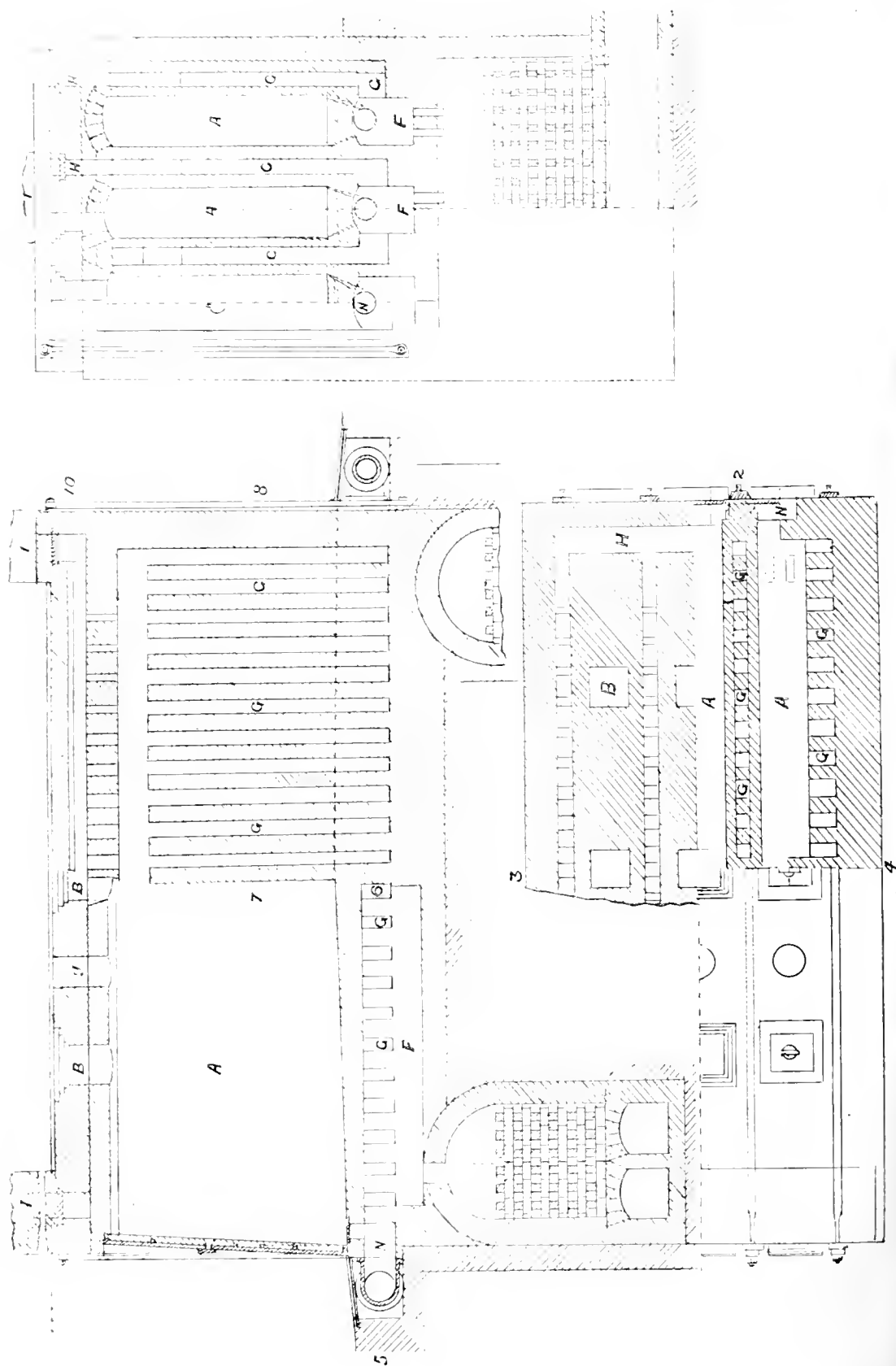
Fig. 9.



PEAT OVEN FOR RECOVERING BY-PRODUCTS.

\* Dyer's patent.

Fig. 10.



SECTION OF PEAT OVEN FOR RECOVERING BY-PRODUCTS.\*

on the peat bogs. It is true that the work demanded a large expenditure of energy, especially during the first years, but the results are so satisfactory that I am sure the gallant Colonel will never regret the pains taken, nor the owners the expense. The trees on the sides of the bogs are doing very well, but those planted in the centre do not thrive so well,

TABLE III.  
ANALYSIS OF PEAT.

A.—Peat from the Estate of the Earl of Longford,  
Killucan, Ireland.

No.	Weight of Wet Peat in Lbs. per Cub. Ft.	Volume of One Cub. Ft. after Drying.	Percentage of Water.	Weight of Dried Peat in Lbs. per Cub. Ft.
		Cub. Ft.		
1	48.45	0.753	90.5	1.60
2	51.91	0.413	88.26	6.44
3	55.81	0.416	86.9	7.31
4	60.89	0.4634	85.03	9.11
5	61.10	0.4165	86.80	8.96
6	55.45	0.5608	84.96	7.23
7	57.04	0.4165	87.4	7.19
8	50.77	0.4166	87.7	7.35
9	60.34	0.439	86.85	7.93
10	59.17	0.683	87.2	7.75

B.—Peat from the Estate of Mr. Atkins, Dunmanway,  
Ireland.

No.	Weight of Wet Peat in Lbs. per Cub. Ft.	Volume of One Cub. Ft. after Drying.	Percentage of Water.	Weight of Dried Peat in Lbs. per Cub. Ft.
		Cub. Ft.		
1	51.94	0.1876	84.93	8.28
2	51.36	0.4146	86.94	5.47
3	54.54	0.377	84.96	8.29
4	56.94	0.3281	88.51	6.54
5	57.12	0.377	89.59	5.94
6	61.45	0.2683	88.47	7.08
7	60.76	0.2193	87.73	7.45

C.—Distillation of Peat A. and B. Calculated on Dry  
Peat.

Peat.	Nos.	Char- coal.	Paraffin Oil.	Acetic Acid (Anhy- drid).	Am- monia.	Gas.	Water.
Peat A.	1 (top)	35.20	5.00	1.20	0.85	27.00	30.75
	5 (middle)	17.40	7.50	0.80	0.63	24.60	19.07
	10 (bottom)	44.00	6.75	0.75	0.67	19.50	18.33
Peat B.	1 & 2 (top)	59.25	4.73	0.67	0.77	27.20	16.58
	6 & 7 (bottom)	46.70	5.64	0.72	0.51	25.35	21.48

on account of the excess of moisture. The other samples, which represent the mountain peat, were taken from the estate of Mr. Atkins, at Dunmanway, County Cork. This peat is much blacker than the peat from the North. The difference between the top and the bottom peat is not so great, and the top peat has not the elasticity which we have seen in the case of Killucan. In such cases this peat should be used for distillation purposes only, but when we have the top parts light coloured, I think it would be more practical to treat them separately and prepare from them very valuable moss litter, leaving the lower layers for distillation.

From the results shown in the Table III, you will see that I have taken care to have a fair sample of the peat bog as it is. I have taken a section from the top to the bottom, so that my analyses represents the real nature of the peat. The higher strata has a lighter colour, which is chiefly due to the fact that it is not sufficiently decomposed. The main constituent of this top part is the real moss which is very elastic and like a sponge, having the quality of holding a large amount of water, the most of which you could press out by hand, after which the moss regains its original form and volume. When dried in the air, or artificially, it does not form hard lumps, but always remains soft and elastic. The analysis shows that whereas the volume of the lower layers has been reduced more than one half after drying, the volume of the top has reduced very little. The lower peat is generally very much decomposed, and after drying has not the property of reabsorption, whereas the top section will absorb the same amount of water which they previously contained. These results are in accordance with those obtained by Professor Fleischer, the director of the German official peat investigation station at Bremen.

As it is seen from the analysis, the difference between the low land peat and mountain bogs is chiefly in the producing of more charcoal. The quantity of other products is less than in lowland bogs, and I think that is mainly due to the physical condition of the top part of the bog. Whereas the top part at Killucan is very porous and elastic, and therefore the absorption of ammonia from the air is great, the top part at Dunmanway's bogs do not differ from the bottom part. But in the average the difference is very little.

The distillation of peat for by-products is not a new idea. It was proposed long ago, but, unfortunately, for the reasons already explained, the industry has not flourished. The production of mineral oil and paraffin from peat was established in 1889 in Brazil at Marabá. In the Journal of the Society of Chemical Industry there is an account of this establishment, which was producing not less than 80 tons per month of solid paraffin for candle-making. What is very important for the practical establishment of such industry is the treatment of peat before distillation.

The analysis shows that the peat as originally won contains from 85 per cent. to 93 per cent. of water, which when pressed out is found to contain in solution salt, and therefore it is very important to squeeze out as much as possible, as thereby the amount of ashes in the charcoal will be reduced. We have certainly in the market a great many different systems of compressing machines, and I am quite sure that the English engineers will soon find out the best means for compressing out this large amount of water at a cheap price. Apart from this, special attention must be paid to the drainage of the peat bogs themselves. If the peat bogs are not properly drained, as very often is the case, the amount of work necessary for cutting and drying is increased tremendously. At the same time you must always bear in mind that in draining off the water it is necessary to leave a sufficient amount to keep the lower parts in a sufficiently spongy state. This, however, is not a place to explain all the necessary conditions under which peat bogs must be worked, nor have I gained sufficient practical knowledge to warrant my advising upon the best methods of cutting and drying. My researches have been directed to the treatment of the industry from a chemical point of view, and I certainly think there is something to be done, and I am also sure that painstaking work will show fresh means of treatment, and larger openings for products obtained.

The CHAIRMAN, before inviting Dr. Dvorkovitz to read his paper, said they were very glad to see amongst them that night an eminent foreign chemist, one of their own members, in the person of Dr. Menéndez.

#### DISCUSSION.

Mr. BOVERTON REDWOOD said he did not know that he could say much that was novel in connection with this paper, but it appeared to him that the author had hardly given sufficient attention to the work which had already been done in connection with the utilisation of peat in Ireland. Though he was very loth to damp the ardour of Dr. Dvorkovitz, especially when he had such a laudable object in view as the amelioration of the condition of Ireland by the introduction of a new industry, yet it would be undesirable to raise up hopes which were never likely to be realised. It was well known that as long ago as 1849 Mr. Reece Rees patented a system of peat-distillation, and in 1851 the results obtained in the practical application of that system were very carefully investigated by Sir Robert Kane and Dr. Sullivan. The process consisted in distilling the peat in a kind of blast-furnace of brick encased in sheet-iron, somewhat similar to an iron blast-furnace, but with an arrangement at the top for taking away the products of distillation and conveying them through scrubbers and condensers. The furnace was about 10 ft. in diameter, and took a charge 100 tons. The charge having been lighted at the base, a current of air of about 3,000 cub. ft. per minute was introduced, and the distillation was effected by the heated gases produced. At the expiration of 24 hours the operation was completed, and Mr. Rees claimed that the ash underwent a species of fluxing, and came away as fluid slag. The products consisted of gas, an aqueous liquid, and tar. He professed to obtain from 100 tons 6,000,000 cub. ft. of gas containing hydrocarbons, a certain amount of free hydrogen, carbonic oxide, and some carbonic acid. The aqueous liquid contained ammonia, and from the tar lubricating oils and solid paraffin were obtained. Sir Robert Kane and Dr. Sullivan found great difficulty in carrying out the process described in the patent specification. According to their report, if the blast of air was sufficiently strong to give a combustible gas, there was practically no tar distillate, and, on the other hand, if they moderated the air-blast so as to get a fair yield of tar, they did not get the combustible gas. In fact, under the best conditions they found it exceedingly difficult to obtain a gas which could be used as fuel, and that was practically the basis of the process. Mr. Rees asserted that out of the 6,000,000 cub. ft. of gas which the 100 tons of peat produced, 2,000,000 would be sufficient to supply all the fuel necessary, and the remaining 4,000,000 would be available for use in lime-burning and various other industrial operations. But as a matter of fact, the outcome of this investigation was not confirmatory of the statements made, and was altogether of a very discouraging character. In 1861 Dr. Paul worked at the subject for Sir James Matheson, and obtained satisfactory commercial results, the balance sheet representing a year's working, showing a very fair margin of profit. It must, however, be borne in mind that at that period both the oil which was obtained and the solid paraffin fetched very much higher prices on the market than they would at present, and it seemed difficult to understand how it was possible, looking at these products as a source of income, to establish any profitable industry in connection with the distillation of peat. It might be that when they had the paper fully before them, and could understand a little more clearly the principles upon which the author was proceeding, they might be led to form a more favourable opinion of the prospects of the undertaking. It was possible that a modification in the method of working might give more favourable results than any hitherto obtained, but the difficulty would always remain that peat was of an exceedingly variable character. In the 27 samples of peat which were examined by Sir Robert Kane and Dr. Sullivan, there was an enormous variation in the physical and chemical characters. To give only one example, the highest specific gravity was 1.058, and the lowest 0.235, the density of the greater number being below 0.6. With

a material varying so greatly, it was very difficult to form a definite opinion as to the results to be practically obtained. Dr. Dvorkovitz had exhibited courage in taking up a subject which was certainly not of a promising character, and he should be only too glad to congratulate him if the outcome of his investigations was the establishment of a new industry tending to benefit Ireland.

Mr. WATSON SMITH said he had listened with great interest to this paper. Possibly Dr. Dvorkovitz would be interested to know that his friend the late Dr. R. Angus Smith read a most valuable treatise on this subject, which was unfortunately buried in the archives of the Literary and Philosophical Society of Manchester, and he would therefore give a summary of the results arrived at by Dr. Smith. He took a comparatively modest view of the possibilities of utilising peat. Speaking of the varieties of the substance, Dr. Angus Smith said that when anyone mentioned "peat" it was essential that he should define the kind of peat he referred to, so great are the differences. Mr. Boverton Redwood had shown this in speaking of the wide divergencies of specific gravity. Dr. Angus Smith states that a cubic foot of the more porous variety weighed 4 lb., and one of the lightest kinds from 48 to 80 lb. Certain peats contained a considerable quantity of bitumen soluble in benzene. In heating effect approximately  $2\frac{1}{2}$  parts of dry peat were equal to 1 of coal, whilst  $2\frac{1}{2}$  of dry woody fibre were equal to 2 of coal. Dr. Angus Smith stated that he could imagine—and on this point he laid great stress as regards benefit to the Irish peasant—farms of no great size growing fuel as fast as it could be used by a limited population, and even giving oil and ammonia by distillation and compression. It was pointed out that peat, or rather the growing peat, *i.e.*, peat-moss, always flourished best on a surface of its own kind, and therefore when a peat-bog was cut, the upper living surface ought to be carefully preserved, and immediately replaced on the cut surface, and in that way the surface plants (*sphagnum*) went on growing, and thus peat might be preserved and cultivated. With regard to the nitrogen and the quantity of ammonia to be derived by distillation, sometimes 2.4 per cent. of nitrogen, and occasionally 2.5 per cent., was found in dry peats. Certain peats, however, contained as little as only  $\frac{1}{2}$  per cent. Dr. Angus Smith thought it extremely likely that ozokerite was derived from oils which originally flowed from Austrian peats. Such oils had, indeed, been found under a large heath in Germany, the Luneberger Heide. On distilling the peat, oils and waxes were obtained, and several experiments showed a yield of as much as 6 per cent. The late Professor Schorlemmer had an opportunity of examining some of these oils, and found that they contained paraffin. It was curious that an oil from the heavy peat from Stornoway, when boiled with caustic soda, nearly all dissolved, and the solution on acidification yielded a resin closely resembling shellac in appearance. Dr. Angus Smith further suggested the utilisation of the living peat for storing water, because of the immense quantity absorbed and retained, as much as 75 per cent. of water being held in some peat-bogs over which animals and men could walk. It was certainly a novel and original idea to grow a water-reservoir, as Dr. Angus Smith had quaintly suggested, on the side of a hill, instead of digging one! In cutting a 10-ft. bog, we may allow by shrinkage one-fifth to remain dry. Such a bog, 10 ft. deep, will hold as much water as a reservoir of the same dimensions  $7\frac{1}{2}$  ft. deep. There was no doubt that before peat could be distilled in ovens like those proposed by Dr. Dvorkovitz, it would have to be compressed and dried, and probably be charged in in blocks. With ovens in construction so generally resembling the Otto-Hofmann coke-ovens, a very high temperature might be expected, and he presumed the gas used in firing would be the return-gas derived by the distillation of the peat. By distillation of a fuel containing so much woody fibres and matters akin thereto, under such conditions, it would be highly interesting to know what the characters of gas, tar, and aqueous residual would turn out to be. He at present, and in absence of distinct data, suspected there would not be anything like gas enough of a heating and inflammable kind, furnished, to keep such ovens going.

The CHAIRMAN said he did not know whether the peat-bags of Ireland, of which there seemed to be not only plenty, but a perennial supply, would come into immediate use while coal was to be had at anything like its present price, but the day might come when coal would be a costly article, and they might be obliged to resort to peat. In any case the collection of such information as that of Dr. Dvorkovitz would be of great value.

Dr. P. DVORKOVITZ said Mr. Redwood had expressed the belief that the treatment of peat could not be made practicable, and had supported it by the valuable report of Sir Robert Kane and Dr. Sullivan, but he thought he would admit himself that the admission of air into the retort was sufficient to account for the failure, as it prevented the production of any oil. It was no wonder, therefore, that the Rees process had failed. He was quite satisfied that if Sir Robert Kane and Dr. Sullivan had been more careful in taking their samples they would not have come to the conclusion they did as to the immense variety of peat. He had taken samples on the spot, which he had put in waxed paper and then in brown paper, and after bringing them home he found that 15 per cent. had already evaporated. He had analysed 17 samples of this peat, and nearly all, excluding only the top layer, gave the same results upon distillation. The samples should be weighed as soon as cut, and then you could tell whether they were different or not. Some samples when gradually dried in air would lose 75 per cent. of their weight, and of course all these points had to be allowed for in determining the specific gravity. He was not aware of Dr. Paul's experiment in 1861, though he had read a great deal on the subject. In fact there was too much written about peat, and at the same time too little. Generally peat had been treated very badly. The geologist did not want to know about it, or the botanist, and most of the books he had read had been written by reverend gentlemen. With regard to the practicability of using peat, he would remind them that it was found very practicable in Scotland to treat shales and obtain only 12 per cent. of oil and a certain proportion of ammonia. The quantity of ammonia in peat was tremendously large, and he thought it could be increased artificially. Peat absorbed ammonia very largely, as was shown by experiments made by Professor Fleischer and Dr. A. König. It had such great affinity for ammonia that if you put peat into carbonate of ammonia you would find after two days a large quantity of the ammonia was absorbed by the peat. Surely if it was worth while to distil shales for 12 per cent. of oil and a certain amount of ammonia, it was equally worth while to distil peat if you could get 6 per cent. of oil, 40 per cent. of charcoal, which was very valuable, and the ammonia would be not less than in the shale, apart from which you had acetic acid. He did not see why such prejudice existed against it, and why so many people believed that this business could not be established. When he went to Ireland he was told it was no use wasting his time about peat; lots of people had tried it already and failed. But if 99 had failed he wanted to be the 100th, who would succeed. If the members of the Society would pay more attention to the matter he was quite satisfied something could be done. People were quite ready to invest their money in Australia to find gold mines, but if they found them lying at their own feet they did not care about them; they are too near to them. It would be a great pleasure to him if he could see more attention paid to this matter, and the process of distillation established for the sake principally of the by-products, which he was sure would be practicable.

Mr. BOVERTON REDWOOD asked leave to add one word in justice to Sir Robert Kane and Dr. Sullivan in reference to the concluding remarks of Dr. Dvorkovitz. They did, as a matter of fact, make some comparative experiments also in the distillation of peat in closed vessels without an air-blast, and he believed he was right in saying that the results which they thus obtained were substantially the same as the best of those yielded by the application of a moderated air-blast.

Dr. DVORKOVITZ said he should have explained that he was aware of these results, and he did not wonder that they

were not more favourable. In distilling peat in closed vessels without squeezing out the water, you must use a large amount of heat to distil so much bulky material, and this large amount of heat used in closed vessels abstracted a large amount of the oily matters and a large amount of gas—much more really than that used.

The CHAIRMAN said, as this was the last occasion on which he should occupy the chair, he wished to express in a very few words his sense of the honour which was done him two years ago in electing him to the office of Chairman. From that time he had been very sensible of his own short comings, but he would be ungrateful if he did not thank those who had so ably assisted him—the Secretary, Mr. Heron, the members of the Committee, and the members of the Section at large.

## Manchester Section.

CHEMICAL THEATRE, OWENS COLLEGE.

Chairman: Ivan Levinstein.

Vice-Chairman: Edw. Schuonck.

### Committee:

G. H. Bailey.	P. Hart.
F. H. Bowman.	J. M. Irving.
R. F. Carpenter.	E. Knecht.
G. E. Davis.	W. H. Perkin, jun.
C. Dreyfus.	Sir H. E. Roscoe, M.P.
H. Grimshaw.	C. Truby.

### Hon. Local Secretary:

J. Carter Bell.

Bank House, The Cliff, Higher Broughton, Manchester.

The name in italics is that of a Member of Committee that retires at the end of the current session.

The following has been elected to fill the vacancy, and will take office in July next:—Committee: W. Thomson.

Meeting held Friday, 1st June 1894.

MR. IVAN LEVINSTEIN IN THE CHAIR.

### PRESENTATION TO MR. J. CARTER BELL.

THE Chairman said that before proceeding with the ordinary business of the meeting they had a most pleasant duty to perform. About ten years ago Sir Henry Roscoe had expressed an opinion that there was not material for the establishment of a section in Manchester, and that there was not sufficient interest taken in matters connected with the chemical industries in the neighbourhood at that time to ensure success. But owing to the great zeal exerted by their hon. secretary, Mr. Bell, during this long period of ten years the Section had grown to be one of the most important in connexion with the Society of Chemical Industry. Not only had the meetings been the means of imparting much valuable information to the members, but the social meetings, which had been organised by Mr. Bell in connexion with the Chemical Club, had also formed and cemented friendships which otherwise would never have existed. In acknowledgement of the very valuable services thus rendered by Mr. Bell at a great sacrifice of time and labour, and also in appreciation of his high professional



gratefully received with pleasure in presenting to him on behalf of the members of the Manchester Section of the Society of Chemical Industry, and of the Manchester Chemical Club the testimonial now before them, and in doing so he hoped Mr. Bell would enjoy the gift for many years to come.

The testimonial consists of an English brass quarter-line clock, on gongs and bells, with brass vases to match; also a pair of brass pedestal oil lamps. The clock bears the following inscription:—

"Presented to J. Carter Bell, Esq., A.R.S.M., F.I.C., by the members of the Manchester Section of the Society of Chemical Industry, and of the Manchester Chemical Club, June 1st, 1894."

Mr. BELL, in acknowledging the presentation, said it was not easy on such an occasion to express his feelings, but he assured them that he really felt they had done him a great honour and he was exceedingly grateful to them for the magnificent gift. Mr. Levinstein had been too modest in not taking credit to himself for being the first to suggest the formation of the Chemical Club, which had strengthened the Section, and so Mr. Davis must be given the credit of starting the Section, as well as the parent Society. Mr. Levinstein and Mr. Davis had planted, and he (Mr. Bell) had only watered. He could only add that the work he had done had been a labour of love. Of the 30 years he had been in Manchester the last ten had been by far the most enjoyable and profitable, and any services he had rendered had been amply repaid by the many friendships he had formed.

## COMPARATIVE RESULTS OF SOME MODERN SYSTEMS OF SEWAGE TREATMENT.

BY W. NAYLOR, F.C.S.

### DISCUSSION.

THE CHAIRMAN thought it would be desirable to avoid any reference to the way in which analyses of sewage were obtained, in view of the contemplated action of the Mersey and Irwell Committee against the Salford Corporation. Sir Henry Roscoe, M.P., chemical adviser to that committee, and Mr. Scudder, his assistant, had been invited to attend the meeting, but found themselves unable to do so. Sir Henry had written saying that it seemed to him that the materials incorporated in Mr. Naylor's paper were insufficient to base any definite proposals upon for the treatment of sewage, and both he and Mr. Scudder submitted that any conclusion to be satisfactory must be based on average, and not on single results. Mr. Scudder had sent the following communication on Mr. Naylor's remarks upon the electrical process.

MR. SCUDDER wrote as follows:—I very much doubt the propriety of entering into a discussion of a paper of this kind. It was said to be one on the comparative results of some modern systems of sewage treatment, but I venture to think that it is nothing of the kind. The table of results is far from satisfactory, and not at all suitable for drawing any recommendations therefrom. No one will, I hope, think of suggesting a line of action on a single experiment, and on the incomplete data given in the table of results. With reference to the scientific portion of the paper I should like to know why in considering two such abortive chemical equations as given by the author we must bear in mind that "ferrie hydrate is only sparingly soluble in water"? Mr. Naylor raises a question as to the cost of the electrical process based on the contention that additional electric energy will be required as the iron plates become thinner to compensate for the assumed increase of resistance caused by the increased space between the plates. Now, I have had the advantage of discussing this point with Mr. Octavius March, the electrical engineer who superintended the electrical details at Salford, and he contends that no additional

electrical energy will be required, but on the contrary, up to a certain point at any rate, there will be a reduction rather than an increase of total resistance. An electrolyte as a conductor is *not* comparable with a metallic conductor. If the resistance of a given length of a metallic conductor of uniform section be  $R$ , double the length will have a resistance of  $2R$ . But the portion of the electrolyte contained between two plates is *not* analogous. The passage of the electric current is *not confined* to that part of the electrolyte only; consequently if the specific resistance of electrolyte between two plates  $\frac{1}{2}$  in. apart be  $R$  the resistance when such plates are placed 1 in. apart will *not* be  $2R$ . By experiment it has been found that when the resistance between two plates  $\frac{1}{2}$  in. apart is 3, at 1 in. apart it is 4, not 6. The specific resistance of a given electrolyte is governed by three factors:—

- A. The area of electrodes immersed.
- B. The distance between the electrodes.
- C. The total bulk of electrolyte employed.

The first two are apparent. The third may be shown by taking a pair of electrodes fixed together at a given distance and immersing them in the electrolyte, in a vessel just large enough to take them. Note the current passing and the potential employed.

Then immerse them to the same depth in a larger vessel containing similar electrolyte, using the same potential, and it will be found that a larger current passes, thus showing a diminution of resistance.

The specific resistance of electrolyte is only a small factor in the calculation required to ascertain the E.M.F. necessary to obtain a given current through a given cell.

The arrangement of the Salford plant was such that the total resistance of each cell was 0.002 of an ohm—of this, about half would be represented by resistance of conductors and contacts, leaving 0.001 as the specific resistance of electrolyte. The mean counter E.M.F. was found to be 0.8 of a volt.

With the current to be employed a potential of 0.33 per cell would be required to overcome the specific resistance of the electrolyte. The same to overcome the resistance of conductors and contacts and 0.8 to balance the counter E.M.F. This gives us 1.46 volts as the total E.M.F. required per cell.

If the space between the plates were doubled, all other conditions being maintained, the resistance 0.001 would be increased to 0.0013, and the E.M.F. required to overcome it would then be 0.438. This + 0.33 + 0.8 gives 1.568, an increase of about 7 per cent.

With the arrangement proposed the space between the plates *could never be doubled*. The plates being  $\frac{1}{2}$  in. and the spaces  $\frac{1}{2}$  in., if the plates were reduced to their centre line, the space would only be increased from 5 to 9.

The dissolving action on the iron does not reduce it uniformly over the whole surface, but tends to pit it, converting a comparatively smooth surface into an increasingly rougher one and so materially *increasing* the surface of iron exposed. A square foot of iron plate  $\frac{1}{2}$  in. thick, would have a surface on one side of 144 square inches. If 96 holes 1 in. diameter were bored in this plate, the area of such holes would be 72 sq. in. and the remaining surface of iron also 72 sq. in., but the area of the sides of each hole would be 12 sq. in., so that the surface of iron exposed would be increased from 144 to 222 sq. in.

If the holes were bored  $\frac{1}{4}$  in. deep instead of through the plate, the exposed surface would be increased to about the same extent, viz., from 144 to 219, an increase of more than 50 per cent. This increase of surface would therefore more than compensate for the increased mean distance between the exposed surfaces. (For convenience in this calculation I take the area of the circle to be diam. 2.75 instead of 0.7854 and the circumference to be diam.  $3\frac{1}{2}$  instead of 3.1416).

With reference to the weight of iron that may be regarded as available, it is impossible to do more than estimate this. From a careful examination of the plates that were in constant use for some months at Salford, Mr. March came to the conclusion that  $\frac{2}{3}$ ths of the iron might be used, but as

a matter of fact in all the calculations, and estimates a loss of  $\frac{1}{2}$  was assumed in order to be on the safe side.

I have dwelt rather lengthily on this question because it is most important to point out the injustice that is caused by writers of papers not fully investigating the work of others and asking for their help, before they attempt to discuss questions about which they have no intimate knowledge, and levelling criticism of an order not calculated either to assist materially in the solution of the sewage problem or to aid those who have had sufficient energy and willingness to embark on the financial path of the problem.

Mr. BELL said he quite agreed with Sir Henry Roscoe and Mr. Seudder that the results obtained by Mr. Naylor were only isolated results, and must not be taken as conclusive. Mr. Naylor had referred to the amines process and, according to his analysis, the process gave no purification. They had treated 10,000,000 gallons per day at Salford on the Amines system and a very clear effluent was obtained. This, in his opinion, was principally due to the large excess of lime, from 20 to 30 cwt. being used to the million gallons. Of course the effluent would be very alkaline, but would keep better than salts of iron effluents. Unfortunately all lime effluents possessed a most peculiar odour, and they were said to decompose much sooner than others. To form an opinion of any process it should be tried week by week and month by month, as in the case of the Salford experiments. If he found a process which would give effluents containing 0.2 down to 0.1 of albuminoid ammonia in the 100,000 parts, he should call that a very good process, and above all he must emphatically say it was not so much the amount of albuminoid matter in the effluent, but its liability to secondary decomposition.

Mr. GRIMSHAW said, in common with Mr. Carter Bell and others, he had gone pretty extensively into the question of sewage purification, and he was bound to say that he agreed generally with his remarks on Mr. Naylor's analyses. At the same time, he did not suppose that Mr. Naylor intended to say that it was an exhaustive *resumé* upon sewage matters generally, but that it was a contribution intended to assist an accurate knowledge of the purification of sewage. He could supplement Mr. Naylor's figures as regards the iron treatment at Horwich, where Mr. Naylor gave a certain figure on a particular day. On referring to his book he had found an odd result which agreed with Mr. Naylor's figures. Speaking from memory, he believed Mr. Naylor obtained 0.4 in the effluent. Taking a long series of figures, it appeared that the process averaged out 0.2 to 0.28, and in these figures there was 0.4 for the highest and 0.2 for the lowest. With regard to what Mr. Bell had said in relation to lime treatment, they knew that if plenty of lime were put into sewage a good-looking effluent resulted, and in many cases that effluent would not decompose afterwards. On the other hand, it very often did and was sometimes worse than before treatment, hence the lime treatment, *per se*, stood universally condemned. In such a process as that of the Amines, involving the use of 40 cwt. of lime per million gallons, which, converted into carbonate, would give something like 11,000 tons of sludge per annum in the case of Salford, independently of the sewage residuum itself, he supposed that no river authority would allow such an alkaline effluent to be run into a water-course. They would have also to consider that the cost for lime would make it a very dear process. In regard to the experiments at Salford, the trials there had given a vast store of information which, although very ill digested, had been very useful to sewage chemists. Whilst Mr. Bell had put the question very neatly as to the experimenters who had been there, he did think that in one or two cases the Salford Committee had made mistakes, in so far that they expected the experimenter to become familiarised with all the conditions of a huge sewage works in a week or two, and expected them to produce in about a fortnight a result sufficiently satisfactory. That was to say, they asked these experimenters to do in three weeks what the corporation themselves had failed to do in six or eight years. In fact, the experiments had been suspended just when they were beginning to be successful. With regard to the question of after decomposition of sewage effluents and manufacturers'

effluents, it was not necessary for all to be skilled bacteriologists and to determine as to the exact germ to which the decomposition was due. For instance, the water of the Irwell, in consequence of the chemicals turned into it, was unable to putrefy during its flow, but anyone who knew anything about the Ship Canal would observe the fermentation which was taking place there. The point after all which had to be taken into consideration in the application of sewage treatment was whether the effluent would be one which would not be dangerous to health. He thought it would be more profitable if chemists would bear that fact in mind. They had been led into error by giving too exclusive attention to albuminoid ammonia. They wanted more facts in relation to other impurities. He believed much good might be done by sewage chemists comparing the results of their special tests, with a view to arrive at a conclusion as to which test or group of tests gives in the simplest and quickest way a means of deciding whether the effluents from sewage works and manufactories will afterwards become objectionable or will remain innocuous, and he hoped that the next contribution to the Society on this subject would be in this direction.

Mr. NAYLOR said in reply:—The chief ground of complaint appears to be that a greater number of results have not been presented. This is solely due to the fact that a man cannot give more than he possesses, and were it not for this unfortunate circumstance further results would certainly have been given.

At the same time, since the works on the subject, by Corfield, Slater, Crimp, Wandle, and others, and the reports of the Rivers Pollution Commissioners, the United States National Board of Health, the Massachusetts State Board of Health, and the experiments conducted by the Leeds, Bradford, London, and Salford Corporations, have one and all been unsuccessful in bringing about any approach to unanimity of opinion, I do not for a moment anticipate that any number of results compilable by a single individual would do any more good than will be done by what this paper simply purports to be, viz., a series of results representing fairly the work of some present day systems under normal conditions.

In Lancashire, at any rate, the question has been fought in the law courts during the past three years, principally upon the clause in the 1876 Act, which stipulates that an authority is not in default if he is using the best known practicable and available means. Now it appears to me that the best known practicable and available means are exactly those which result in leaving in the effluent the least amount of objectionable matter, compared with others.

By this I of course do not mean the amount of objectionable matter taken from the effluent but the amount left in. What this was to begin with, and therefore the percentage of purification matters nothing, so long as an average sewage is proved to be capable of treatment. If the sewage be unusually strong, then must the authority pay for its water carriage system with a small volume flush by double or even a triple treatment. At any rate, this much I contend, that every system must come in line with that which is shown by results to produce the best effluent under normal conditions.

I must confess that I cannot agree that the absence of secondary decomposition apart from the quantity of contained organic matter is any criterion of fitness to enter a river. The possibility of secondary decomposition depends entirely upon constantly changing conditions of chemical and bacteriological environment, and upon these grounds alone can it be discussed.

The action of lime, so far as my experience goes, is practically nil in reducing organic matters in solution. As a precipitant for suspended matter or as a clarifier, it has a good effect, but practically nothing further.

Alumina-ferric has been found to have a decolorising effect upon colouring matters if the liquor to be treated is rendered slightly acid, but no effect is noticeable with vegetable colouring matters, aniline blacks, and other aniline colours, particularly benzopurpurin.

Dealing more particularly with Mr. Seudder's criticism, I have yet to learn that financial losses incurred in the practical working of any process render such processes free from criticism, or that any *injustice* is caused by criticisms levelled by persons having no intimate knowledge of the subject matter.

In such a case criticism is likely to be more absurd than unjust: notwithstanding I claim both the financial loss and intimate knowledge with respect to a similar process, patent rights for which I took out four years ago, but which were allowed to lapse, as the working of the process was not a success from the very causes put forth in this paper.

M. Seudder's criticism is worthy of considerable merit and attention as a very ingenious contention of facts. He begins by saying that "an electrolyte as a conductor is not comparable with a metallic conductor," and, as he makes no allusion to the reference I gave which shows that it is comparable, I must take it that he claims a difference due to his statement that the passage of the current is not confined to that part of the electrolyte only between the plates.

Now, one may perhaps follow his comparison of similar electrodes at similar distances apart in differently sized vessels, and may perhaps even admit that the current may pass through some portion of the electrolyte other than between the plates in the large vessel, but in the case of Salford the electric shoot was completely filled with electrodes, and it was really absolutely impossible for the current to pass anywhere else than between the plates.

The channel was 90 ft. long, and full from end to end with electrodes, which at any point were not more than half an inch apart; and since they were not submerged with the sewage, the condition C instanced by Mr. Seudder, viz., "the total bulk of electrolyte employed," has no bearing on the matter.

The condition A, namely, "the area of electrodes immersed," is constant, which therefore leaves only the condition B, viz., the distance between the electrodes, which was the one upon which I based my remarks. It is then stated that "the specific resistance of electrolyte is only a small factor in the calculation required to ascertain the E.M.F. necessary to obtain a given current through a given cell."

In my paper I do not refer in any way to the E.M.F. necessary "to obtain a given current through a given cell," but only to the quantity of current "passed between the electrodes through the intervening sewage."

The resistance offered by the conductors, contacts, and electrodes themselves has nothing to do with the actual amount of current required for the electrolysis of the sewage.

The calculations therefore based upon the resistance of contacts, electrodes, and electrolyte are outside the question, and I still claim that the initial resistance offered by the electrolyte itself in the Salford shoot to the final, if the plates distances were doubled, would be as 5 to 9 as near as working conditions would allow.

The question of pitting does not affect the matter one way or the other. If any advantage were derived from pitting, why not bore through the plates to begin with? Even assuming that uniformly bored plates give an increased surface exposure, iron plates when used as electrodes do not pit to order, uniformly, but generally just in one or two places, and just as easy as it is to suggest 144 holes from pitting in a 1-ft. square plate, so is it to suggest one 9-in. hole in a plate 10 ins. in diameter, in which case nearly all the electrode would be gone.

## Newcastle Section.

Chairman: A. Allhusen.

Vice-Chairman: John Pattinson.

Committee:

P. P. Badson.

G. T. France.

T. W. Hogg.

T. W. Lovibond.

W. Martyn.

John Morrison.

B. S. Proctor.

W. W. Proctor.

W. L. Rennoldson.

W. A. Rowell.

John Watson.

Hon. Local Secretary and Treasurer:

Dr. J. T. Dunn, The School, Gateshead.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

The names in italics are those of Members of Committee who retired at the end of the current session.

The following have been elected to fill the vacancies, and will take office in July next:—Chairman: G. T. France; Vice-Chairman: A. Allhusen; Committee: John Pattinson and T. W. Stuart.

### SESSION 1894.

Meeting held in the Durham College of Science,  
Newcastle, on Thursday, May 24th, 1894.

MR. A. ALLHUSEN IN THE CHAIR.

### NOTE ON THE POISONOUS ACTION OF LEAD SALTS.

BY PROF. P. PHILLIPS BEDSON.

In a monograph on Lead Poisoning published in 1891, Dr. Oliver describes a series of experiments which were conducted in my laboratory by Mr. R. D. Best, with the object of studying the action of gastric juice upon white lead, as it would appear that the digestive canal is the channel by which lead finds its way into the system.

The experiments were made by taking a given weight of white lead, placing it in a parchment tube, and adding to it a measured volume of gastric juice or other liquid,\* the action of which it was desired to study. The parchment tube with its contents were suspended in a beaker containing a measured volume of pure distilled water, and this again was immersed in a larger vessel containing water, which was heated for several hours at a temperature of 20°–30° C. At the end of this time the contents of the inner beaker were examined for lead, and the amount of lead, existing in a soluble form, which had diffused through the parchment tube, was determined. The results of these experiments, tabulated below, show gastric juice, both natural and artificial, have an appreciable solvent action on the lead carbonate; and further, that the active constituent is the hydrochloric acid. It will also be seen that when gastric juice is employed in the digestion of proteid matter there is a smaller portion of white lead attacked.

White Lead taken.	Lead Dissolved.	Nature of Liquid.
Grms.	Mgrms.	
1	0.005	Per 10 cc. of gastric juice (natural) diluted with twice its volume of water.
1	0.002	" " "
1	0.002	" " "
1	0.001	Per 10 cc. of artificial gastric juice.
1	8.75	Per 10 cc. 0.3 per cent. solution of HCl and 1 grm. pepsin.
1	6.88	Per 10 cc. 0.3 per cent. solution of HCl and 0.5 grm. pepsin.
1	6.93	Per 10 cc. 0.3 per cent. solution of HCl and 0.25 grm. pepsin.
1	7.4	Per 10 cc. 0.3 per cent. solution of HCl.
1	None	Per 10 cc. of solution containing 1 grm. of pepsin.

\* 25 cc. of liquid was used in all cases.

It is usually stated that lead sulphate is non-poisonous, and possibly upon this belief is based the method of giving white lead workers water containing free sulphuric acid to mitigate the ill effects of white lead. In fact, Dr. Farr, in his report on a process for the manufacture of a new white lead, states that: "If a physician were called upon to attend a case of lead poisoning by carbonate, he would administer sulphuric acid in order to convert it into a sulphate or insoluble form of lead. It is obvious that the white sulphate of lead, therefore, carries with it, so to speak, the antidote to the poison."

Such statements as the above appeared to me to suggest a repetition of experiments, similar to those made with the ordinary white lead, using both precipitated sulphate of lead and the new white lead. At my suggestion, Mr. Elsdon, one of the students in this college, made such a series of experiments, the results of which are given below. The outcome of these experiments is to demonstrate that both these materials are dissolved by water to some extent, and more readily by water containing hydrochloric acid in amounts similar to those found in gastric juice. Further, the new white lead appears more susceptible than the precipitated lead sulphate. Undoubtedly, the proportion of the "new white lead" dissolved by the diluted hydrochloric acid is smaller than is the case with the ordinary white lead, the proportion of solubility being about 1:5. Still, such results necessitate a modification of our views as to the non-poisonous action of lead sulphate, and a more guarded attitude towards such traditional empiricism as that which teaches us that lead sulphate is non-poisonous because of its insolubility. Solubility is a matter of degree, and every student of chemistry knows he can decompose lead sulphate by boiling it with hydrochloric acid; in the light of which fact the results of these experiments are not remarkable, since they show that the change which takes place at a high temperature with such ease and completeness, goes on more slowly and gradually at a lower temperature and with very diluted hydrochloric acid.

New White Lead. Weight Taken.	Weight of Lead Dissolved.	Nature of Solvent.
Grm.		
1	0.048	Per 10 cc. of water.
1	0.084	" "
1	1.5	Per 10 cc. of 0.2 per cent. solution of HCl.
1	1.5	" " "
1	1.748	Per 10 cc. of 0.2 per cent. solution of HCl + 1 grm. pepsin.
1	2.0	Per 10 cc. of 0.2 per cent. solution of HCl + 1 grm. pepsin.
<hr/>		
Lead Sulphate.	Grm.	
1	0.0576	Per 10 cc. of water.
1	0.0376	" "
1	0.75	Per 10 cc. of 0.2 per cent. solution of HCl.
1	0.75	" " "
1	1.00	Per 10 cc. of 0.2 per cent. solution of HCl + 1 grm. pepsin.
1	0.89	" " "

## NITRATE OF SODA IN EGYPT.

BY W. COSSAR MACKENZIE, F.R.S.E., F.C.S.

THAT the natives of Upper Egypt used a natural manure, got from the hills on both sides of the river, has been known for some time to those who have to go there and who take an interest in the doings of the people, but what was the exact nature of the fertilising ingredient does not seem to have been known. Beyond a casual knowledge of the fact that this material was used as a manure, no further interest seems to have been taken in the subject.

In one of his journeys there, Mr. R. A. Floyer obtained several samples of this species of "tail"—"tail" is the name applied to clays used for pottery as well as these manurial clays—and handed them over to me to examine. The samples were pieces selected at random from heaps of the material on land or in boats, and cannot be taken as representative of the whole. As may be seen from the subjoined table, the valuable ingredient is nitrate of soda, and this is present in proportions varying from 2.5 to 18.5 per cent.

Thinking the matter of more than ordinary importance, I determined to take the first opportunity that presented itself of going there to see the deposit and take samples personally, especially as the exact spot from where Mr. Floyer's samples were got was not known. A short vacation at the end of Ramadan—the Mohammedan month for fasting—afforded a suitable opportunity, of which I availed myself. The Nile Valley, above the delta, is generally from 4 to 9 miles wide and is flanked on each side, as far south as Edfon, by ranges of hills of rammed limestone. South of Edfon the rock becomes sandy in character. Between Kench and Esneh, a distance of about 75 miles, these nitrate deposits are found in detached places near the foot of the limestone hills. Luxor, as being about the centre of this stretch and most suitable in every way for the purpose in view, was visited, and an excursion made to the "Gebel el Tail"—clay hill. As guide, a man was got who goes there for the manure, and a start was made about 7 a.m. The journey took, by camel, about three hours, and the distance was probably about 12 miles across the desert. About 4 miles before reaching the "Gebel el Tail" we passed the "Gebel el Gheir"—lime-hill—a very white limestone, with 10 per cent. of sand, but otherwise very pure, and from there, on, the valley was strewn with symmetrical flint nodules. A short distance before reaching the main deposit, a bank of dark coloured material was passed, which the guide stated to be poor tail, and on analysis it was found to contain only 0.28 per cent. of sodium nitrate. The main deposit lay on the sloping face of what appeared to be the highest hill of the district, but to which the guide could give no name but "Gebel"—hill—or "Gebel el tail," and appeared to have a height of 10 or 12 metres; how deep the deposit was, I am unable to say. This deposit did not seem extensive, and lay approximately in the centre of a bay or segment of a circle formed by the limestone rocks, but the guide said there was more "tail" to the south and to the north. The rocks rose perpendicularly for some 10 metres above the "tail" and contained many flint nodules, in some places so numerous as to give the rock the appearance of conglomerate. The strata in Egypt dip gradually from south to north, and this precludes the idea of this being a stratum of nitrate bearing clay cropping out at intervals from north to south. That the nitrate came from the remains of old villages and was absorbed by the clay does not seem possible, looking to the appearance and situation of the deposit. It seems more probable that caves or hollows in the limestone cliffs acted as receptacles for clay and organic matter from the water. After the retirement of the water, nitrification of the organic matter set in, aided by the warmth of the climate and the presence of calcium carbonate, so that the nitrate was formed where it is now found.

## COMPOSITION OF NITRATE-BEARING CLAYS.

*Received from Mr. Floyer.*

	1.	2.	3.	4.	5.	6.
Silica, $\text{SiO}_2$ .....	25.41	25.18	15.83	31.12	37.05	27.68
Silicic acid.....	14.34	27.53	16.37	22.53	21.67	22.81
Carbon dioxide.....	11.77	9.20	8.13	7.92	6.37	5.92
Phosphoric acid, $\text{H}_3\text{PO}_4$ .....	0.57	0.28	1.72	0.29	0.13	0.27
Sulphuric acid, $\text{H}_2\text{SO}_4$ .....	3.82	12.65	9.87	7.13	12.30	6.87
Chlorine.....	4.90	9.20	12.92	6.31	1.18	6.31
*Nitric acid, $\text{N}_2\text{O}_5$ .....	9.40	1.63	9.94	8.82	3.89	11.77
Alumina and iron oxide.....	15.40	2.090	11.25	18.79	18.65	18.26
Lime.....	18.90	9.90	13.31	13.33	11.84	11.51
Magnesia.....	0.67	1.68	1.13	1.12	2.56	0.96
Potash.....	6.40	0.15	3.45	6.18	0.50	0.21
Soda, as hydrochloride.....	19.71	7.00	15.77	12.55	12.49	13.84
Less oxygen equivalent to Cl.....	1.05	2.90	2.69	1.35	0.84	1.35
*Equivalent to sodium nitrate.....	14.89	2.53	15.65	13.89	6.13	18.53
Total soluble matter.....	27.26	36.14	50.58	32.50	29.60	37.04

Samples were taken from the deposit: the loose upper surface was first removed and pieces of the clay were taken from different places and afterwards analysed with the following results:—

## COMPOSITION OF NITRATE CLAYS.

*From Gebel el Taql, Luxor.*

	1.	2.	3.	4.	5.	6.	7.	8.	9.
Silica.....	13.98	14.32	11.14	22.12	11.12	27.11	16.42	23.28	48.88
Carbon dioxide.....	20.80	22.16	32.91	23.27	29.18	19.08	28.84	13.15	6.49
Sulphuric acid, $\text{H}_2\text{SO}_4$ .....	1.50	2.59	6.90	1.52	1.95	1.74	0.59	1.70	1.18
Chlorine.....	1.43	2.74	0.81	6.29	1.82	1.07	1.35	7.33	0.38
*Nitric acid, $\text{N}_2\text{O}_5$ .....	1.98	5.45	1.35	1.48	3.78	1.89	0.77	5.90	0.18
Iron oxide and alumina.....	7.96	8.86	7.12	11.46	6.16	13.91	7.32	12.66	30.56
Lime.....	36.18	34.94	19.27	32.16	36.82	27.23	38.68	19.00	2.66
Magnesia.....	3.15	3.56	3.05	4.22	3.25	4.12	3.67	3.83	2.29
*Equivalent to sodium nitrate.....	3.12	8.59	2.13	2.34	5.63	2.97	1.21	9.29	0.28
Total soluble matter.....	9.71	18.69	6.17	7.21	12.85	8.11	6.04	25.42	4.11

The first eight samples in the above table were all taken from the main deposit, number nine was from the bank of dark-coloured material, said to be poor tail, already referred to. The first eight samples were taken from different places at different heights, but all from the same deposit; number one was from the bottom, and number eight from the top, while the intermediate numbers were taken from places between one and eight.

As is at once seen, the percentages of nitrate in these samples from the same place vary very much; and this is readily understood. Wherever the rock was cracked crystals of salt were to be seen, indeed all exposed surfaces gave visible indications of salt, but where freshly broken no crystals and no appearance of salt was found though the clay had a distinctly saline taste. One large block had a crystalline coating with crystals half an inch long. On analysis this efflorescence was found to consist

of 65 per cent. of nitrate and 35 per cent. of chloride of sodium. Taking these facts into consideration it is evident that great care must be used, in taking samples, to have the surfaces removed and the rock cut into so that specimens may be obtained which have been affected as little as possible in this way. Since this discovery of nitrate was reported Mr. Floyer and Professor Sickenberger, of the Cairo Medical School, have been sent to Upper Egypt to enquire into the whole subject, and endeavour to form an opinion as to the quantity of material available. The annual consumption of this material in Upper Egypt has been estimated at 500,000 tons, but this is, in my opinion, too high an estimate: half of that quantity will more nearly represent it.

Whether Egyptian nitrate of soda will ever appear as a commercial product in European markets cannot be decided until the results of Floyer and Sickenberger's investigations

are known, but unless richer varieties are found than that got at Luxor the extraction and purification of nitrate for commerce is not very probable.

Means of conveying the tail from the hills to the river are wanting. Egypt is a narrow strip of arable land along the banks of the river, and so the river is the principal "road" of the country, and others are almost unnecessary. Out into the desert there are of course no roads. The railway does not yet extend up there, and when it does it will not pass near the tail deposits, but through the cultivated land. Nothing is left but the means of conveyance at present in use. The construction of a railway or tramway is hardly feasible, seeing that the tail is got in detached deposits, sometimes at considerable distances from each other. At present the tail is transported by camels and donkeys. One journey per diem is all that can be expected from each animal, and taking the load of a camel at 500 lbs., and his cost per diem at 1s., the cost of bringing 10 tons of clay to the river bank amounts to 2*l.* 5s. If the clay contains 10 per cent. of nitrate, 2*l.* 5s. will be the cost of conveying 1 ton of nitrate to the river, for of course the extraction must be done at the river, there being no water in the desert, and is a very large item to be added to the cost of extraction. There seems room, however, for profit, even at that percentage. Should, however, the average percentage be only five, the cost for carriage per ton of nitrate would then be 4*l.* 10s., and would probably render the extraction of nitrate profitably, hopeless.

### AN ABNORMAL MILK.

By R. R. GREIG SMITH, B.Sc., F.C.S.

IN the spring of last year, attention was drawn to the peculiar property of the milk yielded by a cow in the possession of Mr. Scott, Belford, Northumberland. With it a churn was unnecessary, for, by simply stirring the soured cream with a stick, butter was produced. At the invitation of Mr. Scott, the writer called upon him, and was shown the cow, which was what is known as an Irish-bred beast, and about 10 years old. It had calved prematurely on the 16th April 1893, and consequently was not in its usual condition, neither was there a plentiful supply of fodder, for the spring had been very dry and the meadows were parched, yet, from the 1st to the 31st of May it had yielded 1,257 lbs. of milk, and from the 1st to the 16th of June, 567 lbs., in all 1,824 lbs. during 47 days, or a daily average of 39 lbs., equal to nearly four gallons. The soured cream was semi-solid, and led one to expect that butter made from it would contain a large amount of casein. Mrs. Scott's method of butter making consisted in vigorously stirring the soured cream, contained in an earthenware dairy jar, with a clean wooden stick for about a minute and a half, and, when the butter had broken, adding a little cold water and again stirring for half a minute, by which time the butter was ready for washing and working. When made up it had a very pleasant taste, and was similar in every respect to butter made with a churn in the ordinary way. Samples of the milk, cream, and butter were sent to the laboratory of the Durham College of Science and examined.

The cream, from which butter was repeatedly made in the laboratory, using a spatula and a porcelain basin, contained 57.40 per cent. of fat, an amount considerably above the average of that contained in ordinary cream, whether obtained by the slow methods or by the separator, but similar to that found in clotted cream. The cream rose quickly and formed a very compact yellow layer with its lower surface so well defined, that one would almost have

thought it had formed a cake. The analysis of the milk gave the following numbers:

Fat.....	7.50	..
Solids not fat.....	8.70	..
Sugar by difference.....	..	1.70
Protein N x 6.38.....	..	3.50
Ash.....	..	0.67
Specific gravity.....	1.027	..

The only striking figures in the analysis are those giving the percentage of fat, which is considerably above the average, though not uncommon with the milk of individual cows. The relations between the constituents are slightly different from what we find in mixed milks, inasmuch as the fat calculated by Deep Richmond's formula (Analyst, XIV., 121) is 7.76, a difference of 0.26 per cent., and the ratio between the percentages of ash, proteins, and sugar, which Vieth gives as being in the very constant proportion of 2.9:13.5 to this case as 2:10.8, 12.5.

The ash had an alkalinity equal to 2.05 per cent.  $\text{Na}_2\text{O}$ , and the following figures show its composition:—

Iron and calcium oxides.....	0.84
Lime.....	25.54
Magn. salt.....	2.96
Potash.....	15.08
Salts.....	8.12
Phosphoric anhydride.....	33.16
Sulphuric anhydride.....	3.24
Chlorine.....	13.20
Loss.....	0.77
Oxygen = chlorine.....	10.00
	100.00

The published analyses of milk ash agree in having the constituents very similar to those found, with the exception of potash, lime, and phosphoric anhydride; Kirschner in his *Milchwirtschaft* quotes two analyses which have—

Potash.....	25.42	23.54
Lime.....	21.45	22.57
Phosphoric anhydride.....	21.11	27.68

These would indicate that, in this abnormal milk, there may be a deficiency in one or all of the potassium compounds, viz.:—monopotassic phosphate, dipotassic phosphate, potassic citrate, and an excess of one of the lime salts, probably dialcic phosphate, which might be there to the exclusion of much of the trialcic form.

The butter, made in this curious way, was quite normal, and though higher than the average English butters, did not show the amount of casein that one would have expected from the process of manufacture.

Analysis of the butter:—

Fat.....	83.32
Water.....	15.28
Curd, &c.....	1.30
Salt.....	0.10
	100.00

The volatile acids determined by the Reichert-Meisler-Wollny method had a number 28.4.

A milk somewhat similar was produced by a milking shorthorn cow, the property of Mr. Hedley, Overthwarts, Edlingham, who kindly supplied samples of milk, cream, and butter. The cream was made into butter, in the laboratory, as easily as in the Belford sample; the milk had the following composition:—

Fat.....	4.35	..
Solids not fat.....	9.36	..
Sugar by difference.....	..	5.13
Protein N x 6.38.....	..	3.51
Ash.....	..	0.72
Specific gravity.....	1.0324	..

This milk has an analysis entirely different from the Belford one, though they resemble each other in having the fat lower than the calculated amounts for mixed milks, the fat calculated being 1.66, showing a difference of 0.31 per cent. The Vieth ratio is as 2:9.75:14.25.

In examining the Belford milk microscopically, a few large globules were observed which varied up to 0.001 inch in diameter, but the majority were fairly uniform, and close to an average of 0.0004 inch. According to Kirchner, variations are found up to 0.00039 inch—practically the average diameter in the abnormal milk—the mean being 0.00016 inch. McConnell in his *Agricultural Note Book* gives 0.00019 inch as the average of the Jersey and 0.00014 inch as that of the Ayrshire cow. It will be seen then, that the fat globules in this abnormal milk are very large, practically double the diameter, that is eight times the size, of those in the milk of our Jersey breed, which is admitted to have the largest. The mean diameter of the globules in the Edlingham milk was less than the Belford sample, having an average of 0.0003 inch. The surface tension of the suspended fat globules, in both milks, was very low, pressure on the cover glass being sufficient to make them lose their apparently circular shape and run together with great readiness.

In all probability, the size of the fat globules explains the abnormality, which appears to be much more common than is generally supposed, for there are at present in Belford other three cows, unrelated, so far as is known, to that of Mr. Scott, which possess this property. When the abnormal case was made known in Edlingham, the milks of several individual cows were, when tried, found to be similar. Another case occurs at Riding Mill, and it is likely of frequent occurrence in Britain, though apparently unheard of till lately.

only recently seemed to prove positively the presence or the absence of adulterants, and thus were excellent means of establishing the purity of lard, are at the present time of no value whatever.

We know that, besides some adulterants which have never attained any importance from a commercial point of view, cotton-seed oil, cotton-oil, stearine, lard-stearine, oleo-stearine, are chiefly used in compounding lards. All these substances are to-day differently treated and more carefully refined; and, being therefore in a greater state of purity, they do not give reactions formerly thought to be very characteristic.

We may add that Dr. A. Goske (*Chem. Zeit.* 1892, 1560) referred to lard compounded with oleo-stearine, lard-oil, and lard. The mixtures which he thinks might pass as pure lard, showing the correct iodine number and being free from vegetable oils, are the following:—

	I.	II.
	Per Cent.	Per Cent.
Oleo-stearine .....	25	35
Lard .....	15	25
Lard oil .....	20	40

The first mixture would show, according to Goske, 59.75 for iodine number, and the second one 57.27. On account of the high price of lard-oil we thought it more than improbable that such mixtures would be produced commercially. To confirm our opinion we requested some of the leading firms to give us quotations for oleo-stearine, lard, and lard-oil. According to the average prices on February 20th, 1893, the raw materials of 1 lb. of mixture No. I. and II. would cost—

I. 13.1375 cents.

II. 13.125 „

whereas the price for pure lard at the same day was—

13.125 cents.

For practical reasons, therefore, such mixtures will not be found in the market, and the chemist called upon to determine the purity of lard may safely neglect the search for adulterants of this kind. Moreover, the microscopical examination and the crystallisation-point would establish the presence of oleo-stearine.

Again Ad. Jolles and Ed. Wild (*Chem. Zeit.* 1893, 879) detected sunflower-oil in a sample of oleo-margarine. As they found out that cotton-seed oil is about twice as expensive as sunflower-oil, they think that fits for eating purposes could be advantageously adulterated with the latter oil. Sunflower-oil is a clear liquid with slightly yellowish colour, agreeable smell, and sweet taste. If the difference in price is actually as great as Jolles and Wild state, we do not doubt that adulterations of lard with sunflower-oil instead of cotton-seed oil will be observed by the analyst. We have hitherto been unable to procure a sample of sunflower-oil, in the New York market, and could not get any quotations. We intend, however, to buy a larger quantity of the oil, and propose to compound lard with the same. The various mixtures will be subjected to all the tests for lard, &c., and we hope to be able to publish our results at an early date.

On reviewing the different known properties and reactions of lard and its adulterants, we see, as already stated by many authorities, that the specific gravity, the melting point, the saponification equivalent, the determination of volatile, free, and fixed acids, and the determination of the refractive index, are quite useless for the determination of the purity of lard, as mixtures can be prepared which would give approximately the same figures for all these constants as pure lard itself.

Even for a final confirmation of the result, obtained by more decisive reactions, the above-named tests—very rare cases, where more scientific research is intended, excepted—are of no concern for commercial analytical purposes, and, as far as our experience goes, the analyst may safely dispense with them altogether.

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The names in italics are those of Members of Committee who retire at the end of the current session.

The following have been elected to fill the vacancies, and will take office in July next:—Committee: J. F. Kempson and T. Mason.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

Meeting held at University College, Nottingham,  
on Wednesday, May 23rd.

MR. J. M. C. PATON IN THE CHAIR.

### ON COMMERCIAL ANALYSIS OF LARD.

BY H. SCHWEITZER AND E. LINGWITZ.

WITHIN the last few years the art of refining animal and vegetable oils and fats—and in connection with it, the art of compounding lard, i.e., adulterating lard—has made great progress. Some physical and chemical reactions which

Other reactions—as those with nitrate of silver, after Beechi, after Beechi-Holmer; with chloride of gold, after Labiche and Hirschsohn, which were pronounced to be of the utmost importance in the analysis of lard—have since lost their decisive value.

Meike and Wimmer (Zeits. f. ang. Chem. 1891, 518) found different samples of cotton-seed oil and adulterated lard which behaved like pure lard when subjected to those tests, and thus confirmed the observation previously made by E. Dieterich (Chem. Zeit. Rep. 1891, 117).

Warren's chloride of sulphur test (Chem. News, 1888, 113), which Wiley (Bull. U.S. Dep. of Agric., Divis. of Chem.) thought might prove of great value qualitatively and quantitatively, is, according to recent investigations, useless for analytical purposes, as Warren's observations are not quite correct (Chem. Zeit. 1893, 635—636; Muspratt, 4th edition by Stolmann and Kerl, Vol. III, p. 316).

Of all the better-known tests we have thus left the iodine number, the colour reaction with nitric and sulphuric acids, the test with nitrate of silver after Beechi-Milliau, the rise of temperature with sulphuric acid, the microscopical examination, and the crystallisation point.

Of more modern date is the reaction with phospho-molybdic acid and with saturated solution of picric acid in ether (Pharm. Zeit. 1891, 798; Zeits. f. ang. Chem. 1892, 99).

Recently, F. Ganter (Zeits. f. anal. Chem. 32, 305) described a new method for the colour-test with sulphuric acid, and in the Analyst (Analyst, 1893, 23, 221; Chem. Zeit. Rep. 1893, 251) a new way of reacting with nitrate of silver has been recommended.

All these tests have been again and again examined by us, and we have finally arrived at the conclusion that for the determination of the purity of lard the following tests, when taken in a group, are reliable and decisive:—

- (1.) Test with phospho-molybdic acid.
- (2.) Milliau's nitrate of silver test.
- (3.) Iodine number.
- (4.) Rise of temperature with sulphuric acid.
- (5.) Crystallisation point.
- (6.) Microscopical appearance.

**Test with Phospho-molybdic Acid and with Nitrate of Silver, after Milliau.**—Both reactions are used for the detection of cotton-seed oil and cotton-oil stearine. The first one is carried out as follows:—25 drops of the melted lard are dissolved in 5 cc. of chloroform in a test-tube, then 2 cc. of phospho-molybdic acid solution (10 per cent. Merck) are added. The test-tube is thoroughly shaken, and after letting stand for a short time the liquid settles in two layers. The lower layer is colourless and the upper layer assumes a bright green colour when cotton-oil or any other vegetable oil is present. On addition of ammonia to the acid solution the green colour turns blue.

If the lard is absolutely pure the upper layer remains yellow, the original colour of the reagent (phospho-molybdic acid) not being changed. The addition of ammonia makes the liquid colourless.

All the vegetable oils which we have had occasion to subject to this test gave a decided reaction. A sample of cotton-oil, refined by ourselves by means of chemicals *without heating*, which did not give Beechi and Labiche and Hirschsohn's test showed the above reaction perfectly.

According to Welmans, the discoverer of the test, mineral oils do not show the reaction, and among the fats of animal origin only cod-liver oil acts like the vegetable oils. With some practice it is easy to detect the presence of about 10 per cent. of cotton-seed oil in a compounded lard, but an addition of less cotton-seed oil can hardly be traced by this reaction. On such cases there is only one reaction, which, as far as our experience goes, never fails, and this is the Milliau test with nitrate of silver, as described at length in the Journal of the Am. Chem. Soc. (Journ. Am. Chem. Soc. XV., 164; J. Soc. Chem. Ind. 716, 1893). By strictly adhering to the explicit directions of Milliau, the presence of cotton-seed oil down to 1 per cent. is always shown by the mirror-like precipitate of metallic silver, by which the fatty acids of the mixture are blackened.

No other test can be so confidently relied upon and give a such decided a reaction. In doubtful cases the chemist should at all events resort to its execution.

**Iodine Number.**—The iodine number is valuable for the detection of both cotton-seed oil and oleo-stearine. If determined alone it has no meaning whatever, on account of the fact that mixtures can be produced showing the same iodine number as pure lard, and also because the iodine number of adulterated pure samples of lard varies within very wide limits. Wiley (Bulletin, 13, 18) found pure lard with iodine numbers from 57.34—85.03, Anthor and Zinck (Zeits. anal. Chem. 31, p. 534) from 49.2—52.90, and Nentfeld (Archiv. f. Hygiene, 17, p. 152) from 46—61, the latter fixing the maximum figure for the iodine number of pure lard at 61, which is decidedly wrong. But for a final confirmation of the results obtained by the above-named tests, the iodine number is a very satisfactory determination. Although different chemists have of late proposed changes of the original Hubl method for the determination of the amount of iodine absorbed by the fatty acids of the oleine series, we have, after many experiments, decided not to adopt any of those changes. We still carry out the test as described in Wiley's publication and in R. Benedict's book, "Die Analyse der Fette und Wacharten."

We weigh 0.3 gr. of the lard by means of our weighing pipette (Journ. of the Am. Chem. Soc. 15, 190) in a glass-stoppered reagent bottle of about 500 cc. capacity, dissolve it in 10 cc. of chloroform, and add 25 cc. of iodine solution. We manipulate as described in the above publications. Hubl stated (Dingl. Polyt. J. 253, 281) that the reaction is completed after 1½—2 hours, but that it is immaterial whether the final titration is made after 5 or 48 hours.

Frequently we were requested to carry out the examination of fats and oils in very short time, and we were thus forced to find a method permitting the determination of the iodine number in less than five hours. Although Benedikt states that iodine, which acts very slowly on fat at ordinary temperature, acts very irregularly at higher temperature and shows more complicated reactions, we tried to apply heat to the liquid in which the absorption of iodine took place.

We carried out very many experiments, changing the time and the temperature of the reaction. In the following lines we give only those figures from our records which seem to us to be necessary to justify the changes which we adopted for this determination.

Cotton-seed oil, oleo-stearine, absolutely pure lard, and compounded lard were subjected to examination. With every one of these different experiments a blank test with 10 cc. of chloroform and 25 cc. of iodine solution was titrated after having been exposed for the same length of time to the same temperature. The titrations of these blank tests gave always the figures obtained in standardising the original iodine solution directly. From this it appears that, under the conditions described below, heat does not influence the action between chloroform and iodine solution. The following results were obtained:—

Fat.	After 14 Hours at ordinary Temp.	50° C.			
		5 Min.	10 Min.	15 Min.	20 Min.
Cotton-seed oil.....	108.4	107.2	107.6	107.7	108.8
Oleo-stearine.....	28.0	27.8	26.4	27.0	28.4
Pure lard.....	67.3	67.2	67.1	66.5	67.7
Compounded lard (Roche's Brand)	50.9	58.0	50.0	50.7	50.6

This series of experiments prove that 20 minutes at 50° C. give figures for the iodine number, which are a trifle too high. We therefore repeated the determinations at 40° C. with the same regular result, but found that 20 minutes gave somewhat too low figures. Finally, we resorted to heating at 45° C. and obtained the most constant and satis-





Meeting held Saturday, June 9th, 1894.

PROFESSOR A. SMITHIES IN THE CHAIR.

## CONTRIBUTIONS TO THE ANALYSIS OF FATS. IV.—COLOUR REACTIONS.

BY DR. J. LASKOWITZ.

BEFORE the "quantitative reactions" had opened out a new field of research in the commercial analysis of fats and introduced scientific accuracy into this branch of technical analysis, the qualitative tests or colour reactions were chiefly relied upon, in conjunction with some physical constants and some organoleptic methods, to give such information as the chemist was required to furnish. It must be added that these colour reactions were resorted to only for want of better methods, as is still the case in other domains of technical analysis into which the light of chemistry has not fallen yet or not been allowed to fall.

Writers of older text-books were in the habit of giving tables and systematic schemes by means of which the individual fats, and especially the oils, were said to be classified and identified. Unfortunately, tables of this kind have been perpetuated in even more recent treatises, and the bewilderment of the analyst who tries to find his way through this almost inextricable maze of colour reactions is more easily imagined than described. Nor must it be forgotten that those colour reactions were not always obtained with typical samples, representing what they professed to be, little or no regard having been taken of their source, their age, their mode of purification, and all that host of circumstances that have a vital influence on the colour the reagents produce. With the progress of technical knowledge the purification of oils has simultaneously gone apace, and a number of impurities, the very substances that gave origin to the colours supposed to be characteristic, have ceased to occur in commercial samples.

No wonder therefore that most of that class of tests have been abandoned, and that even some writers have gone so far as to banish altogether colour reactions—with very few exceptions—from the examination of fats and oils.

A given colour reaction can only be of value if it be produced by a well-defined substance, naturally occurring in the oil or fat, and characteristic of it to such an extent that it may be easily identified by that reaction. Of course these characteristic substances, occurring only in minute quantities, must not be easily removable by the processes of refining usually applied in practice.

As a type of a most valuable reaction of this kind may be considered Baudouin's test for *sesamé oil*, which has recently obtained its scientific explanation and confirmation by the isolation of the colour-producing principle in that oil. The colour reaction for cholesterol, although highly characteristic, requires more circumspection, as other substances also give the same or a very similar coloration (this Journal, 1892, 144).

I do not purpose reviewing here all the colour reactions that have been recommended for the identification of individual oils—such as Beechi's test for cotton-seed oil—as I hope to treat this subject shortly in another place. It is rather my intention to deal here with—and to dispose of—some reactions that are still looked upon as allowing a classification of oils and fats.

The following four reagents, sulphuric acid, chlorine gas, phosphoric acid, and phosphomolybdic acid, will be considered here, other group-reagents, as caustic alkalis, nitric acid, &c., being absolutely valueless for purposes of classification.

### A.—Sulphuric Acid.

Small quantities of impurities, such as resinoid and albuminoid substances being apt to produce colorations with sulphuric acid, the sulphuric acid colour test is of but limited value.

At best it is possible, with a good deal of practice, to discriminate between drying oils, semi-drying, and non-drying oils, if the acid be applied to the oil direct. The

first-named oils may be recognised by their forming dark clots when, say, two drops of acid are stirred into 20 drops of oil. A discrimination between semi-drying and non-drying oils, however, is more difficult, and in fact scarcely possible in every case.

The better drying an oil is the darker it will be coloured by the acid, so that it may be possible, judging from the depth of the colour, to distinguish between oils standing at the extreme ends of these classes, such as cotton-seed oil and olive oil, whereas it is impossible to differentiate by this test alone, e.g., rape oil from cotton-seed oil. The colour reactions obtained with a solution of the oil in carbon bisulphide cannot be said to yield more reliable results, the dilution tending to obliterate the otherwise sharp distinction between the eminently drying and the other oils.

In the case of the liver oils, the blue and purple colorations, due to the presence of cholesterol and colouring principles—lipochromes—are very characteristic; they are best observed if the oil be dissolved in carbon bisulphide previous to being treated with acid. But it should be pointed out that upon rancidity setting in, the chromatic principles undergo a change preventing the appearance of the blue colour, purple and red colorations being produced instead. Thus, rancid cod-liver oils, such as "cod oil" and "coast cod oil," not always show the fine blue colour a pure medicinal cod-liver oil will give. On the other hand, blubber oils will also give occasionally the blue colour, e.g., seal oil, so that it would not be safe to assume in such cases a—perhaps accidental—admixture with liver oil.

The sulphuric acid colour test may therefore only be used for purposes of preliminary information, and even then its indications should be interpreted with the greatest caution. It will be found far better to substitute for it the iodine absorption method, which, although entailing more work, furnishes an infallible means for classifying oils and fats.

### B.—Chlorine Gas.

Chlorine gas has been proposed by Fauré (1839) as a general reagent for the distinction of vegetable oils from animal oils, the latter oils—with the exception of the foot oils from terrestrial animals—being said to become black on treatment with this reagent, whereas the vegetable oils are hardly changed. Seal oil was later on added to the exceptions, but still chlorine gas has been generally retained as a reagent characteristic of the fish, liver, and blubber oils (the liquid waxes, sperm oil, and bottle-nose or Arctic sperm oil, being included in the last group).

Being in possession of a large number of oils and fats of undoubted genuineness (many of them had been prepared in the laboratory, especially the oils and fats from terrestrial animals, whereas the others were obtained from absolutely reliable sources as genuine), I have treated most of them with chlorine gas under exactly the same conditions. The following table contains the results:—

### OILS AND FATS TREATED WITH CHLORINE GAS.

#### A.—Vegetable Oils.

No.	Class of Oil.	Kind of Oil.	Colour after Treatment.
1	Drying oils.	Linseed, No. 1	Green
2		" No. 2	Slightly darkened
3		" No. 3	Slightly darker than No. 2.
4		Poppy seed	Unchanged
5	Semi-drying oils.	Cotton "	Slightly darkened
6		Cotton seed heated to 200° C.	Turned dark
7		Sesame, No. 1	Unchanged
8		" No. 2	"
9		Brazil nut	Slightly darkened
10		Rape oil, "cola"	Unchanged
11		" "	"
12		" "	Darkened

## A.—Vegetable Oils—cont.

No.	Class of Oil.	Kind of Oil.	Colour after Treatment.
13	Semi-drying oils.	Mustard seed	Darkened quickly, becoming dark brown.
14		Cotton	Turned dark, then greenish.
15		Castor, No. 1	Turned slightly brown
16		" No. 2	Unchanged
17		Peach	"
18	Non-drying oils.	Almond, bitter	Darkened very slightly
19		" sweet	Darkened less than No. 18.
20		Arachis	Unchanged
21		Olive, Tuscan	"
22		" Galipol.	"
23	Vegetable fat	Laural	Darkened

## B.—Animal Oils.

No.	Class of Oil.	Kind of Oil.	Colour after Treatment.
24	Fish oils	Japan fish	Turned light red, then dark reddish brown.
25		Cod liver, medicinal	Turned light brown.
26	Liver oils	" " rancid	Black.
27		Cod	"
28		Cod, East Coast	"
29		" rancid	Very deep brown.
30		" Norwegian	Almost black.
31	Blubber oils	Seal	Light brown.
32		Seal, Newfoundland "straw,"	Darkened, becoming very deep brown.
33		Right whale, pale	Turned brown.
34		" " dark	Nearly black.
35	Blubber oils, liquid waxes	Sperm No. 1	Very slightly brown.
36		" " 2	Light brown.
37		Bottle-nose (Arctic sperm), No. 1.	Very slightly brown.

## B.—Animal Oils—cont.

No.	Class of Oil.	Kind of Oil.	Colour after Treatment.
38	Blubber oils, liquid waxes.	Bottle-nose (Arctic sperm), No. 2.	Light brown.
39		Bottle-nose (Arctic sperm), No. 3.	"
40		Horses foot	Almost black.
41	Terrestrial animal oils	Neats "	Unchanged.
42		Sheeps "	"
43		Tallow oil	Darkened slightly.
44	Animal fat	Lard "	Darkened very slightly.
45		Horse fat	Became lighter.

From an inspection of this table the conclusion becomes evident that the chlorine cannot be admitted as a reagent for marine animal oils, the black colour being conditioned by the state of purity, No. 9—13, the treatment, No. 6, and especially the state of rancidity, and not by the origin of the oil. In fact, the pure cod-liver oil (25), and seal oil (31), changed their original colour so slightly when compared with No. 6, 9, 13, that they might be placed—judged by the chlorine test alone—amongst vegetable oils. On the other hand, horses foot oil, No. 40 (rendered some years ago by myself), became almost black.

The chlorine test must therefore be struck out of the list of special reagents for marine animal oils.

## C.—Phosphoric Acid.

In the table given by Grace Calvert (1854) syrupy phosphoric acid appears as a useful reagent for marine animal oils. Schaedler recommends it most strongly for this purpose, substantiating its claim by the assertion that even 0.1 per cent. of a marine animal oil may be detected by means of the red coloration phosphoric acid produces. Holde (this Journal, 1890, 419) has already stated that the phosphoric acid test is uncertain, as on the one hand resin oils also produce the red colour with this acid, and on the other hand distinct colorations only appear when large quantities of blubber oils are present in other oils. The following table proves that no reliance must be placed on this reaction, as the acid seems only to indicate impurities in oils, that can be eliminated by refining (No. 26), or products of oxidation (No. 7) or rancidity (No. 16).

## OILS TREATED WITH SYRUPY PHOSPHORIC ACID.

1 Volume of Oil with 5 Volumes of Acid.

## A.—Vegetable Oils.

No.	Class of Oil.	Kind of Oil.	Acid and Oil allowed to stand in the cold: not shaken.	Acid and Oil shaken after warming.
1	Drying oils.	Linseed, No. 1.	No reaction	Greenish-brown.
2		" No. 2.	Slight brown ring	"
3		Henap seed	Very slight ring	Brown.
4		Papay seed	No reaction	Very slight coloration.
5		Cotton seed, fresh	"	Light brown.
6	Semi-drying oils	" old.	Brown ring, darker than No. 15.	Dark brown.
7		" heated to 249° C.	Dark brown ring	Black.
8		Sesame	No reaction	Yellowish-green.
9		Rape oil, colza	"	Slight coloration.
10		" pale.	Slight brown ring.	" "
11		Croton	No reaction	Brown, acid layer black.
12		Castor	"	Very slight coloration.
13		Olive	"	" "

## B.—Animal Oils

No.	Class of Oil.	Kind of Oil.	Acid and Oil allowed to stand in the cold, not shaken.	Acid and Oil shaken after warming.
14	Fish oils	Japan fish	Reddish-brown ring	Deep brown.
15		Cod-liver, medicinal	Light brown ring	Brown.
16		" rancid	Black ring, strongest reaction	Dark brown.
17	Liver oils	Cod oil	Black ring	Black.
18		" Norwegian	Reddish-brown ring	Deep brown.
19		" "Coast cod" oil	Deep reddish-brown ring	"
20	Blubber oils	Seal oil	Dark brown ring	Dark brown.
21		" Newfoundland	Red ring	Brown.
22		Right whale, pale	Slight brown ring	"
23		" dark	Black ring	Black.
24		Sperm	Dark brown ring	Dark brown.
25	" " liquid waxes	Bottle-nose (Arctic sperm)	" "	Black.
26		" refined	Brown ring	Brown.
27		Horses foot	Black ring	Black.
28		Neats foot	No reaction	Brown.
29	Terrestrial animal oils	Sheeps foot	"	Slight coloration.
30		Tallow oil	Very faint brown ring	Brown, darker than 28.
31		Lard oil	" " "	Dirty yellow.

## C.—Phospho-Molybdic Acid.

Welmans (this Journal, 1892, 548) has proposed this reagent for the detection of seed-oils in lard, and generally for the discrimination of vegetable oils from oils and fats of animal origin, with the exception of cod-liver oil. Guided by the consideration that the fixed oils as products of the vegetable organism may contain minute traces of alkaloids or glycosides, he applied to a number of vegetable oils the reagents used for the detection of alkaloids, with the result that phospho-molybdic acid was found suitable.

The reaction is carried out as follows:—1 gram. (or 25 drops) of an oil or fat is dissolved in 5 cc. of chloroform in a test-tube and agitated with 2 cc. of a freshly-prepared solution of phospho-molybdic acid, or of sodium phospho-molybdate and a few drops of nitric acid. After standing for a short time, the chloroformic layer becomes colourless, whereas the upper layer shows, in the case of a vegetable oil and cod-liver oil, a green colour. On adding ammonia or a fixed alkali a beautiful blue colour appears, the intensity

of which corresponds to that of the green tint noticed before. Animal fats, such as tallow, goose fat, butter fat, and lard do not give this reaction, according to Welmans, nor is a green colour said to be obtained with vegetable oils refined by means of acids, as rape oil, or only after very long standing (compare, however, rape oil, in the following table). This reaction has been recommended by several chemists—Engler and Rupp, Wimmer, Mansteld, Goske—with the only limitation by Wallenstein (this Journal, 1893, 55), that also tallow ("premier jus") gives a green resp. blue coloration.

The importance of a reaction of this kind naturally attracted my attention, and I made an extensive trial of it with the result tabulated below. The extreme sensitiveness of the phospho-molybdic acid to minute traces of reducing substances such as impurities in commercial ammonia (organic bases) required a preliminary examination of the ammonia and the chloroform used. Both left the phospho-molybdic acid yellow, and the latter became colourless on supersaturation with ammonia.

## OILS AND FATS TREATED WITH PHOSPHO-MOLYBDIC ACID.

## A.—Vegetable Oils and Fats.

No.	Class of Oil.	Kind of Oil.	Colour of the Acid Layer.	Colour when made Alkaline with $\text{NH}_3$ .
1	Drying oils	Linseed, No. 1	Light green	Blue.
2		" 2	Darker green	Deep blue.
3		" 3	Green	"
4		Hempseed	"	"
5		Peppy seed	Very light green	Bluish.
6		Cotton seed	Green	Especially blue.
7	Semi-drying oils	" heated to 210° C.	Greenish	Faint "
		After standing for some time: green, and then	green, and then	Blue.
8		Sesame No. 1	Greenish yellow	Bluish.

A.—Vegetable Oils and Fats—continued.

N.	Class of Oil.	Kind of Oil.	Colour of the Acid Layer.	Colour when made Alkaline with $\text{NH}_3$
		Sesame No. 2.....	Green.....	Deep blue.
10		Brazil nut.....	".....	Bluish.
11		Rape oil edza No. 1.....	".....	Deep blue.
12		" " 2.....	".....	"
13	Semi-drying oils.....	" pale.....	Light green.....	"
14		" dark.....	Deep ".....	"
15		Mustard seed.....	Green.....	Blue.
16		Cream.....	".....	Deep blue.
17		Castor No. 1.....	".....	"
18		" 2.....	".....	"
19		Peach.....	(a.) <i>Pale yellow.</i> (b.) Very faint green.	(a.) <i>Nearly colourless.</i> (b.) Very faint blue, almost colourless.
20		Almond, sweet.....	(a.) <i>Yellow.</i> (b.) Very slight green	(a.) Faint blue. (b.) Faint blue.
21		" bitter.....	" " "	(a.) " (b.) Very faint blue.
22		Arachis.....	(a.) " (b.) Faint green....	(a.) <i>Nearly colourless.</i> (b.) Bluish.
23		Olive, Tuscan No. 1.....	(a.) <i>Greenish yellow.</i> (b.) Very faint green.	(a.) Blue. (b.) Very faint blue.
24		" " 2.....	Greenish.....	Blue.
25	Non-drying oils.....	" Zante.....	".....	"
26		" Syrian.....	".....	"
27		" Messina.....	(a.) <i>Yellow.</i> (b.) Very slight green	(a.) Very faint blue. (b.) Bluish.
28		" Adramyti.....	" " (b.) <i>Yellow</i> .....	(a.) Bluish. (b.) Very faint blue.
29		" Tuscan cream.....	" " "	" " "
30		" Candia.....	" " (a.) Very slight green	(a.) Bluish. (b.) Blue.
31		" Gioja.....	<i>Yellow</i> .....	Bluish.
32		" Gallipoli.....	(a.) <i>Yellow.</i> (b.) Greenish.....	(a.) Bluish. (b.) Bluish.
33		Laurel.....	Green.....	Very deep blue.
34		Mowrah seed.....	Light green.....	Bluish.
35	Vegetable fats.....	Palm.....	Deep ".....	Deep blue.
36		" bleached.....	Yellow.....	Very light blue.
37		Palmnut.....	<i>Yellow</i> .....	Very faint blue.
38		Cocconut.....	(a.) <i>Yellow.</i> (b.) <i>Yellow</i> .....	(a.) Very faint blue. (b.) Faint blue.

B.—Animal Oils and Fats.

No.	Class of Oil or Fat.	Kind of Oil or Fat.	Colour of the Acid Layer.	Colour when made Alkaline with $\text{NH}_3$
39	Fish oils.....	Japan fish oil.....	Deep green.....	Very deep blue.
40		Cod liver, medicinal.....	Very deep green.....	" "
41		" " rancid.....	" ".....	" "
42	Liver oils.....	Cod oil.....	" ".....	" "
43		" Norwegian.....	Deep green.....	Deep blue.
44		Coast cod.....	".....	"
45		Seal oil.....	Very dark green.....	Very deep blue.
46	Bulbous oils.....	Right whale, No. 1.....	Dark green.....	" "
47		" " No. 2.....	".....	" "
48		Sperm, No. 1.....	Green.....	Deep blue.
49		" No. 2.....	Greenish.....	Blue.
50	Bulbous oils, liquid waxes.	" No. 3.....	Green.....	"
51		Bottle-nose (Arctic sperm) No. 1.....	".....	Deep blue.
52		Bottle-nose (Arctic sperm) No. 2.....	(a.) Greenish. (b.) <i>Greenish</i> .....	(a.) Blue (b.) Blue.
53		Bottle-nose, refined.....	Yellow.....	Bluish.

## B.—Animal Oils and Fats—continued.

No.	Class of Oil or Fat.	Kind of Oil or Fat.	Colour of the Acid Layer.	Colour when made Alkaline with $\text{NH}_3$ .
54	Terrestrial animal oils.	Horsefoot.....	Green .....	Distinct blue.
55		Sheepfoot.....	(a.) Yellow, (b.) Yellow.....	(a.) Faint blue, (b.) Faint blue.
56		Neatsfoot.....	(a.) Yellow, (b.) Yellow.....	(a.) Faint blue, (b.) Bluish.
57		Yellow oil, No. 1.....	(a.) Green, (b.) Green.....	(a.) Blue, (b.) Bluish.
58		Lard oil.....	Yellow .....	Bluish.
59	Animal fats.....	Horse fat.....	Deep green .....	Deep blue.
60		Beef tallow .....	(a.) Yellow, (b.) Yellow.....	(a.) Very faintest blue, (b.) Bluish.
61		Lard, freshly rendered, unfiltered.	(a.) Yellow, (b.) Yellow, (c.) Yellow.	(a.) (b.) (c.) Colourless.
62		Lard, freshly rendered, filtered.	(a.) (b.) (c.) Yellow .....	" "
63		Lard, six years old, slightly rancid.	(a.) Yellow, (b.) Yellow.....	(a.) Very faint blue, (b.) Faint blue.

A glance at the tables proves clearly that, even excepting the liver oils, a distinction between vegetable and animal oils and fats cannot be made by means of the phospho-molybdic acid. Several kinds of olive oil as also almond oil, arachis oil, and peach oil, showed far less distinct reactions than tallow oleine and even lard oil. The reduction of the phospho-molybdic acid is evidently caused by minute quantities of impurities in the oils and fats themselves or by some secondary products appearing with even slight rancidity setting in. This is most notably proved by the reaction obtained in the case of No. 63.

Of all the samples tested, only lard had remained colourless, and there seemed to be ground for the opinion that cotton-seed oil or any other seed oil employed for adulterating lard might be easily detected by phospho-molybdic acid. I prepared therefore a number of mixtures of pure, freshly-rendered lard, yielding no coloration in Welmann's test and of cotton-seed oil giving a distinct blue reaction. The results are given in the subjoined table.

## MIXTURES OF LARD AND COTTON-SEED OIL TESTED WITH PHOSPHO-MOLYBDIC ACID.

No. 1.	Lard.	Cotton-seed Oil.	Colour of the Acid Layer.	Colour when made Alkaline with $\text{NH}_3$ .
	Per Cent.	Per Cent.		
1	100	..	Yellow	Colourless.
2	95	5	"	"
3	90	10	"	Almost colourless.
4	85	15	"	Very faint blue, like No. 63 of preceding table.
5	80	20	"	Faint blue.
6	75	25	"	"
7	70	30	"	"
8	65	35	Slightly greenish	"
9	60	40	"	Bluish.
10	50	50	Greenish	"
11	45	55	"	"
12	40	60	"	"
13	35	65	"	"
14	30	70	Slight green	"
15	25	75	"	"
16	20	80	"	"
17	15	85	Green	Blue.
18	10	90	"	"
19	5	95	"	"
20	0	100	"	"

There was a gradual increase in depth of tint, which, of course, could not be expressed in the table as distinctly as would be necessary to mark the slight increments.

This table clearly demonstrates the necessity of using Welmann's test with great caution. In practical analysis, an adulteration with 10 per cent. of cotton-seed oil would escape detection if freshly-rendered lard had been used. Indeed, pure lard mixed with 15 per cent. of cotton-seed oil gave a reaction just like sample No. 63 in the preceding table. If in the examination of that sample of lard preponderant importance had been given to the results of Welmann's test, an adulteration with from 15 to 20 per cent. of cotton-seed oil would have been pronounced upon. As all other physical and chemical constants would have pointed to a normal lard, the analyst would have naturally proceeded to look for the compensating beef stearine, and he would perhaps—under the strong bias—succeeded in finding it, and thus might have added another page to the already too voluminous chapter of adulterations, winding up with a panegyric on the ingenuity of the adulterator, who is credited with being several years ahead of the analytical chemist.

The fact that rancid lard has a reducing action on phospho-molybdic acid is a welcome corroboration of Bevan's (Analyst, 1894, 88) recently published statement that lard which had been exposed to air gave a strong reaction with silver nitrate. Both reactions show that products of an aldehydic nature are formed when the fat turns rancid.

Welmann's reaction can therefore be only admitted as a preliminary test, the indications of which may in some cases give valuable hints as to the directions in which the examination of a given sample has to be carried out, or may serve as a useful corroboration of results obtained by other methods. This becomes all the more evident if we examine the following table, containing some unsaponifiable substances often met with in the examination of fats and oils.

## BEHAVIOUR OF UNSAPONIFIABLE SUBSTANCES WITH PHOSPHO-MOLYBDIC ACID.

No.	Substance.	Colour of the Acid Layer.	Colour when made Alkaline with $\text{NH}_3$ .
1	Cholesterol.	Yellow.	Colourless.
2	Resin oil.	Very deep green.	Very deep blue.
3	Mineral oil.	Green.	Blue.
4	Mineral oil, Russian.	Greenish.	"
5	Mineral oil, Scotch, No. 1.	Green.	"
6	Mineral oil, Scotch, No. 2.	"	"
7	Mineral oil, Scotch, No. 3.	"	"
8	Kerosene, American.	Very slightly green.	Very faint blue.
	Kerosene, Russian.	Yellow.	"

## Journal and Patent\* Literature.

Class.	Page
I.—General Plant, Apparatus, and Machinery .....	622
II.—Fuel, Gas, and Light .....	625
III.—Destructive Distillation, Tar Products, &c. ....	627
IV.—Colouring Matters and Dyes .....	628
V.—Textiles: Cotton, Wool, Silk, &c. ....	632
VI.—Dyeing, Calico Printing, Paper Staining, and Bleaching .....	633
VII.—Acids, Alkalis, and Salts .....	634
VIII.—Glass, Pottery, and Enamels .....	637
IX.—Building Materials, Clays, Mortars and Cements..	637
X.—Metallurgy .....	639
XI.—Electro-Chemistry and Electro-Metallurgy .....	645
XII.—Fats, Oils, and Soap Manufacture .....	647
XIII.—Pigments and Paints: Resins, Varnishes, &c. ; India-Rubber, &c. ....	648
XIV.—Tanning, Leather, Glue, and Size .....	649
XV.—Mannres, &c. ....	652
XVI.—Sugar, Starch, Gum, &c. ....	652
XVII.—Brewing, Wines, Spirits, &c. ....	653
XVIII.—Chemistry of Foods: Sanitary Chemistry and Water Purification; Disinfectants .....	656
XIX.—Paper, Pasteboard, &c. ....	657
XX.—Fine Chemicals, Alkaloids, Essences, and Extracts	657
XXI.—Photographic Materials and Processes .....	659
XXII.—Explosives, Matches, &c. ....	659
XXIII.—Analytical Chemistry .....	661

## I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

*The Prevention of Scale in Boilers.* J. Walter. Chem. Ind. 16, 487.

If the water is fed into the boiler, together with small quantities of a solution of sodium carbonate, the deposit of scale is not prevented, but is localised more upon those parts which are nearest to the opening of the feed-water pipe in the boiler. The probable cause of this is the difference between the temperature of the water in the boiler and that of the feed-water. If therefore the temperature of the feed-water were raised to the temperature of the boiler before coming in contact with the latter, all incrusting matters would be precipitated beforehand and no scale could form upon the boiler plates. To effect this result a 6-in. tube is placed on low supports along the centre of the boiler at a distance of about 6 in. from the bottom. The front end of this tube is open, the back end closed with a blank flange. From the back end rises a vertical pipe reaching above the level of the water in the boiler and loosely encasing the feed-water pipe. The water fed into the boiler does not therefore mix immediately with the water in the boiler, but passes first through the narrow vertical tube and subsequently through the wider horizontal tube. During this passage its temperature rises to that of the boiler, and the incrusting matter is precipitated within the horizontal tube, no subsequent formation of scale upon the boiler plates taking place. Any of this precipitate washed out of the tube, will be found to form only a layer of fine sludge on the bottom of the boiler or a granular coating on the sides, which is easily removed by a brush. The greater part of the precipitated incrusting matter will be found in the horizontal pipe, which after being cleaned is reinserted. This arrangement was found to form a reliable means of preventing the formation of hard scale.—C. O. W.

## PATENTS.

*Improvements in and relating to Apparatus for Heating or Evaporating Liquids by Means of Steam.* D. B. Morrison, Hartlepool. Eng. Pat. 9548, May 12, 1893.

This invention relates to heaters composed of tubular coils inserted in evaporating vessels, and consists in arranging the coils in such a way as to facilitate their withdrawal from the vessel for cleaning or other purposes without necessitating the breaking of any pipe joints. For this purpose the coils are connected to the pipes both for the admission of the steam and for the removal of the condensed water, by means of hollow taper plugs on which they can be moved as on hinges, and can be swung clear from the inside to the outside of the vessel whenever required. The figures accompanying the specification illustrate a variety of arrangements based on the same principle.—B.

*Apparatus for Extracting Substances by Volatile Solvents.* J. Meikle, Glasgow. Eng. Pat. 10,701, May 31, 1893.

A closed vessel, divided into upper and lower compartments by a horizontal perforated partition, is suitably jacketed at its ends and sides, and the two compartments are provided with heaters and stirrers. The substance to be treated is charged into the upper compartment, and the solvent is sprayed over it by means of a perforated ring pipe, while the substance may be stirred and heated as desired. The solvent passes through the perforated diaphragm, and falls to the bottom of the vessel, where it may be vaporised by the heater and caused to pass by a central vertical pipe to the upper chamber, where it condenses on meeting with the upper cover, through which latter a current of cold water is passing. The liquid, thus condensed, gravitates through the substance and collects below, where it is vaporised again; a continual circulation is thus kept up. The extracted substance, with the solvent, is finally drained off from below and the vessel is cleared for a fresh charge of substance.—E. G. C.

*Improvements in Apparatus for Cooling, Heating, Drying, or similar Purposes.* J. Fischer, Vienna. Eng. Pat. 10,870, June 5, 1893.

This invention applies to apparatus in which the heating or cooling unit is the flat hollow plate, through which the heating or cooling medium circulates, and the improvements refer to the method of constructing the plates, and of connecting the various units together in a way to secure due circulation from one to the other and the regular withdrawal of the condensed liquor. In some cases the induction of counter-currents in the liquids or gases requiring to be heated or cooled is also provided for.—B.

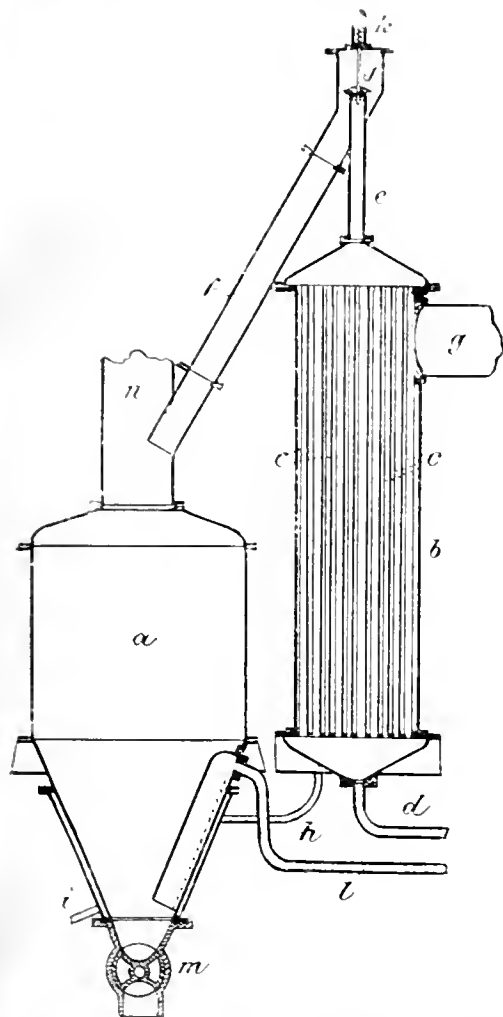
*Improvements in Evaporating Brine and other Liquors, and Apparatus therefor.* F. W. Scott and F. W. Scott, jun., London, and E. G. Scott, Liverpool. Eng. Pat. 12,774, June 29, 1893.

In the apparatus hitherto used for the evaporation of brine or other liquids *in vacuo* the heating system has been generally situated within the evaporating vessel, whereby crystals or other deposits frequently covered the heating surfaces and so impaired their efficiency. The object of this invention is to overcome that difficulty by heating the liquid in a separate vessel, before allowing it to enter the evaporating vessel, in which the crystals are subsequently thrown down under the influence of the vacuum. The accompanying figure illustrates the principle.

Here *b* is the heating vessel traversed by the tubes *c*, fed with the liquid through the inlet tube *d*. It is surrounded by the tube *e*, closed at its upper end by the valve *j*, weighted to any desired degree by the spring *k*. The heating steam enters the vessel *b* by the tube *g*, and the heated liquid escapes under pressure by the tube *f* to the evaporating vessel *a*, wherein the crystals are deposited under the action of the vacuum, and are removed through

\* Any of these specifications may be obtained by post by remitting sd.—the price now fixed for all specifications, postage included—to Sir Henry Reader Luck, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

the valve *m*, which is constructed as described in a previous patent, Eng. Pat. 6478 of 1892 (this Journal, 1893, 347). Any remaining liquid is drawn off through the pipe *l*, and



the water of condensation from the vessel *b* finds its way through the tube *h* into the hot-water jacket surrounding the vacuum vessel, and can be withdrawn at *i*. The system can also be applied to multiple effect apparatus.—B.

*Improvements in the Construction of Blast Furnaces, Shafts, or Chimneys, or like Structures, and in Bricks or Blocks to be employed therein.* W. P. Ingham, Middlesbrough-on-Tees. Eng. Pat. 12,910, July 1, 1893.

IN the construction of blast furnaces and other structures built of bricks or blocks it has hitherto been usual to form the blocks in plan of a tapered shape, the sides of each block being formed in continuation of radial lines drawn from the centre or axis of the structure, but this method of construction does not form a structure that will resist much internal pressure.

The patent relates to the use of bricks of such shape that the joints between them are not radii of the circular cross section of a structure of the class of a chimney or blast furnace, but are tangential to a circle of moderate radius concentric with the circle constituting the cross section. Drawings are given of bricks which fulfil this condition. Greater stability of structure is thus attained.

—B. P.

*Improvements in Apparatus for Crushing Dry or Wet Materials, such as Cement, Lime, Powder, Anthracite, Kainite, Quartz, Phosphate Minerals, and the like.* C. Moré, La Roche, France; J. A. Hempel, Dronne, France. Eng. Pat. 13,771, January 22, 1894.

THE crushing mill consists of a heavy annulus weighing several tons provided with a recess or groove on its inner surface for the reception of the material to be crushed. The ring is supported by two rollers running in the track formed by the inner groove, and one of these rollers is rotated by any suitable means to cause the annulus to revolve. The material is fed into the hopper of the machine by an Archimedean screw arrangement, or any equivalent device, from whence it is delivered into the annular groove and is distributed evenly by means of spherical rollers. The material passes over the rollers, where it is crushed, and afterwards meets an adjustable angled scraper, which delivers it into a sieve or other receptacle. E. G. C.

*Apparatus for Distilling and Sterilising Water.* J. Nagel, Chemnitz, Saxony. Eng. Pat. 25,888, February 6, 1894.

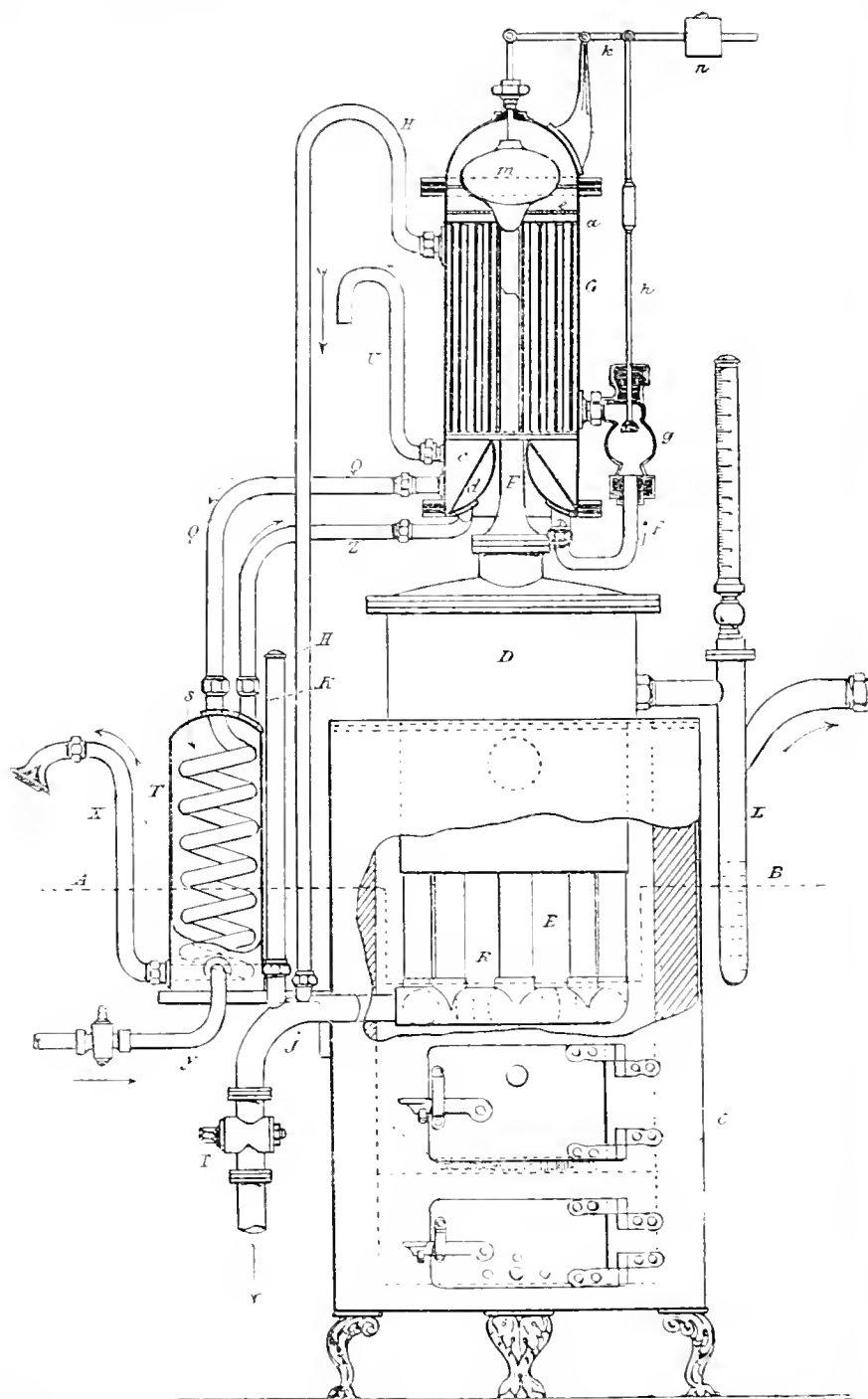
THIS apparatus (see next page), which is designed for the simultaneous distillation and sterilisation of water, is constructed as follows:—

The water to be treated enters the closed vessel *F* by the pipe *Y*, rising by the pipe *Z* into the space *d* under the condensing vessel *G*, thence by the pipe *f* and through the regulating valve *g* into the body of the said vessel *G*, where, after filling the space surrounding the vertical tubes *N*, it finds an outlet near the upper end through the bent tube *H*. On descending the latter tube (the cock *r* being closed) the water reaches the system of heating tubes *R* and *E* over the firegrate, and the evaporating vessel *D*, which it fills to a height indicated by the gauge glass *K*. The vessel *D* is surmounted by the steam-tube *F*, closed at its upper end by the weighted and balanced valve *m*, which passes through the perforated plate *e*, and is in connection with the regulating valve *g* through a lever and the rod *h*. On steam being raised to the desired pressure in the vessel *D* it escapes through the valve *m* into the dome-shaped space above, and, passing through the tubes *N*, is condensed. The resulting water drops into the conical space *c*, thence passing through the tube *Q* and the cooled coil *S* finally issues, as distilled water, by the pipe *X*. The small bent tube *V* acts as a safety-valve against undue steam pressure, whilst the siphon-pipe *L*, which can be provided with the thermometer shown, enables a portion of the heated water to flow off direct from the vessel *D* after having been sterilised by boiling. The automatic feeding of the apparatus is regulated by the action of the valve *m*, which, on rising through increased steam pressure, simultaneously opens the feed-valve *g*, thereby admitting fresh water, which in its turn has a cooling effect upon the contents of the vessel *D*, tending to reduce the steam pressure and to close the steam-valve. The tap *r* is provided for the purpose of emptying the apparatus.—B.

*Improvements in Apparatus for Separating, Classifying, and Purifying Minerals and other Materials.* A. F. Beyer and A. G. Beyer, Paris. Eng. Pat. 3478, February 20, 1894.

THE object of the invention is the simultaneous pulverisation and classification according to density, of minerals and other materials, and in particular, natural phosphates of lime, the classification being effected through the continuous agitation of the raw materials with air or other gas simultaneously introduced into the apparatus during pulverisation. A simple separating apparatus as constructed under this patent is composed of:—1st. A compressed air pulverising beater running at great speed. 2nd. A sloping delivery tube for the pulverised products, the axis of the mouth of the tube being set at an angle to a vertical plane passing through the axis of the beater. 3rd. A classifying chamber with centrifugal action placed at the far end of the delivery tube. 4th. An auxiliary fan placed upon the axis of the beater for introducing a current of air in the path of the





material leaving the sorting apparatus. Modifications are included both in the treatment and in the apparatus, as well as in the construction and arrangement of details.—B.

*Improvements in Apparatus for Creating a Vacuum in Vacuum Pans.* P. Pié, Lima, in the Republic of Peru. Eng. Pat. 4906, March 8, 1894.

A CENTRIFUGAL pump has branch pipes connected to the vacuum pan and to a cold-water supply, so that when the pump is set in motion, the steam from the vacuum pan is

drawn into the inlet pipe by the action of the pump, where it meets with the cold water. The vapours immediately condense by contact with the cold water and are discharged through the outlet pipe. The cold water is finely divided by a rose, and can be regulated by a cock so as to produce the required vacuum.—E. G. C.

## II.—FUEL, GAS, AND LIGHT.

*Liquid Fuel.* G. Stockfleth. See Arts. 42, 616—625.

In introducing the author—formerly engineering expert of the Nobel Petroleum Production Company.—Mr. Boverton Redwood animadverted upon the trifling progress made in the practice of liquid fuel burning in this country, since Dr. B. H. Paul's paper before the Society in 1868, although he himself, in 1886, had been able to describe its then already very extended use in Russia, this use being largely due to the efforts of Mr. Uspubart, the locomotive superintendent of the Grazi-Tsaritzin Railway.

After some remarks upon the oils suitable for liquid fuel and upon the distillation of petroleum, Mr. Stockfleth stated that in 1892 the production of Russian *astatki* (residue remaining in the petroleum stills at 300—320° C.) was about 3,350,000 tons, of which 3,000,000 tons were exported to Caspian ports, and 250,000 tons to Batoum, while 100,000 tons were consumed in Baku.

*Astatki* is even used for domestic cooking and heating by leading it from a tank, at the top of the house, to the various stoves, where it drips on to a small cast-iron plate placed in front of the stove door, which is provided with a small opening, creating a strong draught; when once the plate is warm and the dripping of the oil properly regulated, it burns without further attention.

For the larger uses (steam-raising, distilling, &c.) many "*fasunkas*" (atomisers or injectors) have been invented; but "it has been found that the most primitively constructed pulverisators answer as well as the more complicated kinds." That atomiser which at present is giving "entire satisfaction" under the still, neither wasting drops of oil nor giving smoke nor chimney-flame, is made by simply flattening under the hammer the ends of two  $\frac{1}{2}$  in. pipes, so that the apertures are about  $\frac{1}{4}$  in.  $\times$   $\frac{1}{8}$  in. or  $\frac{1}{16}$  in., and binding them together with a piece of wire; steam is then led through the one, its jet catching and completely spraying the slightly warmed *astatki*, which descends by gravitation in the other.

According to the author, the action of the steam is "solely mechanical and serves only for cutting up the oil in small particles, which, being surrounded by the necessary air for their combustion, catch fire before they reach the bottom of the flue." Compressed air has been tried for spraying *astatki*, "but the results have not materially differed from those obtained with steam." The author points out that any chemical action which the steam might have upon the oil would probably already have taken place in the stills, where steam is used to facilitate distillation; the question of the possible dissociation of the steam, at or near the moment of combustion of the *astatki*, was not discussed in the paper.

The amount of steam, consumed in atomising the *astatki*, is stated to be "usually estimated at from 5 to 7 per cent." in Russia; but (in the discussion of the paper) Mr. E. Charrington gave 2½ per cent., as the result of his experience with stationary boilers; Mr. Nelson Boyd stated that, in the recent voyage of the "*Baku Standard*" across the Atlantic, each ton of liquid fuel had consumed a ton of water as atomising-steam, a result which, Mr. E. Henwood and Mr. W. Warren explained, was probably due to the wasteful and imperfect apparatus employed on the ship; whilst Mr. Boverton Redwood, in alluding to the importance of this part of the subject, stated that some burners used much more than others, and that, in comparative critical trials, many of the modern burners would probably be found to yield more economical results than the somewhat primitive Russian apparatus described by the author.

The advantages of liquid fuel are:—ready adaptability to existing furnace arrangements; greater steam-raising power; complete and ready control over the fire, and absence of smoke and ash. Steam can, by this means, be very rapidly raised and the fire can be instantly lighted or extinguished; there is little waste and noise of steam by the safety-valve, and the boiler-pressure can more readily be regulated. The furnace doors need seldom be opened, heat being thereby saved and leaky boiler-tubes less frequently caused by cold air currents. Great saving (about

two-thirds) of the space at present devoted to storage would be effected, for only half the tonnage of stock need be stored, and this amount would occupy a less space than the same quantity of coal. Almost all the labour employed in storing, loading, and handling coal, as also that of feeding and stoking the fires, is directly saved.

As to ships, the valuable space, given up at present to coal bunkers, becomes available for cargo, the oil being stored in ballast tanks at the bottom of the ship; the stability of the ship is thereby augmented, and the oil, as consumed, is replaced in the tanks by water. One-quarter the number of stokers are required, and the size of the stoke-hole may be greatly reduced; the danger of fire in the coal-bunkers will no longer exist, and there is less risk of accidents from scalding when water, during stormy weather, puts the fires out. Liquid fuel is especially suitable for torpedo boats.

As yet there is no boiler specially designed for liquid fuel in the market but the author considers the recent water-tube type much more suited to the fierce fire of liquid fuel than are older types, with straight or curved surfaces of ordinary thick boiler-plate.

As to the cost of liquid fuel, the author did not consider it "at present very probable that this fuel will be adopted to any considerable extent in this coal-producing country, with its annual output of 185,000,000 tons," but that in other parts where oil is found on the spot and coal is expensive, very good markets could certainly be created.

In the discussion, Mr. Nelson Boyd did not think any kind of liquid fuel could be delivered, in the Thames under 2½d. per gallon; but calculating 2d. per gallon (16s. 8d. per ton) and allowing the efficiency of petroleum to be twice that of coal, this would compare with coal at 23s. per ton. Now, except in a time of coal famine, the best steam coals only cost (at Cardiff) 12s. to 13s., while a very good coal could be bought for considerably less. In steam-raising, Mr. Henwood had obtained an "evaporation" considerably over 3 to 1 as compared with coal, and he contended that English shale could yield petroleum at under 2d. per ton. Mr. Charrington found, in practice, *astatki* compared, as to efficiency, with coal in the ratio 2:1:1. *Astatki* was 25 per cent. dearer than green oil, creosote oil, and tar residuals; a ton of mixed green oil and creosote oil at 2d. per gallon was equal to 1 ton of coal at 16s. 6d. There were 25 oil-consuming locomotives on the Great Eastern Railway, and, during the coal strike, even petroleum had been found to pay; ordinarily, oils from various gasworks along the line were used; these oils cost ½d. per gallon delivered into tank trucks on the line. This use of liquid fuel was thus one existing under quite exceptional circumstances. Upon the questions of the present and future sources of the supply of liquid fuel, the author stated that it was improbable that any great supply could be expected from the American oil-fields; for only 20 per cent. of residuals, higher in density than kerosene, is yielded and part of this is used in preparing other petroleum products; the 65 per cent. of *astatki*, yielded by Russian petroleum, gives a considerable quantity available from these fields. As to whether other regions will ever yield such enormous quantities, as do these two, "all that can be said is, that several places in different parts of the world present quite as good indications of the presence of oil, as the others did before they were worked."

Among such promising countries are specially to be noted Java and Peru, which produce heavy petroleum, as also Burmah, Trinidad, and several of the West Indian islands, Canada, Mexico (both on the Pacific and the Gulf sides), Galicia, Sumatra, and Japan are also highly petroliferous countries.

During the discussion Mr. Henwood said, that although there was an immense amount of petroleum in Trinidad, the impost of 8s. per ton retarded its development. Venezuela also was highly petroliferous. Mr. Warren stated that the entire United States production for 1893 was 37,000,000 barrels, which would only produce 1,500,000 tons of *astatki* and light naphtha, and seeing that the Pennsylvania Railway used 5,000 tons of soft coal per diem, this company alone could consume the entire petroleum liquid fuel output of the States. The coal output of England for 1892 was

183,000,000 tons, and that of the United States 181,000,000 tons, or together nearly 400,000,000 tons, whilst the entire petroleum output of Russia and the States was together only 10,000,000 tons; it was therefore unlikely that the supply of oil would ever exceed the demand. Sir Westley Pervail mentioned the extensive petroleum deposits of New Zealand.

In some concluding remarks upon drilling for oil the author stated that, in the United States, a complete outfit costs 10,000 dollars, and drillers' wages average from three to four dollars per diem—if for work outside the States, 125 dollars per month and travelling expenses. In Russia the total cost of a well is about 3,000l. under contract. Before putting down such expensive wells, it is recommended to test a supposed petroliferous property by means of 4-in. holes carried down 500 ft., the necessary plant and casing for such work costing 350l. to 475l. Wells in Russia have a top diameter of 20–24 in. and a depth of about 800 ft., while in America the corresponding dimensions are 8 in. and 2,500 ft.

Mr. Byverton Redwood summed up the liquid-fuel question in the following sentences:—"Unfortunately, up to now, users and producers have waited for each other, and it seems that there ought to be a little more courage, on the part of those who have valuable sources of supply, in developing them. There is too much disposition to wait until they are assured of a demand for so many thousand tons per annum; while, on the other hand, those who might be inclined to use this material say they cannot be expected to make arrangements to use it until they are assured of a constant supply at a certain price. The result is almost entire stagnation."—E. R. B.

*Spontaneous Combustion of a Lamp Shade.* A. Dupré. Times, May 23, 1894.

See under Analyt. and Scient. Notes, page 670.

## PATENTS.

*Improvements in the Manufacture of Coke, Coal-gas, and the Obtaining of By-products, and in Apparatus employed therein.* P. Dvorkovitz, London. Eng. Pat. 5546, March 14, 1893.

According to this invention the oven, retort, or chamber is constructed so that heat can be applied to the interior of the mass of coal as well as to the walls of the chamber containing it. The external heat is applied by causing generator gas to circulate through suitable flues arranged round the chamber. To thoroughly heat the mass of coal, hydrogen—which may be in the form of water-gas, coal-gas, or highly superheated steam, either alone or mixed with air—is introduced into the chamber. These gases, which are introduced at a temperature above the initial heat of the chamber and its contents, mingle with the coal and rapidly effect the coking of it. The gas and by-products ("coal paraffin oil") produced in the chamber are exhausted through suitable outlets and treated in the ordinary manner. It is stated that a chamber containing five tons of coal, with a stream of gas equal to about 5,000 cub. ft. per hour at a temperature of 1,200° F., gives good results, and that the coke obtained is practically free from sulphur; also that, on account of the internal heating, that applied externally may be less than is now usual, and that the hydrogen introduced takes up nitrogen and forms ammonia. This invention may also be applied to the obtaining of by-products from shale and other carbonaceous matter, as well as from coal.—E. R. B.

*Improvement in or connected with Gas Hydraulic Mains.* J. H. R. Dinsmore, Liverpool. Eng. Pat. 5648, March 16, 1893.

The hydraulic main is divided into two compartments by a longitudinal partition plate extending from the cover plate nearly down to the bottom, so that the only communication between the two compartments is well below the level of the sealing liquor.

One compartment is open to atmospheric pressure; into the other pass the gas inlets, which dip into the sealing liquor, but only for a small depth. When there is no gas pressure on, these pipes are sealed, but when gas is passing into the main its pressure depresses the sealing liquor in the second compartment, a corresponding rise taking place in the first one, unseals the inlet pipes, and the gas passes along and out through the exit of the hydraulic main.—E. R. B.

*Retort and Apparatus connected therewith for the Purpose of Converting Coal-tar into Gas for Illumination and other Purposes.* John Smith, Upper Bangor, North Wales. Eng. Pat. 7605, April 11, 1893.

The inventor's intention is to produce permanent gas from coal-tar, the heat employed being but "little more than that necessary for vaporising the tar," while the solids left in the retort are such that they may be drawn off as pitch or converted into coke at will. An ordinary D or oval retort is employed, so set that the mouth is 3 ins. higher than the back, and is charged to a depth of 3 ins. with the coal-tar; the latter enters through a 1-in. cock, placed axially and near the top of the "ascension pipe." The vapours which accompany the gas are for the most part condensed in the hydraulic main and collected therefrom into a tank. This oil, while still warm, may most profitably be utilised as liquid fuel, but, if so desired, it may be allowed to deposit its anthracene, which it does, for the most part, upon cooling, and its naphthalene, upon further standing. The "naphtha vapours" carried over with the gas are removed in passing through the ordinary condensers.

From 1 ton of tar, 10,000–14,000 cub. ft. of gas and 6–7 cwt. of good hard coke, are produced.—E. R. B.

*Improvements in Gas Condensers.* S. Cutler, London. Eng. Pat. 11,734, June 14, 1893.

BENT, curved, or spiral tubes, rigidly connected to their end tube-plates, are employed; the expansion boxes, permitting of the varying contraction and expansion of the usually employed straight tubes, being no longer necessary.

The water enters the tubes from a common chamber, one of whose sides is the lower tube-plate (in the vertical form of condenser), and ascending them, is discharged into an upper chamber, the bottom of which is the upper tube-plate; the outlet from this chamber is such that an equal pressure is maintained in all the tubes. Sometimes the upper tube-plate carries a descending flange immersed in a water lute, the latter attached near the top of the walls of the condenser.

A further improvement is the employment of radiating plates attached, in either horizontal or inclined position, to the outside of the tubes; thereby greatly increasing the internal (cooling) surface of the condenser.—E. R. B.

*Improvements in and relating to Electric Furnaces.* H. H. Lake, London. From A. C. Girard and E. A. G. Street, Paris. Eng. Pat. 13,340, July 8, 1893.

WHEN the material to be treated is solid and a conductor of electricity, it is fed at a regular rate, as a uniform rod, through a longitudinal hole in a block of refractory material. Transverse or radial holes meet this longitudinal one and carry carbons, between which and the travelling rod, the arc is formed which is to supply the heat required. The temperature attained can be regulated by the rate of travel of the rod.

Where the material is pulverulent, it is caused to pass through the furnace in a hollow tube of carbon. This tube may either carry the material with it or be itself stationary.

Arrangements are also described for collecting the material where it is intended to be melted by the temperature arrived at; for causing the action to take place in any atmosphere desired; and for causing the whole of the

material to be acted on uniformly either by rotation of itself or by rotation of the arc—one method involving the use of a magnetic field in which the arc will, by the electric action, rotate automatically without relative movement of the parts of the apparatus.—E. T.

*Process of and Apparatus for Utilising Producer-Gas for Calcining Limestone.* A. J. Boulton, London. From A. E. Hatch, Chicago, U.S.A. Eng. Pat. 1291, January 30, 1894.

See under VII., page 636.

*Improvements in the Mantles or Hoods for Incandescent Gas Burners.* L. K. Böhm, New York, and T. C. Crawford, New Brighton, U.S.A. Eng. Pat. 4732, March 6, 1894.

The new composition material consists of about 90 per cent. of magnesium oxide, about 10 per cent. of silicic acid, and about 1 to 2 per cent. of an alkali. The silicic acid may be partly replaced by calcium or magnesium phosphate. The most suitable alkali is potassium hydrate or carbonate. The silicic acid may be used as dry powdered  $\text{SiO}_2$ , but the authors get the best results by employing gelatinous silica. The gelatinous silicic acid is obtained by precipitating a solution of water-glass with hydrochloric acid, filtering, and washing. One part by weight of the moist precipitate is well mixed with 4 parts by weight of a saturated aqueous solution of sugar, and 1 part of magnesium oxide and a small quantity of alkali gradually added with constant rubbing. In this way a plastic mass is obtained which may be moulded into the form required for the mantle or hood, or spun into threads and the mantle woven. The hoods are then burned in a baking oven. In this way a mantle or hood is produced such that, whilst consisting mainly of free magnesium oxide, it is an efficient glow-body, contains a skeleton of a double potassium and magnesium silicate giving great stability and hardness.—L. T. T.

*Appliance for Breaking down Crust in Gas Producers.* T. G. Hall, Chicago, U.S.A. Eng. Pat. 6551A, March 28, 1894.

This appliance is a kind of stamper, and consists of a rod capable of an up-and-down motion in the vertical axis of the producer, and carrying at its lower end a heavy head, while at its upper end it is attached to the following mechanism, viz.:—a flexible connection which can be wound round a drum, the shaft of which also carries a pulley and a ratchet wheel with dog; this latter can be released by the actuation of the arm of a lever placed across the path of the rising rod.

The stamper falls immediately after the upper end of its rod has thus struck the lever, unless caught by a pivoted latch lever. The latch of this last mechanism may be drawn back at any time by hand, or may be released automatically, after any arranged interval of time, by a further mechanism, worked also from the pulley shaft.—E. R. B.

the kerosene series mount by increments of 7 instead of by increments of 11, and they hold that the real atomic weight of carbon is 6, and not 12."

The material employed was Baku kerosene, of sp. gr. 0.825 at 15.5°; it was almost colourless, and had very little smell. Upon rapid distillation of two half-litre quantities it yielded, below 170° mil; from 170° to 250°, 68.8 cc.; from 250° to 300°, 20.5 cc.; the residue measuring 107 cc.

After "several months" of fractionation, full details of which operation, however, the authors do not give, 24 different liquids were obtained, besides 13 per cent. of dark-coloured residue, boiling above 290°, and of sp. gr. 0.880 at 15.5°; the two most volatile members being only present in very small quantity, while the remaining 22 were present in about equal quantities.

The authors give the following:—

TABLEAU STATEMENT OF THE CONSTITUENTS OF KEROSENE.

No.	Boiling Point.	Vapour Density.		Formula.
		Found.	Theory.	
1	77.0	..	..	..
2	87.0	..	..	..
3	93.5	3.60	3.63	$\text{C}_{15}\text{H}_{32}$
4	106.0	3.94	3.87	$\text{C}_{16}\text{H}_{34}$
5	116.5	4.08	4.11	$\text{C}_{17}\text{H}_{36}$
6	127.0	4.36	4.35	$\text{C}_{18}\text{H}_{38}$
7	138.0	4.59	4.59	$\text{C}_{19}\text{H}_{40}$
8	148.0	4.84	4.84	$\text{C}_{20}\text{H}_{42}$
9	158.0	5.02	5.08	$\text{C}_{21}\text{H}_{44}$
10	168.0	5.20	5.32	$\text{C}_{22}\text{H}_{46}$
11	176.0	5.51	5.56	$\text{C}_{23}\text{H}_{48}$
12	186.0	5.77	5.80	$\text{C}_{24}\text{H}_{50}$
13	197.0	6.08	6.04	$\text{C}_{25}\text{H}_{52}$
14	207.0	..	6.28	$\text{C}_{26}\text{H}_{54}$
15	214.0	6.53	6.52	$\text{C}_{27}\text{H}_{56}$
16	222.0	6.83	6.77	$\text{C}_{28}\text{H}_{58}$
17	230.0	..	..	..
18	237.6	..	..	..
19	246.0	..	..	..
20	253.0	..	..	..
21	260.0	..	..	..
22	267.0	..	..	..
23	274.0	..	..	..
24	280.0	..	..	..
25	Residue (about 13 per cent.)			..

Except as regards the few highest members of the series, each of the foregoing commences to boil at the temperature stated, and 50 per cent. distils without a greater rise of temperature than 2° or 3°, 75 per cent. within 5° or 6° of the initial boiling point, and 90 per cent. before the boiling point of the next fraction is reached.

The authors state that they have "exhaustively treated" only the five fractions, Nos. 3 to 7 inclusive, but that they have been unable to resolve them into fractions with different boiling points; yet the "final testing" of fraction

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*The Method of Fractional Distillation illustrated by the Investigation of Kerosene.* J. A. Wanklyn and W. J. Cooper. Phil. Mag. May 1894.

As result of this investigation, the authors "believe that the conclusion cannot be resisted that the hydrocarbons of

No. 3 (which is the only fraction treated at any length) is stated as follows:—

Experiment I.	Experiment II.	Experiment III.			
136 cc. of No. 3	132 cc. of No. 3	130 cc. of No. 3.			
yielded aggregate distillates up to the temperature stated.					
Temperature.	Distillate.	Temperature.	Distillate.	Temperature.	Distillate.
°C.	Cc.	°C.	Cc.	°C.	Cc.
136.2	5	96	5	95.8	5
157.2	50	97	50	96.8	50
198.0	75	99	100	98.8	100
259.2	100	105	Retort dry	104.0	Retort dry
105.0	Retort dry				

Fractions Nos. 1 and 2 were not obtainable in sufficient quantity for further work, and the remaining 17 fractions do not appear to have been investigated more fully.

—E. R. B.

*Examination of the Waste Liquors from the Refining of Petroleum.* R. Zdzioleki. Dingl. polyt. Journ. **290**, 258.

The waste liquor used for this examination was obtained on treatment of a mixture of crude Galician oils with caustic soda of 5 lb., subsequent to a previous treatment with sulphuric acid. After several weeks standing three separate layers had formed in the liquor. The first of these layers, constituting almost one-half of the total volume, consisted of yellow-coloured petroleum, which was siphoned off. The brown lower layer contained crystals of sulphite and sulphate of soda. These two layers were separated by a third consisting of a viscous emulsion of the oil with the brown solution. After running off the latter, direct steam was passed into this emulsion, which thereupon separated into an aqueous solution with a supernatant oily liquid. After removing the latter the two aqueous solutions were mixed and freed from mechanical impurities by filtration. On acidulating the mixed solutions, and heating, a dark brown oily liquid possessing an acid reaction separated out, the neutralised liquor at the same time becoming almost colourless. The dark oil thus obtained dissolves completely in water, although the solutions may not be quite clear. It saponifies with caustic alkalis, but these soaps also do not form clear solutions with water. This is probably due to the oil containing some unaltered hydrocarbons. The specific gravity of this oil is 0.9850 at 20° C. On heating it in a sealed tube for some hours with water at 160° C. an oil is obtained, the specific gravity of which was found to be 0.924 at 20° C., whilst the water contained a large quantity of sulphuric acid, and it also still contained some unaltered oil in solution. By saponification, and extraction of the soap solution with ether, these oils were separated into a saponifiable and an unsaponifiable fraction. The saponifiable fraction evidently consists of sulphonic acids. The unsaponifiable dark and viscid oil began to boil at 230° C., not without decomposition and evolution of sulphuretted hydrogen. Part of the distillate was still found to consist of saponifiable oils of the nature of petroleum acids. The specific gravity of the distillate was found to be 0.855 (20° C.), as against 0.868 of the undistilled oil. Part of this distillate consists of dodecyl alcohol,  $C_{12}H_{25}OH$ , besides which a hydrocarbon of the series  $C_nH_{2n}$  was observed. The above-mentioned saponifiable fraction consists chiefly of petroleum acids and sulphonic acids.—C. O. W.

*Campholene.* Guerbet. Comptes rend. **118**, 286.

The author is engaged in investigating this hydrocarbon and now points out its relation to the naphthenes of Baku petroleum.

He has obtained by heating campholene ( $C_9H_{16}$ ) in sealed tubes with hydriodic acid to 280° C., a hydrocarbon of sp. gr. 0.783 and boiling-point 132°–134° C., with the formula  $C_9H_{16}$ , which he considers to be identical with hexahydropsendocumene as extracted from Baku petroleum (see Markownikow and Oglobine, Jour. Russ. Chem. Soc. **15**, 331), while campholene itself he regards as tetrahydropsendocumene. The new hydrocarbon has all the characteristics of the benzene hexahydrides, for it does not fix bromine and is not attacked by either fuming sulphuric or nitric acids in the cold: bodies which are apparently however trinitropsendocumene and tribromopsendocumene have been obtained from it by methods described.

When campholene is repeatedly treated with fuming sulphuric acid there is obtainable, by rectification, from the supernatant hydrocarbon layer, hexahydropsendocumene identical with that described above, whilst from the acid layer is obtainable dicampholene ( $C_9H_{16}$ )<sub>2</sub>, an oily hydrocarbon boiling at 165°–168° C. with a pressure of 30 mm. of mercury and (with partial decomposition) at ordinary pressures at 266°–270° C. This dicampholene has an odour like terebenthene, and oxidises somewhat readily in the air, becoming discoloured and acquiring a resinous character. Campholene and the tetrahydrotoluene of rosin oil (Renard, Ann. Chim. Phys., 6th Series, **1**, 231, and Maquenne, *idem*, 6th Series, **28**, 279) are thus similarly affected by sulphuric acid.

It will be borne in mind that according to Markownikow (J. Russ. Chem. Soc. **22**, 275–279) the naphthenes proper are to be considered as identical with hexamethylene (hexahydrobenzene) and its homologues, and that non-napthene is probably hexahydropsendocumene.—E. R. B.

*Liquid Fuel.* G. Stockfleth. J. Soc. Arts. **42**, 616–625.

See under II., page 625.

PATENTS.

*Improvements in and relating to the Treatment of Vegetable Substances and the like for Preserving and Vulcanising the same, and for Obtaining Products of Distillation therefrom.* F. M. Grumbacher, New York. Eng. Pat. 10,979, June 5, 1893.

In wood-preserving and hardening processes where high temperatures and high air pressures are used, the fibre sometimes becomes partially carbonised. The patentee proposes to replace the air in the air-tight receptacles by carbonic acid at pressures varying from normal to 10 atmospheres. He finds that then carbonisation is effectually prevented, a higher temperature may be safely employed, and better results obtained.—L. T. T.

*Improvements in the Treatment of Waste from Gas Liquor to obtain Useful Products therefrom.* H. W. Crowther, West Bromwich, Stafford. Eng. Pat. 11,964, June 17, 1893.

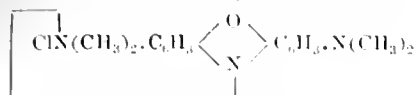
See under VII., page 635.

IV.—COLOURING MATTERS AND DYES.

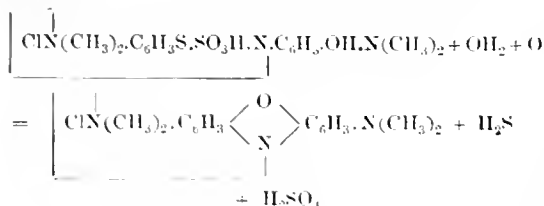
*On some Derivatives of the Oxazine and Euxhodine Series.* C. Lauth. Comptes rend. 1894, **118**, 473–476.

By substituting di-ethyl or di-methylmetamidophenol for dimethylaniline in the preparation of methylene blue by oxidation of amido dimethylaniline thiosulphonate in presence of dimethylaniline, the author sought to produce

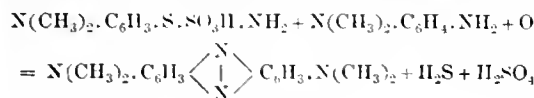
a hydroxylated methylene blue, or, failing this, to ascertain the relative stability of the groups  $\text{S} \cdot \text{SO}_3\text{H}$  and  $\text{OH}$ . Hydrochloride of nitrosodimethylaniline, reduced with zinc and hydrochloric acid, was largely diluted with water, treated with sodium thiosulphate and bicarbonate, and soda crystals, and, after the addition of diethyl-amido-phenol dissolved in acetic acid, was oxidised with bichromate. The liquid was then strongly blue, and on boiling threw down a precipitate, which was filtered, washed, and purified by boiling with very dilute hydrochloric acid, precipitating with sodium chloride, again precipitating by sodium and zinc chlorides, this time from solution in very dilute boiling sodium carbonate, and finally recovering the pure solid by evaporation from alcoholic solution. This substance contains no sulphur, and cannot therefore be a thionine; it is an oxazine having the formulæ—



Its distinguishing characteristics are as follows:—It has a blue-violet colour, and dyes silk and wool, or cotton prepared with tannin, that shade. It dissolves in water or alcohol, and to a slight extent in sodium carbonate solution, with a red-violet colour; it is more soluble in ammonia, from which solution ether fails to extract an appreciable quantity. It dissolves in strong sulphuric acid, giving a very red-violet, in strong hydrochloric acid an olive green, and in dilute acids a pure blue solution. A simple mixture of amido dimethylaniline and diethyl-amido-phenol does not yield this body, but produces a blue substance in the cold, which is apparently an indamine, being soluble in alkalis, altered by acids, destroyed by boiling with water (yielding a red substance), and converted into safranine by boiling with aniline acetate in presence of bichromate. It has not yet been transformed into an oxazine, but its formation throws light on that of the oxazine. Oxidation of a mixture of mercaptan and diethyl-amido-phenol produces a thiosulphated indamine, which, on boiling with water in the presence of an oxidising agent reacts according to the equation—



In support of his views as to the constitution of the believed oxazine, the author instances the production of a tetramethylated curholine, according to the equation—



by the oxidation of metanido-dimethylaniline in the presence of paranido-dimethylaniline thiosulphonate. After half an hour's boiling, this solution being extracted with very dilute soda, precipitated by sodium and zinc chlorides, yields a violet-red eurhodine, becoming blue in solution with hydrochloric, and green with sulphuric acid, and returning to its original tint on dilution, which tint is unaltered by ammonia.

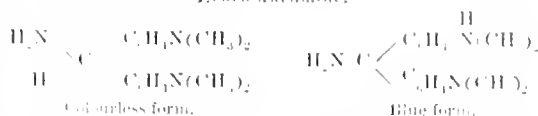
—W. G. M.

*The Blue Coloration produced by Leuco-auramine in Contact with Acids.* A. Rosenstiehl. Comptes rend. 118, 741—743.

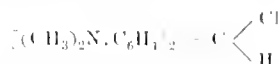
In a recent paper (Bull. Soc. Chim. 11—12, 213) the author has expressed his views as to the cause of colour in derivatives of triphenylcarbinol (this Journal, 1893, 748, and 1894, 382). He supposes that the molecule contains

two radicals of opposite chemical functions, one of which is attached to the central carbon atom and the other in the para position in two of the three phenyl groups. Most cases can be explained on this hypothesis, an exception, however, being the blue colouring matter from leuco-auramine described by Gräbe (Ber. 20, 3261; this Journal, 1888, 30), and to which he gave the following formulæ:—

*Leuco-auramine.*



The author observes that tetramethylbiamido benzhydrol behaves in the same way towards acids that leuco-auramine does, and is of opinion that leuco-auramine does not exist in the blue form, but that the body produced by the action of acids on it is the chlorhydride of tetramethylbiamido-benzhydrol—



which has a blue colour, and when represented by this formula also accords with the hypothesis he has put forward.

—T. A. L.

*The Carboxylic Acids of Dimethylaniline.* C. Lauth. Bull. Soc. Ind. Mulhouse, 1894, 24—35.

THE author has investigated the influence of the carboxyl group in the *o*-, *m*-, and *p*-positions in dimethylaniline in certain reactions, and gives the preparation of the first of these compounds (dimethyl-anthranilic acid), as it does not appear to have been hitherto prepared. It is obtained by heating for three hours on the water-bath, 10 grms. of *o*-amido-benzoic acid, 7 cc. of caustic soda solution of 36 B., 24 grms. of methyl iodide, and 30 cc. of 95 per cent. alcohol. The crystals, which separate on evaporating the alcohol, are purified by recrystallisation from water. That the substance has the constitution  $\text{C}_6\text{H}_3\text{N}(\text{CH}_3)_2\text{CO}_2\text{H}$  is proved by treating it with alcoholic soda and reprecipitating with an acid, when it remains unaltered. An attempt was made to prepare the body by heating the hydrochloride of *o*-amido-benzoic acid with methylic alcohol, but  $\text{CO}_2$  was given off. Another method attempted was, heating salicylic acid with dimethylaniline, but in this case also  $\text{CO}_2$  was evolved, and in a similar manner the oxidation of dimethyl-*o*-toluidine with potassium permanganate did not yield the substance required. The mother-liquor, obtained in the method given above, when evaporated, gives crystals of a quaternary compound, which can also be obtained directly by the action of 3 molecular proportions of methyl iodide on equimolecular proportions of anthranilic acid and soda. *Dimethyl-*o*-amidobenzoic acid* crystallises in long colourless prismatic needles, melting at 175° C. after sublimation, but if crystallised from water or benzene it melts at 179° C. It dissolves in 250 parts of boiling and 500 parts of cold water, and is very soluble in alcohol, ether, and hot benzene. All these solutions have a blue fluorescence. It sublimes in beautiful needles, but undergoes partial decomposition into dimethylaniline and a violet-coloured substance, and gives dimethylaniline when heated with lime. It gives salts with acids and bases, behaving like an amido acid, but a nitroso compound has not been obtained. The colours obtained from it by combining it with diazo compounds are yellow, orange-red, and brown, but are of no particular importance. Attempts to obtain a closed chain compound of the indigo type by means of dehydrating agents, either with or without oxidising agents, were unsuccessful. By the action of oxidising agents such as bleaching powder, bichromate, or ferric chloride, greenish or violet precipitates are obtained. Lead peroxide in presence of acetic acid gives a greenish-blue substance insoluble in water and alcohol, and decolorised by alkalis. The decolorised liquor on fresh treatment with  $\text{PbO}_2$  in presence of acetic acid reproduces the greenish-blue substance. The author refers

to a similar reaction he described with the oxidation product of tetramethyl-benzidine, and he believes that a benzidine derivative is formed in this case also. With chloride of copper, or preferably chloranil, a beautiful violet-red colouring matter is obtained from the dimethyl-*o*-amido-benzoic acid. One part of the acid is heated with  $1\frac{1}{2}$  to 2 times its weight of chloranil to 100–110° C. The mass gives off water, and after the reaction is complete the melt is extracted with benzene and the residue dissolved in sodium carbonate and precipitated with sulphuric acid. It is further purified by extracting with alcohol, and after evaporation, the residue is dissolved afresh in sodium carbonate and precipitated with salt. The final purification consists in dissolving it in sodium carbonate, precipitating with sulphuric acid, drying, and washing with boiling ether. The compound thus obtained being 60 per cent. of the weight of the acid taken, is a greenish-bronze coloured substance, insoluble in water, soluble in alcohol, and having in general the properties of the triphenyl-methane colouring matters. Its alcoholic solution is decolorised by zinc and acetic acid, and the leuco-compound obtained regenerates the colouring matter on oxidation with  $\text{PbO}_2$ . It differs, however, from the triphenylmethane colouring matters hitherto known, as it is soluble in alkalis and reprecipitated by acids. According to the analysis, which gave numbers corresponding to  $\text{C}_{22}\text{H}_{20}\text{N}_2\text{O}_2$ , or to  $\text{C}_{20}\text{H}_{18}\text{N}_2\text{O}_2$ , it may be regarded as penta- or tetra-methyl rosaniline tricarboxylic acid or as a mixture of the tetra-, penta-, and hexa-methyl derivatives. As regards its dyeing properties, it has an affinity for metallic mordants, especially chromium oxide, on cotton, and also dyes wool and cotton mordanted with tannin like a basic dyestuff, and its alcoholic solution does not dye silk excepting in the presence of acetic acid. The shades obtained in spite of the four or five methyl groups are those of a partially alkylated rosaniline, the effect of the three carboxylic groups being to considerably reduce the product. Attempts to prepare the same substance by the action of carbon tetrachloride in presence of aluminium chloride were unsuccessful, and this was also the case with benzylidene chloride. By heating the substance with acetic anhydride in presence of sodium acetate it is decolorised, but the colour reappears on boiling with hydrochloric acid. The dimethylantranilic acid will also yield the leuco-compounds of colouring matters on condensation with tetramethyldiaminobenzhydrol (benzhydrol is diphenyl carbinol) and with benzaldehyde, which on oxidation give the colouring matters themselves, that from the former giving a violet and from the latter a blue. Meta-nitrobenzaldehyde gives a rather greenish blue. All these colouring matters dye wool, silk, and cotton mordanted with tannin, and by virtue of their carboxyl groups will also combine with metallic mordants. Condensation products have also been obtained with formaldehyde in presence of sulphuric acid, but no colouring matters have been obtained therefrom. As mentioned above, the mother-liquor from the preparation of the dimethyl-*o*-amidobenzoic acid deposited crystals on standing. These consisted of the sodium salt of the methyl iodide of the above acid, and have the formula  $\text{C}_{10}\text{H}_4\text{N}(\text{CH}_3)_3\text{I} \cdot \text{CO}_2\text{Na}$ . The substance is very easily soluble in water, dissolves in 2.8 parts of alcohol, and fuses below 100° C. On decomposing with hydrochloric acid it yields the corresponding acid, which separates in long crystals melting at 175° C., and is very soluble in water, alcohol, and acetone, but insoluble in benzene and ether. When decomposed with silver oxide it yields a compound which has all the properties of an ammonium base, and when the base is heated to about 200° C. it decomposes into carbonic acid, methyl alcohol, and dimethylaniline. It gives a characteristic orange-red colour with lead peroxide in presence of acetic acid, and when heated with chloranil it yields a violet which appears to be identical with the carboxylated tetramethyl violet described above. The author has also investigated the behaviour of *p*- and *m*-dimethylamido-benzoic acid towards oxidising agents, and finds that neither gives colouring matters, and although he obtained condensation products from the *p*-acid with benzhydrol (diphenyl carbinol) and benzaldehyde, he showed that the substances

produced were methyl violet and malachite green, that is to say, that the carboxyl group had been eliminated. In the case of the *m*-acid no colouring matters were obtained on oxidation, and the condensation product with benzhydrol only gave a very small quantity of hexamethyl violet, which probably was due to a decomposition of the hydrol itself. In addition to the three acids mentioned, the author has also investigated the dimethylamidosalicylic acid, corresponding to the 1,2,5-nitro-salicylic acid melting at 226–228° C., as this acid ought to give valuable mordant colouring matters. Attempts to methylate the amido acid directly, proved unsuccessful, and he only succeeded by Griess' method of preparing the periodide of the betan of salicylic acid, converting this into the dimethyl ether, and then saponifying the product. He obtained in very small yield, a crystallised acid melting at 226° C., which gave no coloration on oxidation, nor was it possible to condense it with benzaldehyde. Benzhydrol gave a very blue violet, which, however, would not dye on metallic mordants. The acids described in the paper give the following characteristic tests when their solutions are treated with lead peroxide in presence of acetic acid. *o*-Dimethylamidobenzoic acid gives a dark greenish-blue precipitate, the *m* acid gives a fugitive dark reddish-brown coloration, the *p*-acid a brown coloration changing to violet, and the dimethylamidosalicylic acid gives a reddish-orange coloration changing to brown. The following conclusions are deduced from the results:—

1. The colouring matters obtained from dimethylaniline containing a carboxyl group in the ortho position, have very different chemical and tinctorial properties from those obtained from dimethylaniline.

2. The position of the carboxyl group determines the course of the reaction: if it is in the ortho position, carboxylated colouring matters are obtained; if in the para position, the carboxyl group is eliminated, and the colouring matters produced are identical with those obtained from dimethylaniline; whilst if the carboxyl group is meta to the dimethylamido group, the property of forming colouring matters will have almost entirely disappeared.—F. A. L.

*The Titration of Aniline and other Organic Bases with Methyl Orange.* G. Lange. Chem. Ind. 16, 490.

See under XXIII., page 667.

## PATENTS.

*The Manufacture and Production and Application of New Colouring Matters or Dyes from Naphthalene.* J. Y. Johnson, London. From the "Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 10,996, June 5, 1893.

When naphthalene is converted into its dinitro-compound, a mixture is obtained which consists of the 1.1' and 1.4' derivatives. Of these, only the 1.4' has hitherto been employed in the manufacture of naphthazarine (Eng. Pat. 7833 of 1887; this Journal 1888, 431), and for the preparation of 1.4' naphthylene diamine, whilst the 1.1' derivative has been a waste product. According to the present specification, the 1.1' dinitronaphthalene can be converted into valuable black colouring matters for cotton by the action of alkaline reducing agents. Three products are described, and designated A, B, and C. A is prepared by reducing 1.1' dinitronaphthalene in a boiling alkaline solution with grape sugar, when a violet precipitate having a metallic lustre separates out. It gives black to violet-black shades, which are best obtained by producing them on the fibre directly. B is formed by using a rather dilute solution of an alkaline sulphide, and is precipitated from this solution by the addition of an acid. It is insoluble in sodium carbonate solution, and is dyed on cotton, linen, jute, and the like by using a strong bath containing up to 25 per cent. of sodium carbonate, and adding grape sugar or a similar reagent. If a more concentrated solution of sodium sulphide be employed, there results the dye C, which differs from A and B in being soluble in sodium carbonate, and can be fixed



on cotton, linen, or jute by boiling in a strong bath of sodium carbonate as with B, but without the addition of grape sugar. The specification contains a table showing the behaviour of these dyes to different reagents.—T. A. L.

*Improvements relating to the Production of Colouring Matters.* A. Ashworth and J. Burger, Bury. Eng. Pat. 11,556, June 13, 1893.

THE action of sulphurous acid and its salts on nitroso- $\beta$ -naphthol produces an amidonaphthol sulphonic acid, but by the moderate action of bisulphites on the compound an intermediate product is obtained which is the subject matter of the present patent. One kilo. of nitroso- $\beta$ -naphthol, preferably as a paste, is mixed with 2 kilos. of sodium bisulphite solution of about 6% T., and heated to about 25° to 30° C. The melt is removed from the water-bath when the reaction commences, and the temperature rises spontaneously about 20°. The whole mass after some time solidifies to a paste of greyish-white crystals of the new compound. It is easily soluble in water and is decomposed by caustic soda regenerating nitroso- $\beta$ -naphthol. It can be used directly for printing and dyeing, giving green shades with iron and brown shades with chromium mordants. The new compound can also be condensed with hydroxy-carboxylic acids, such as gallic acid, or with tannin, giving condensation products which dye brown on chromium mordants and are said to be fast to light and soap. A further property of the new compound is that it will combine with diazo compounds, the following example being given. The diazo compound from 14.3 kilos. of  $\alpha$ - or  $\beta$ -naphthylamine is combined with 25.9 kilos. of the new compound dissolved in sodium carbonate. The mixture is allowed to stand and the colouring matter salted out, filter-pressed, and dried. The new azo compounds form brown or reddish-brown powders, are easily soluble in water, and dye brown to reddish-brown shades on chromium mordants.—T. A. L.

*The Manufacture or Production of Dyestuffs.* II. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 11,892, June 16, 1893.

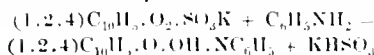
THIS is an extension of Eng. Pat. 8898 of 1893 (this Journal, 1894, 511), and refers to a more general method for producing the colouring matters there described. The method employed in the patent referred to for the production of basic azine dyestuffs or their sulphonic acids from the symmetrical or mixed dialkyl 1:3-naphthylene diamines or their sulphonic acids, was by condensing them with the nitroso derivatives of secondary or tertiary aromatic amines or amidosulphonic acids. These latter, it has now been found, can be replaced by compounds which act in a similar manner, as, for instance, quinone dichloroimide or azo compounds from diazo derivatives combined with primary, secondary, or tertiary amines or their derivatives. The same or similar dyestuffs can also be obtained by oxidising the alkylated 1:3-naphthylene diamines together with alkylated  $p$ -diamines or their sulphonic acids. These results are modified according to the nature of the solvent or diluent employed. Thus, for instance, a different product is obtained in a condensation as described above if a neutral or basic solvent is used, to that formed if an acid be employed. In the latter case, the amine liberated from the azo compounds combines with the acid, forming a salt which is incapable of acting further on the resulting dyestuff. As an example, if azo-benzene and di- $p$ -tolyl-1:3-naphthylene diamine are heated in glacial acetic acid solution and the product is subsequently sulphonated, a dyestuff of yellower shade is obtained than that produced when phenol is used as a solvent. All the dyestuffs produced according to this invention which do not contain sulphonic acid groups and are insoluble or sparingly soluble, may be sulphonated in the usual manner. The colouring matters obtained are principally blues and violets, and dye cotton mordanted with tannin.—T. A. L.

*Production of Azo Colours.* T. R. Shillito, London. From J. R. Geigy, Basle, Switzerland. Eng. Pat. 11,902, June 16, 1893.

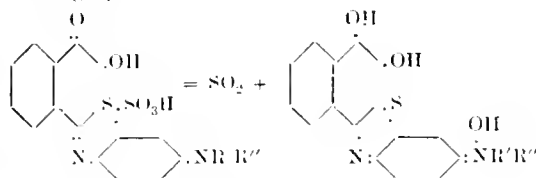
THIS is an extension of Eng. Pat. 15,692 of 1892 (this Journal, 1893, 755), and describes the preparation of colouring matters by combining the diazo compounds of  $\alpha$ - $p$ -amido-phenol disulphonic acid or of  $p$ -amido-phenol sulphonic acid with pyrogallie acid, the following quantities being employed:—30 kilos. of  $\alpha$ - $p$ -amido-phenol disulphonic acid are dissolved in 300 litres of water mixed with 50 kilos. of ice and diazotised by adding 15 kilos. of hydrochloric acid of 21° B. and 7 kilos. of sodium nitrite. The diazo compound is precipitated by adding 50 kilos. of salt and is mixed with 50 kilos. of ice. A solution of 15 kilos. of pyrogallie acid in 20 litres of water is then added, and immediately afterwards a solution of 18 kilos. of sodium carbonate in 60 litres of water. After 12 hours, 50 kilos. of 40 per cent. acetic acid are added, and the paste thus produced may be used directly for printing, or the colouring matter may be filtered off and dried.—T. A. L.

*Improvements in the Production of Colouring Matters Dyeing with Mordants.* W. E. Heys, Manchester. From Sandoz and Co., Bâle, Switzerland. Eng. Pat. 3886, February 23, 1894.

WHEN  $\beta$ -naphthoquinone 4-sulphonic acid reacts with an aromatic compound containing a free amido group, the sulphonic acid group is eliminated. For instance, with aniline and 1:2-naphthoquinone 4 potassium sulphonate the following reaction takes place—



and a bright vermilion precipitate of  $\beta$ -hydroxy- $\alpha$ -naphthoquinone is formed. The same reaction takes place with  $p$ -diamines, derivatives of indophenol being formed. Thus  $p$ -amido-dimethyl aniline yields  $\beta$ -hydroxy-indophenol as a blackish-violet precipitate insoluble in water. By using the thiosulphonic acids of the asymmetrically substituted  $p$ -diamines or  $p$ -diamine sulphonic acids, thiosulphonic acids of the  $\beta$ -hydroxy-indophenols are formed, which are easily converted into thiazine colouring matters according to the following equation:—



THIS conversion can be performed by boiling the aqueous solutions of the hydroxy- and sulpho hydroxy-indophenol thiosulphonic acids with zinc chloride. By printing these acids in presence of chromium mordants on cotton fibre and subsequently steaming under pressure, the thiazine colouring matter is developed and converted at once into its insoluble chromium lake, which is fast to washing, acids, and alkalis. The thiazine colouring matters themselves are obtained from the indophenol thiosulphonic acids by boiling their aqueous solutions with caustic alkalis or by adding them to concentrated monohydrated or fuming sulphuric acid. The first method is used preferably when sulphonic derivatives of the hydroxy-indophenol thiosulphonic acids are employed, whilst the second method is more applicable to the non-sulphonated derivatives since after the conversion into the thiazine the product can be sulphonated by adding sulphuric acid containing a higher percentage of anhydride. The conversion in both cases takes place at 50° C., and the sulphonation at 70°—80° C. until a sample dissolves in sodium acetate solution with a pure blue colour. As an example of the method employed for developing the colour on the fibre, 30 kilos. of hydroxy-indophenol sodium thiosulphonate (prepared by mixing aqueous solutions of 1:2-naphthoquinone 4-potassium sulphonate and  $p$ -amido-dimethylaniline sodium thio-sulphonate) are mixed at 70°



C. with 300 litres of water and 340 kilos. of tragacanth gum thickening. After cooling, 30 kilos. of 40 per cent. acetic acid and 50 kilos. of chromium acetate of 20° B. are added and the whole sieved, when it is used for printing on cotton previously oiled. The fabric so treated is then steamed for  $1\frac{1}{2}$  to 2 hours at a pressure of  $\frac{1}{2}$  to 1 atmosphere. This produces an indigo blue, which is brightened by chalk and soaping. The sulpho-hydroxy-indophenol thiosulphonic acid from sulpho-*p*-amido-ethyl-benzylaniline thiosulphonic acid dissolved in 50 times its weight of water and boiled with a quantity of caustic soda solution of 40 per cent. equal to  $\frac{1}{10}$  of the volume of the water, is easily converted into the corresponding thiazine sulphonie acid, which can be separated from the liquor made acid first with hydrochloric and finally with acetic acid, in the form of its acid salt, by adding common salt. The following example shows the method employed when sulphuric acid is used for the conversion:—30 kilos. of hydroxy-indophenol thiosulphonic acid from *p*-amido-diethylaniline thiosulphonic acid are added to 90 kilos. of sulphuric acid monohydrate and heated to 30° C. for 2 hours, and subsequently for 2 hours more to 50° C. The conversion into the thiazine is then complete, and in order to sulphonate the product, 90 kilos. of 70 per cent. anhydride are added in the cold. The melt is then heated to 80° C., until the sulphonation is complete and the sulphonie acid is separated by pouring the melt into ice-cold water, when the precipitate is filtered off, converted into the sodium salt by boiling with sodium acetate, salted out, filter-pressed, and dried. It dyes unmordanted wool bluish-violet from an acid bath and gives blue and greenish-blue shades on chromium mordants fast to acids, alkalis, and light.—T. A. L.

*Manufacture of a New Dioxynaphthalene-mono-sulpho Acid and of Colouring Matters therefrom.* H. Imray, London. From "The Basle Chemical Works Bindschedler," Basle, Switzerland. Eng. Pat. 4629, March 5, 1894.

The substance referred to is obtained by fusing 50 kilos. of the sodium salt of hydroxynaphthoic disulphonic acid,  $(1,2,4,2')C_{10}H_4(OH).CO_2H.(SO_3H)_2$ , with four times its weight of caustic soda at 230°–290° C. for a few hours. The melt is then dissolved in water, acidulated with a mineral acid, neutralised with sodium carbonate, and filtered. The liquor thus obtained contains the new (1,7,4) dihydroxy-naphthalene sulphonie acid, and has a bluish-violet fluorescence. It can be used directly for the production of colouring matters by combining it with tetrazo compounds. For instance, a colouring matter which dyes unmordanted cotton a greenish-blue is obtained by diazotising 24 kilos. of dianisidine dissolved in 52 kilos. of hydrochloric acid and 250 litres of water with 14 kilos. of sodium nitrite in 50 litres of water. This solution is then poured into a solution containing 20 kilos. of sodium carbonate and 27 kilos. of the sodium salt of the new acid. After stirring for some time the intermediate compound is formed, and a second quantity of the solution of the new acid equal to the first is then added. When the combination is complete, the liquor is raised to 100° C., and the colouring matter is filtered off and dried. Other tetrazo compounds can, of course, be used, and the shades produced are reds, browns, violets, and blues.—T. A. L.

*Manufacture of a New Dioxynaphthoic-mono-sulpho Acid and of Colouring Matters therefrom.* H. Imray, London. From "The Basle Chemical Works Bindschedler," Basle, Switzerland. Eng. Pat. 4630, March 5, 1894.

By carrying out the fusion described in the preceding specification at a lower temperature, viz., at 180°–200° C., a new dihydroxy-naphthoic monosulphonie acid is obtained, which, after dissolving the melt in water and neutralising with a mineral acid, separates as a white precipitate. This compound, like the preceding one, will combine with tetrazo bodies, such as tetrazo-diphenyl and its homologues, the ethers of tetrazo-hydroxy-diphenyl, and of tetrazo-dihydroxy-diphenyl, tetrazo-stilbene, and tetrazo-azobenzene and its homologues. The tetrazo compound from 21 kilos. of

tolidine, 500 litres of water, 55 kilos. of concentrated hydrochloric acid, and 14 kilos. of sodium nitrite, is added to a solution of 35 kilos. of dihydroxy-naphthoic sulphonie acid and 20 kilos. of soda ash. When the formation of the intermediate compound is complete, a solution of 25 kilos. of the sodium salt of *a*-naphthol-*p*-sulphonie acid is run in and the whole stirred for 24 to 48 hours. The liquor is then boiled and the dyestuff salted out, filtered off, and dried. It dyes unmordanted cotton a violet-blue.—T. A. L.

*Manufacture of New Amido-Bases and of New Colouring Matters therefrom.* R. Koreff, Pele Tyrolka, Prague, Bohemia. Eng. Pat. 5143, March 12, 1894.

THE bases are obtained by condensing *o*-toluidine, *a*-naphthylamine, *o*-tolidine, or *o*-dianisidine with formaldehyde in presence of sulphuric acid. By this means the amido groups are left intact, and the new base, for instance, from tolidine, has the constitution—

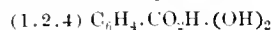


This substance is obtained by dissolving 42.4 kilos. of *o*-tolidine in 400 kilos. of sulphuric acid of 66° B., and adding 7.5 kilos. of 40 per cent. formaldehyde. The reaction is complete when the melt does not smell of formaldehyde and a sample dropped into water no longer gives a precipitate of the sparingly soluble tolidine sulphate. The melt is then poured into 2,500 litres of cold water and gives a clear yellow solution which, when tested with nitrite, shows that the amido groups are intact. Two isomeric bases are said to be formed in this reaction, one giving a sparingly soluble and the other an easily soluble sulphate. Both, however, are easily soluble in dilute sulphuric acid. On adding an alkali to the solution obtained above, the two bases can be fractionally precipitated. All the bases obtained in this way are easily diazotised, and can be combined with various components in the usual manner.

—T. A. L.

*Manufacture of Yellow and Orange Colouring Matters from  $\beta$ -resorcylic Acid.* F. Muhlert, Pele Tyrolka, Prague, Bohemia. Eng. Pat. 6004, March 22, 1894.

THE colouring matters are produced by combining diazotised *m*- or *p*-nitraniline with  $\beta$ -resorcylic acid—



in an acid solution. The dyestuffs are for use with chromed wool or cotton, and are said to give shades fast to light and washing. A solution of 15.4 kilos. of  $\beta$ -resorcylic acid and 13.6 kilos. of sodium acetate in 100 litres of water is poured into the diazo solution from 13.8 kilos. of *m*-nitraniline. The combination takes place slowly and the colouring matter precipitates in yellow flakes. It dyes a pure yellow on chromed wool and cotton and also on wool which has been mordanted with alum and tartar. The colour from *p*-nitraniline gives a more orange shade.

—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

### PATENTS.

*Improvements in the Extraction and Preparation of the Fibres of Rhea and other Grasses and Fibrous Vegetables.* H. C. Fellowes and W. R. Crozier, London, and H. Ferguson, Leytonstone. Eng. Pat. 6584, March 28, 1893.

This invention deals with the preparation of rhea, china-grass, and similar fibrous substances for the spinning and weaving processes, &c. The so-called "ribbons" of rhea are laid by hand lengthwise and evenly in a suitable open-work receptacle, which has the form of a shallow tray and

is provided with a number of parallel ridges. The fibrous material is held in place by means of an open-work cover. The material thus arranged is boiled for about three hours (the time varying, however, according to the condition of the fibres and the strength of the solution) in an alkaline solution, e.g., caustic soda, the proportion being from 90 to 168 lb. of NaHO to 5,000 gallons of water. The patenters regard it as an important feature of their invention that by this boiling process the material is prepared for the separation of the extraneous coatings from the fibres. This separation is effected by the immersion of the boiled material in a tank of warm water (of about 160° F.) for 2–6 hours. Then the fibres are rinsed in cold water, and finally treated for a short time with a cold mixture of vegetable oils, alkalis, and water in order to further improve the appearance of the material.—H. S.

*Improvements in Scouring, Degreasing, and Cleansing Wool, and in Apparatus therefor.* F. N. Turney, Nottingham. Eng. Pat. 8179, April 22, 1893.

THE greasy wool is placed in thin layers in perforated boxes which are introduced into an airtight cleansing apparatus which is provided with three chambers for the receiving, degreasing, and drying of the material. The degreasing chamber is provided with a number of shallow tanks arranged in different heights, and having sloping bottoms. These tanks contain a volatile scouring liquid, in which the boxes with the wool are dipped a number of times, clean solvent being supplied to the upper or last tank and passed from this to the next lower one in a continuous cycle, so that as the wool becomes cleaner it is washed in cleaner solvent. The wool thus readily parts with gritty matters, impurities, and fatty matters. The volatile solvent, when dirty, is distilled off, condensed (together with the vapours of the drying chamber), and re-used. Then the wool is dried in the drying chamber, and finally washed with warm water, or soap and water, in order to further purify the material and to improve its colour.—H. S.

## VI.—DYEING, CALICO PRINTING, PAPER-STAINING, AND BLEACHING.

*Dyeing with Glucosides and Rhamnosides.* E. Schunck and L. Marekewski, J. Soc. Dyers and Colourists, 1894, 95.

It is known that the tinctorial power of plant extracts is considerably increased by boiling with acids, this being due to a decomposition of the glucoside or rhamnoside present. When glucosides are directly employed in dyeing, a simultaneous hydrolysis of the glucoside has been assumed. The experiments of the authors confirm this assumption and prove conclusively that the mordant contained in a hank of cotton yarn is capable in most cases of bringing about a decomposition of the glucoside. The experiments were carried out with 1 per cent. solutions of ruberythric acid, ditisein, rutin, and quercitrin. In one series, cotton hanks mordanted with iron were dyed in the liquors at the boil; the other series were similarly boiled, but without insertion of the mordanted cloth. The spent liquors were then tested for sugar by two distinct methods. The liquors from the hanks dyed in rutin, quercitrin, and ditisein showed the presence of free sugar; that from the ruberythric acid showed no sugar, and the hank was only tinged, not dyed. The liquors containing no hanks did not in any case show a formation of sugar, proving that boiling with water alone had effected no decomposition.—W. E. K.

*The Fading of Colours.* A. Dufton. J. Soc. Dyers and Colourists, 1894, 90.

DÉPIERRE and Clouet have shown that the rate of fading of colours under the influence of light is not simply a function of the wave length of the incident light, but depends on the

colour of the material exposed. Thus they have shown from experiments made with various coloured glasses that red and blue colours are most powerfully affected by light which has passed through yellow glass, whilst other shades fade most rapidly under blue glass. Theoretically it would be expected that it is the absorbed radiation which brings about the fading of a colour, since it is only the absorbed rays which can supply the energy requisite for the decomposition of the colour molecules, and that consequently each colour would be most rapidly affected by those rays for which it shows the strongest absorption, and the author's experiments confirm this view. Thus, a pattern dyed with picric acid is protected as efficiently by a gelatin plate dyed with picric acid as it is by a black opaque screen. On the other hand, wool dyed Victoria blue or cotton dyed diamine sky-blue and exposed under a screen dyed a very full shade of orange G (which absorbs the whole of the more refrangible rays), fades almost as quickly as if uncovered, the screen still transmitting these rays which the blues are capable of absorbing.

In general, patterns exposed under screens dyed with the same colour are not so perfectly protected as is the case with a yellow like picric acid, as most colours are capable of absorbing light of all wave lengths (*i.e.*, they become black when sufficiently concentrated) and hence patterns dyed with such colours can only be perfectly protected by a black screen.

The author explains from these considerations the reason why a colour is relatively faster when dyed a dark than a light shade, the superficial or topmost layers of colour filtering off from the lower portions those rays to which the colour is most sensitive, and consequently the destruction of the colour goes on slowly layer by layer. Similarly in a mixed shade a fast colour protects a fugitive colour if the former absorbs those rays to which the latter are sensitive. The author explains the protective action of copper salts on certain colours as being due to the formation of a copper-lake and not to any filtering off of the actinic rays by the copper salt. He does not suppose the fading of colours in presence of light to be due to the formation of ozone or peroxide of hydrogen, but to the action of atmospheric oxygen under the influence of the absorbed rays.—W. E. K.

*Note on the Solution of Indian Gums by Means of Hydrogen Peroxide.* H. Koechlin. Bull. Soc. Ind. de Mulhouse, 1894, 36.

See under XVI., page 653.

*Basic Sulphate of Alumina.* W. C. Ferguson. J. Amer. Chem. Soc. 16 [3], 153.

See under VII., page 635.

## PATENTS.

*A Process for Dyeing Wool with Fast Colours.* C. D. Abel, London. From Farbwerke vorm. Meister, Lucius, and Brünig, Germany. Eng. Pat. 9371, May 10, 1893.

Wool dyed with azo-colours derived from  $\alpha$ -naphthol, are subsequently treated with chromic acid, by which treatment the shade is modified and its fastness very greatly increased.—W. E. K.

*Improvements in Vats for Dyeing Indigo Blues and the Like.* J. Grime, Bushy, N.B. Eng. Pat. 12,076, June 20, 1893.

THE arrangements and processes generally used in dyeing indigo are followed, but in addition each vat is provided with a smaller subsidiary vat into which, from time to time, the sediments or bottoms from the larger vat are pumped, and, after settling, the clear liquor is run back again into the main vat. It is claimed that in this way a practically clear working vat is obtained, which renders the process of dyeing very simple and regular.—W. E. K.

*Improved Process for Dyeing Textile Materials.* H. B. Lake, London. From E. Michaelis and Co., Cottbus, Germany. Eng. Pat. 15,983, August 5, 1893.

TEXTILE materials dyed indigo in the hyposulphite vat are, in lieu of the usual airing, passed into water containing acetic or other acid, or a salt having an acid reaction, in the presence of which the oxygen dissolved in the water oxidises the indigo-white to indigo-blue.—W. E. K.

*Improvements in the Method of Bleaching Cotton Cloths and other Materials, and in Apparatus employed therein.* G. A. J. Schott, Bradford. Eng. Pat. 15,531, August 16, 1893.

THE operations, which resemble generally those ordinarily followed in the bleaching of cotton goods, are carried out consecutively in a kier (the construction of which is described) without the removal of the goods therefrom.

—W. E. K.

## VII.—ACIDS, ALKALIS, AND SALTS.

*The Fusibility of Mixtures of Isomorphous Salts.* H. Le Chatelier. Comptes rend. 1894, **118**, 350—352.

THE author refers the present incomplete knowledge of the laws governing solution, in part to the influence of Proust's ideas, which has deterred chemists from studying phenomena that are not governed by the laws of definite proportion, and in part to the abnormal molecular constitution presented by the solvents (water, alcohol, and acetic acid) usually employed in laboratories. He proposes to approach the subject from the purely experimental side, and, leaving aqueous solution for the present, to study the mutual solvent action of salts, or the fusibility of mixtures of such bodies. Acting on the assumption that there is no real distinction between the solvent and the body dissolved, and that conglomeration and crystallisation are identical phenomena, he has fused mixtures of truly isomorphous salts, in which the ratio of the molecules of one component to the whole number in the mass is accurately known, and has noted the temperature at which crystallisation is first observed. These results are embodied in the following table, in which the ratio representing the number of molecules of the italicised constituent of any mixture to the whole number of molecules present is given in one line, and the crystallisation temperature of that mixture is stated immediately beneath it.

$K_2CO_3 + Na_2CO_3$ .....	0	0.20	0.38	0.55	0.65	0.79	1.0	..
	860°	770°	715°	690	700	740°	820°	..
$Na_2SO_4 + K_2SO_4$ .....	0	0.11	0.14	0.20	0.33	0.50	0.70	1.0
	860°	830°	825	815	830°	855°	940°	1,045°
$K_2CrO_4 + K_2SO_4$ .....	0	0.15	0.33	0.50	0.66	1.0	..	..
	940	950°	960°	935	1,000	1,045°	..	..
$Na_2SO_4 + Na_2CO_3$ .....	0	0.33	0.5	0.67	0.78	1.0	..	..
	860°	810°	800°	790°	795	320°	..	..
$K_2CO_3 + K_2SO_4$ .....	0	0.33	0.40	0.50	0.67	0.75	1.0	..
	860	880°	900°	920	960	980°	1,045	..
$NaCl + KCl$ .....	0	0.42	0.45	0.50	0.74	1.0	..	..
	780°	660°	650°	640	690	740	..	..
$KCl + KI$ .....	0	0.17	0.33	0.50	0.67	0.80	1.0	..
	640	610°	590°	580	630°	680°	740°	..

These combinations were chosen from bodies shown by Wyrouboff to be isomorphous, or believed to be so from chemical analogy. In graduating the pyrometer, the melting-point of gold was taken as 1,045°. The fusion was made in a platinum crucible, and the mass was stirred while cooling with the end of the thermo-electric couple.

—W. G. M.

*Action of Water on Bicalcium Phosphate.* A. Joly and E. Sorel. Comptes rend. **118**, 738—741.

THE authors have studied the action of boiling water on secondary calcium phosphate, determining the conditions under which the reversion to the more basic salt takes place, and preface their results by a short account of some similar decompositions. The primary or secondary phosphates other than the salts of the alkalis are, it is known, converted into more basic salts by the action of water. In the case of primary or secondary silver phosphate cold water causes a conversion into the tertiary salt. With the phosphates of the alkaline earths the primary are converted by cold water into secondary salts, but require to be heated to 100° C. or higher, in order to effect the conversion into the tertiary salts. The reaction can be still further complicated by a change of the dibasic salt into another of the same composition, but in a different state of

hydration. This is the case with crystallised bicalcium phosphate  $Ca_2H_2(PO_4)_2 \cdot H_2O$  which, when heated to 100° C. in presence of water can give either an amorphous trialcium salt or a crystallised anhydrous bicalcium salt, the extent of the change depending upon the amount of water present. The reaction can be followed step by step, and the alteration in the product determined by analysis and by microscopical examination in polarised light. When crystallised bicalcium phosphate is thrown into 100 times its weight of water the crystals rapidly devitrify, and the mother-liquor, which is acid to litmus paper and neutral to methyl orange, contains a primary phosphate. After boiling some minutes the ratio of lime to phosphoric acid in the amorphous product formed was 2.86, and at the end of 1½ hours, when the deposit was partly crystalline, the ratio fell to 2.5. On changing the water three times, the ratio became successively 2.57, 2.87, and 3.0. The final product is amorphous and dried at the ordinary temperature over sulphuric acid, has the composition  $Ca_3(PO_4)_2 \cdot \frac{1}{2}H_2O$ . By continuing to boil the amorphous product first formed without changing the water, a second reaction takes place between the liquor and the precipitate, and it is observed that small crystals are produced. This change takes place very slowly if the amount of salt present be less than 1 per cent., but more quickly if the weight is between 1 and 50 per cent. The boiling was maintained for three hours in all cases, and

with weights of salt 0.5, 0.65, and 0.8 per cent., the ratios  $\text{CaO}:\text{P}_2\text{O}_5$  were respectively 2.73, 2.74, and 2.79. Microscopical examination showed that these were all mixtures of an amorphous with a crystalline substance. With the following weights of salt, 1, 1.25, 1.5, 2.0, 2.5, 3.0, 4.0, 8.5, and 50 per cent. of the weight of the water, the ratios were 2.3, 2.22, 2.22, 2.18, 2.19, 2.13, 2.1, 2.09, and 2.09. Of these, with 1 per cent. the product was a mixture, whereas with 1.25 and 1.5 per cent. the crystallisation was complete, and the product appeared to be  $\text{Ca}_3(\text{PO}_4)_2 + 4\text{Ca}_2\text{H}_2(\text{PO}_4)_2 \cdot 2\text{H}_2\text{O}$ . In the more concentrated solutions the crystals consist of this substance mixed with anhydrous bicalcium salt. The authors arrive at the conclusion that the conversion of the hydrated bicalcium salt into the anhydrous bicalcium salt when heated in boiling water is due to a reaction which takes place in two stages, amorphous tricalcium phosphate being an intermediate product. The complete transformation is not always possible, and is only complete in an acid medium. The authors have never obtained a salt having exactly this composition except by precipitating a boiling solution of mono- and di-sodium phosphate with calcium chloride. Moreover, in order to obtain hydrated bicalcium phosphate it is necessary to work in an acid menstruum. The formation of this salt by the interaction of calcium chloride and disodium phosphate takes place in two stages. The first product is gelatinous tricalcium phosphate, a liquid, acid to litmus and methyl orange, which changes into crystallised bicalcium phosphate, a reaction which takes place the more quickly the more concentrated the solutions, but is never really complete unless the acidity of the menstruum be increased by the addition of phosphoric acid.—T. A. L.

*Basic Sulphate of Alumina.* W. C. Ferguson. J. Amer. Chem. Soc. 16, [3], 153—156.

MANY samples of commercial sulphate of alumina contain from a trace to 2.5 per cent. of alumina in excess of that required to form the normal sulphate. In the commercial analysis of such products it is usual to calculate the alumina necessary for the formation of the normal sulphate and return it as "combined," any alumina in excess of this being termed "free." The former is alone considered useful for sizing with resinate of alumina. This conventional view of the composition and function of sulphate of alumina containing excess of alumina appears to be irrational, as salts containing excess of base are to be expected from an oxide as feebly basic as is alumina, and, moreover, the "free" alumina may react with resinate of soda and yield resinate of alumina in the same way as the normal sulphate. Experiment confirms this view, inasmuch as even aluminate of soda in which the alumina is acting as a weak acid gives a precipitate of resinate of alumina on the addition of resinate of soda. A comparison was also made between a neutral sulphate of alumina containing 17 per cent. of alumina and a basic sulphate containing the same total percentage, including 2 per cent. of "free" alumina. When these samples were treated with an equal quantity of resinate of soda insufficient to precipitate all the alumina, and the precipitates filtered, washed with the same amount of water, and ignited, it was found that 4.40 grms. were obtained from the neutral sulphate and 6.98 grms. from the basic sulphate—a considerable advantage on the side of the latter. The author summarises the matter as follows:—Excess of alumina in commercial sulphate of alumina is not "free," but present as a basic sulphate. Such basic sulphate forms resinate of alumina more readily than does the neutral sulphate, and is on that account more economical. The method of stating analyses of sulphate of alumina should be changed so as to give its proper value to the alumina in excess of that required to form neutral sulphate.—B. B.

#### PATENTS.

*Improvements in the Production of Chlorine and Refined Lead, and in the Recovery of the Silver the Lead contains.* F. M. Lyte, London. Eng. Pat. 7264, April 8, 1893.

See under X., page 644.

*Improved Method and Apparatus for Collecting the Carbonic Acid generated during the Fermentation of Beer or Wine.* K. Mielch and M. Frank, Munich, Germany. Eng. Pat. 10,263, May 24, 1893.

See under XVII., page 655.

*Improvements in the Manufacture of Chlorine.* A. Brand, London. Eng. Pat. 11,040, June 6, 1893.

HYDROCHLORIC acid gas, obtained by dehydrating the aqueous acid by sulphuric acid, is passed through a series of interchangeable "decomposers" charged with a mixture of nitric and hydrochloric acids. The chlorine and nitrosyl chloride evolved are led through towers to be brought into contact with sulphuric acid, which becomes "nitrous vitriol," and the gas is then passed through water to withdraw hydrochloric acid. The chlorine thus obtained may be applied in the manufacture of bleaching powder or otherwise. The nitrous vitriol that accumulates is denitrated by any of the known methods. The water of the wash-tower eventually becomes aqueous hydrochloric acid, which is applied as described. The sulphuric acid used as a dehydrator is concentrated for re-use. In working the series of decomposers, when the nitric acid in the first vessel of the series is exhausted, the next vessel is made first, and so on, in order.—F. S.

*Improvements in the Treatment of the Waste from Gas-Liquor to obtain Useful Products therefrom.* H. W. Crowther, West Bromwich, Stafford. Eng. Pat. 11,964, June 17, 1893.

THE gas-liquor remaining after distillation from it of ammonia is treated with lime-kiln or other gases containing carbonic acid, the precipitated calcium carbonate is allowed to subside, and the cleared separated liquid is agitated with nearly sufficient precipitated cuprous oxide, mixed with water to a creamy consistence, to form a cuprous salt with the sulphocyanide present. The mixture is then slightly acidulated, preferably with hydrochloric acid. After agitation, subsidence, and removal of the supernatant liquor, the cuprous sulphocyanide is collected on a filter, washed, and used to obtain any desired sulphocyanide of an alkali or alkaline earth by treating it with such in the state of carbonate, oxide, or hydroxide, the latter preferably in the case of an alkali being used. The cuprous oxide reproduced is available for another operation.—E. S.

*Improvements in Evaporating Brine and other Liquors and Apparatus therefor.* F. W. Scott and F. W. Scott, jun., London, and E. G. Scott, Liverpool. Eng. Pat. 12,574, June 29, 1893.

See under I., page 622.

*Improvements in the Production of Cyanides.* J. Addie and J. Cunningham, Glasgow, and W. Macfarlane, Glengarnock. Eng. Pat. 12,572, June 27, 1893.

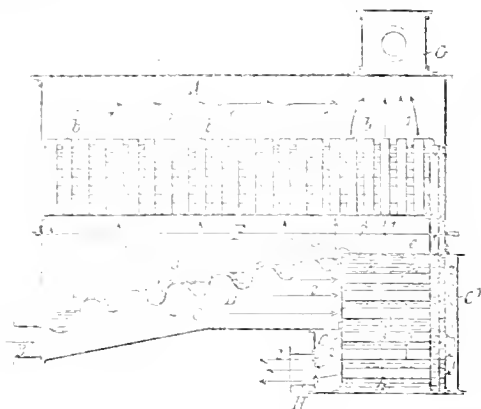
COKE, or other carbonaceous fuel, and an alkali, as pearl-ash, are charged into a blast-furnace, lined with dolomite, or other basic or neutral lining, and the hot blast is admitted. At a certain height in the furnace a horizontal pipe admits passage for the potassic or other cyanide blown forward into a collecting box, communicating with a series of vertical condensing pipes, opening into boxes at the bottom, in which boxes the cyanide accumulates, and is collected in a dry state. It is stated that a high temperature is attained in the furnace.—E. S.

*Improvements in and connected with Tanks for Electrical Decomposition of Chloride of Sodium or Potassium in Solution.* J. C. Richardson, London. Eng. Pat. 12,857, June 30, 1893.

See under XI., page 645.

*In Apparatus for Obtaining Carbonic Acid.* A. Müller, Nürtingen, Schwablen, Germany. Eng. Pat. 13,927, July 18, 1894.

The apparatus is constructed to effect the continuous production of carbonic acid from alkaline liquids, first, by subjecting them to a preliminary heating by the expelled carbonic acid, and then by subjecting them to the direct heat of gases of combustion. The upper portion of the apparatus consists of a rectangular casing connected to the lower section by means of an outer flange, and the entire apparatus, shown in longitudinal section, is made of wrought iron, except the pipe connections and overflow pipes, which are of cast iron.



The saturated liquor from an absorbing apparatus is pumped through pipes not shown in this figure, into the cooling compartment A, so as to reach to the upper edges of the over-flow pipes *d*, whence it flows to the bottom of the tubular boiler E, from which it overflows on to the corrugated plate D, and has outlet by a pipe shown in dotted lines, to be re-introduced into the absorbing apparatus after having first circulated through a cooler. When the apparatus is fully charged with liquid, gases of combustion or of roasting are passed in through the pipe B into the space C, from which they pass through the upper series of tubes *a*, as indicated by the arrows "2" into the space C', passing out into the space C through the lower series of tubes *a*, and having exit by the discharge-pipe H to a purifying apparatus. This passage of hot gases beneath the corrugated plate D raises the liquid upon it to boiling point, and the carbonic acid which begins to be expelled at 80° C., passes through the thin vertical tubes *b* and heats the surrounding liquid, which also evolves the gas, the exit for which is through opposite pipes in the dome G.—E. S.

*Improved Method and Apparatus for Making Vinegar and Maturing Spirits.* R. H. Leaker, Bristol. Eng. Pat. 15,475, August 15, 1893.

See under XVII., page 655.

*Improvements in the Manufacture of Cement from Chance or Le Blanc Alkali Waste or the like.* B. K. Rigby, Ditton. F. A. R. Neill, St. Helens, and A. C. Carr, Rainhill. Eng. Pat. 19,705, October 19, 1893.

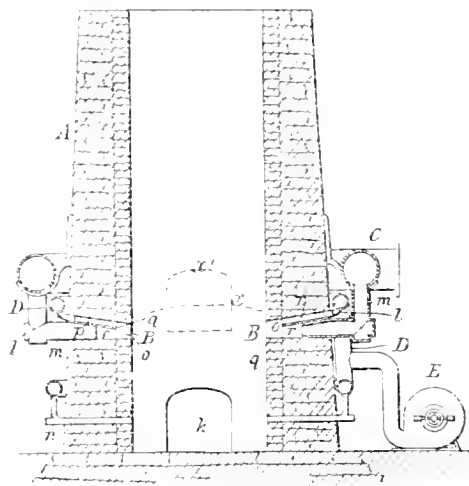
See under IX., page 638.

*Process of and Apparatus for Utilising Producer-Gas for Calcining Limestone.* A. J. Boulton, London. From A. F. Hatch, Chicago, U.S.A. Eng. Pat. 1991, January 30, 1894.

PRODUCER-GAS, if it can be caused to generate a sufficiently intense heat to effect "sticking" at the lower end of the column of limestone in the kiln, is a desirable form of fuel for calcining limestone.

To render producer-gas, which is inherently low in calorific power, suitable for this purpose it is introduced, according to this invention, simultaneously from opposite sides of the kiln, and at the same time air, under pressure, is admitted at points immediately above the gas-inlets. The air and gas, striking against the limestone, become thoroughly mixed and in burning produce the required high temperature.

The kiln A may be of any ordinary construction. At the desired height, where "sticking" is to be effected, are



openings B through the wall. These are divided by diaphragms *q* into gas-inlets *r* and air-inlets *p*. The diaphragms *q* stop short of the inner side of the wall, and so form mixing and combustion chambers *o*, in which the gas and air unite. It is claimed that the method of utilising producer-gas not only permits the proper "sticking" at the lower end of the limestone column to be effected, but also prevents "wall-climbing" by the products of combustion.

—R. B. P.

*A Bleaching Preparation or Mixture, applicable also for other purposes.* H. Wächter, Bielefeld, Germany. Eng. Pat. 4791, March 7, 1894.

THE bleaching preparation is obtained by boiling together stated proportions of soda, lime, zinc, tin, "or similar metals," chlorine solution, and an oil or fat, with water. "By the addition of zinc, tin, or similar metals, chlorine in solution, oil or other fats, the mixture becomes a soap-like mass and the water is eliminated, hydrogen being generated: by repeated stirring, the mixture is turned into a pulverulent mass, and may then be used for all materials without injuring them." This mixture boiled with much water is proposed as a substitute for petroleum for the preservation of boring and shaping machines. It is also stated that the mixture may be applied in purifying petroleum intended for use as a lighting or heating agent, and in purifying fibres in the manufacture of paper pulp.—E. S.

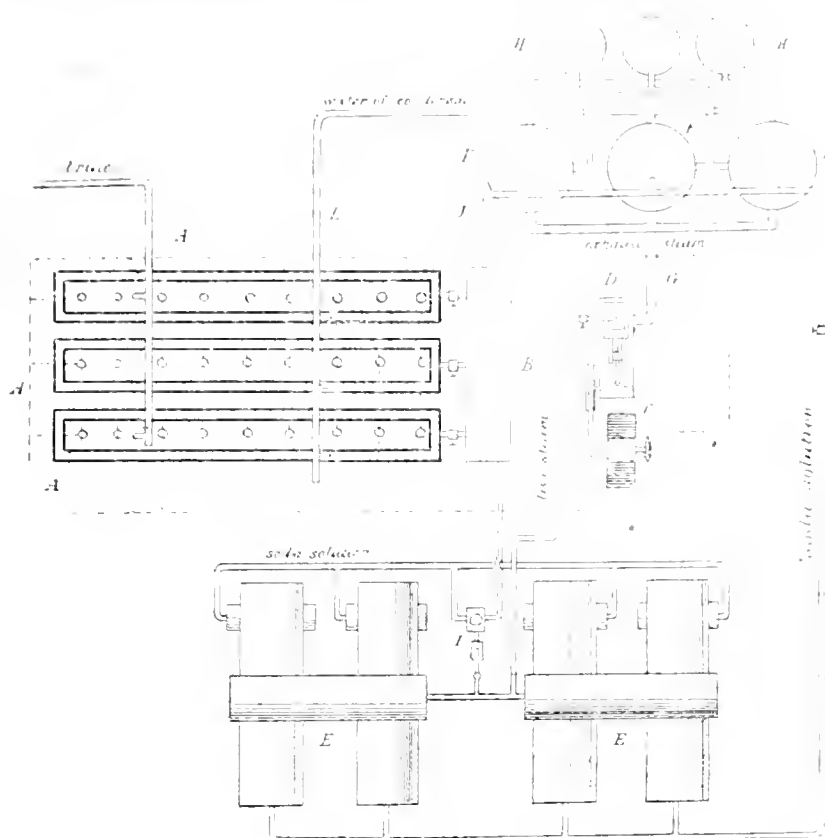
*Improvements in Producing Nitric Acid and Metals from Nitrates.* J. D. Darling and H. E. Forrest, Philadelphia, U.S.A. Eng. Pat. 5808, March 20, 1894.

See under XI., page 647.

*Improved Method and Apparatus for Manufacturing Caustic Soda.* T. Craney, Bay City, U.S.A. Eng. Pat. 6426, March 30, 1894.

CAUSTIC SODA is prepared from salt brine by electrolysis, the decomposition in the cells not being carried to its full limit, but only to the extent of producing a 1 to 2 per

cent. solution of caustic soda, which is drawn off, and its place supplied by water of condensation from the evaporators; while the salt brine on the anode side is made good to its full degree of strength.



In the above drawing, A is the series of electrolytic cells, B the receiver for the dilute caustic soda solution from the cathode cells, C the electric generators, D the steam engines, E the boilers, which are fed with weak soda solution, to be run, when about doubled in strength, into F, the evaporators or vacuum pans, from which the concentrated solution is discharged into H, the fusing kettles. G is the connections for conducting the exhaust steam from the engine to the evaporators; and L is a return pipe by which water of condensation is supplied to the cathode compartments of the cells. A feed pump, I, and a pipe, J, for carrying the surplus of the evaporating pans, are also shown. It is stated that the greatest economy is derived from a continuous operation of all the parts of the plant. No reference is made to the production of chlorine.—E. S.

#### PATENT.

*Improved Methods of treating the Vitreous Surfaces of Articles made of or from China, Earthenware, Glass, or the like Materials for hermetically joining same.*  
H. Doulton, Lambeth, and J. Slater, Burslem. Eng. Pat. 20,947, November 4, 1893.

THE vitreous surfaces of the article to which metallic connection has to be made, are coated with the preparation described in Eng. Pat. 12,939 of 1892 (this Journal, 1893, 763) consisting of one drachm of bichloride of platinum (which has been well washed to eliminate the free acid) one ounce of balsam of sulphur, and three ounces of spirit of turpentine, and fired in an enamelled kiln. "Metallising" can also be effected by the use of gold or silver oxide or platinum sponge. The coated parts can then be joined by soldering in the ordinary way. The process is found useful for joining sheets of glass, between which documents, &c. have been placed for preservation, so that both sides are visible; and also for constructing and sealing glass vessels.—B. B.

### VIII.—GLASS, POTTERY, AND ENAMELS.

*The Utilisation of Blast-Furnace Slag.* A. D. Elbers. Eng. and Mining J. 1894, 392.

See under X., page 643.

### IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*The Chemical and Physical Examination of Portland Cement.* T. B. Stillman. J. Amer. Chem. Soc. 16 [3], 161—173. (This Journal, 1894, 275.)

THE paper contains illustrations of various well-known testing machines and recapitulates the usual canons of

careful cement-testing. Particular stress is laid upon the increase in tensile strength caused by the use of considerable pressure in filling the moulds, as in German practice. Thus, cement gauged in America had a tensile strength of 700 lb. per square inch at 30 days, and the same cement tested at Berlin by Bohme gave 875 lb. per square inch at 28 days. The author advocates the use of mechanical gauging to eliminate the factor of personal skill. He quotes a valuable opinion of H. le Chatelier, given in a paper presented at the last meeting of the American Institute of Mining Engineers, August 1893, which runs as follows:—"The method of tension is at present most widely used, but the preference for it is not well founded. Here, as in rupture by bending, only the *surface* of the briquette acts in a really useful way, and its inevitable irregularities and alterations so greatly affect the precision of the results that they can be in no case trusted nearer than about 20 per cent. This preponderant influence of the superficial parts was first shown by the fact that the resistance of briquettes of different sizes increases, not with the section, but, on the contrary, with the perimeter. Finally, M. Durand-Claye has shown that the interior of a briquette may be removed without notably diminishing its resistance to rupture by tension, and has given a complete theoretical explanation of the phenomenon which seemed at first sight paradoxical."

The author considers that the determination of the value of Portland cement requires the following tests:—(1) chemical analysis; (2) determination of fineness; (3) determination of tensile strength (preferably on mechanically-gauged briquettes); (4) determination of crushing strength; (5) determination of variation of volume by a test of the class of Fajja's hot test.—B. B.

*Determination of the Refractoriness of Fireclays.* H. O. Hoffman and C. D. Demond. Eng. and Min. J. 1894, 367—369.

Two methods are available for determining the refractoriness of a fireclay: (1) by deduction from the results of its analysis; (2) by direct experiment with it at high temperatures. Seger has shown that whilst the fusing point of pure alumina lies above that of his cone No. 36, and that of silica is identical with the fusing point of cone No. 35, mixtures of the two oxides are less refractory than either alone, the maximum refractoriness being found for  $Al_2O_3 \cdot 2SiO_2$  and the minimum for  $Al_2O_3 \cdot 17SiO_2$ , a decrease of fusibility occurring as the proportion of silica is further increased. For an ordinary fireclay therefore (apart from its content of oxides other than silica and alumina) the refractoriness increases with the percentage of alumina. The precise influence of foreign oxides is disputed, Bischof holding that such substances increase in fluxing effect in proportion to their molecular weights, while Seger's dictum is that ferric oxide is especially vigorous in this respect. It is agreed, however, that should the sum of these oxides exceed 6 per cent. of the ignited clay, such clay cannot be considered refractory. Regard must be paid not only to the chemical composition of a fireclay, but also to its mechanical structure, a coarse-grained clay being less fusible than a fine-grained, and a compact than a loose one, *ceteris paribus*. The authors have endeavoured to find a means of determining the refractoriness of clay without the use of extremely high temperatures, and have adopted a furnace fed by gas and air, both heated before combustion, and so constructed that the behaviour of the test-pieces could be observed. The test-pieces were pyramidal, about  $\frac{3}{4}$  in. at the base and having a height of  $2\frac{1}{2}$  in., and were made from materials ground to pass a 100-mesh sieve, mixed with water or dextrin. These were well dried, heated over a Bunsen burner, and subjected to the temperature of the furnace. In order to gauge the refractoriness of the clay, lime (added in the form of calcium carbonate) was used as a flux, being employed in regulated amount alone or in admixture with silica. Two such mixtures that proved useful are three parts of silica to one of calcium carbonate, and five of silica to one of calcium carbonate. The quantity of flux requisite to cause fusion of the test-cones serves as a measure of the refractoriness of the clay. The results are

recorded in a series of reproductions from photographs of the test-pieces, for which the original should be consulted.

—B. B.

## PATENTS.

*Improvements in the Manufacture of Cement.* G. Guj, Paris, France. Eng. Pat. 7211, April 7, 1893.

CARBONACEOUS schists, shales, or slaty substances are used as a substitute for the clay generally employed in the manufacture of cement. Such materials are mixed with limestone in proportions governed by the usual ratio of acid to basic oxides, thoroughly powdered and incorporated, damped, moulded into briquettes, and burnt in a ring kiln. The carbonaceous matter present in the schist is advantageous in that it acts as a substitute for a portion of the fuel necessary to burn the cement to the requisite degree.

—B. B.

*Improved Decorative Material or Surface in Imitation of Marble.* G. A. Goegg and J. C. Früh, Geneva, Switzerland. Eng. Pat. 12,468, June 26, 1893. (Under International Convention.)

DOLOMITE is calcined at such a temperature that it is rendered capable of setting with water. It is veined with pigments in a mould, the bottom of which constitutes the face of the finished slab, and on this face a backing of similar material not coloured or decorated, and mixed, if desired, with "sand, broken tiles, and the like," is cast, a composite slab having a marbled face being thus produced.

—B. B.

*Improvements in the Manufacture of Cement from Chance or Le Blanc Alkali Waste or the like.* B. K. Rigby, Ditton, F. A. R. Neill, St. Helen's, and A. C. Carr, Rainhill. Eng. Pat. 19,705, October 19, 1893.

THE waste is run into tanks, washed by decantation to remove soluble salts, and mixed with sufficient lime, "in the form of oxide, carbonate, or hydrate," to reduce the total percentage of objectionable impurities to 3 per cent. or  $3\frac{1}{2}$  per cent. of the whole, reckoned on the dry material. The deleterious constituents of Chance waste are calcium sulphate, sodium sulphate, ferrous sulphide, and free sulphur; those in ammonia-soda waste (which is particularly specified) are calcium sulphate, calcium chloride, and sodium chloride. The mixture of washed waste and lime is used for making cement in the customary manner. The washing tanks used for purifying the waste are patented.

—B. B.

*Improvements in Apparatus for Crushing Dry or Wet Materials, such as Cements, Lime, Plaster, Alabaster, Kaolin, Quartz, Phosphates, Materials, and the like.* C. Morel, Dromène, France, and A. Heimpel, Drome, France. Eng. Pat. 1377, January 22, 1894.

See under I., page 623.

*Improvements in and relating to the Production of Asphaltic Composition in the form of a Powder or of a Mastic.* C. H. Slicer, Baltimore, U.S.A. Eng. Pat. 2582, February 6, 1894.

THE merit of natural asphalt rock, such as that known as Neufchatel, depends on the fine state of division of the calcium carbonate or other mineral matter and the perfection of the coating of the constituent particles with the bituminous matter. The bituminous matter itself is found, upon extraction from the mineral matter, to be semi-fluid and plastic. The patentee imitates the natural material by taking 1 part by weight of pure asphaltum (rendering it plastic if necessary by the addition of heavy petroleum oil in the usual way), adding to it 50 per cent. of its weight of benzoline or gasoline, and mixing the product with 9 parts by weight of powdered limestone. The state of division



of the limestone is such that about 50 per cent. of it will pass through a 100-mesh sieve, the remainder passing in about equal proportions through a 60-, 40-, and 30-mesh sieve. An alternative plan consists in grinding the limestone so that the whole will pass a 60-mesh sieve. The solvent used for the asphalt is removed by exposing the mass to a temperature of  $200^{\circ}$  to  $250^{\circ}$  F., whereby the solvent is recovered and the asphalt fused, consolidating the mineral matter, each particle of which is coated with asphalt. The solvent may also be removed by evaporation by an air-blast at the ordinary temperature and the asphalt fused *in situ* when the product is being laid down as a paving material. Asphalts containing matter insoluble in benzoline need to be used in greater proportion than is specified above. The process may be used for improving the quality of natural asphalt rocks poor in bituminous matter. Another application of the method is the preparation of a mastic in the following manner. A natural asphalt rock in powder containing a known amount of asphalt is treated with pure asphalt mixed with benzoline, the solvent driven off at a temperature of  $200^{\circ}$  to  $212^{\circ}$  F., and a mastic thus formed. The proportions requisite vary with the richness of the asphalt rock in asphalt, the fineness of the mineral matter, and the quality of the asphalt added.—B. B.

*Improvements in the Manufacture of Artificial Stone and Marble.* P. Baumert and A. Pieck, Berlin, Germany. Eng. Pat. 4223, February 28, 1894.

The artificial stone or marble is made from a mixture of cheap aggregate such as sand or powdered slag and a binding material, e.g., magnesite and enough solution of sulphate of magnesia of "12° to 20°" to moisten and set the mixture. The face of the artificial stone is made in a corresponding manner, save that the aggregate is of better quality, consisting of ground marble or similar stone. The two layers are consolidated by pressure, and the whole allowed to harden.—B. B.

*Improvements in or Connected with Cements or Cement Work.* C. A. Ropes, Liverpool, and J. C. Sellars, Birkenhead. Eng. Pat. 5595, March 17, 1894.

FOUR-AND-A-HALF parts of barytes (barium sulphate or carbonate), 3 of silicate of soda, and  $4\frac{1}{2}$  of Portland or Roman cement, are used to constitute a cement which can be used for making joints or covering surfaces that are more or less exposed to contact with water. The mode of application may be varied according to the purpose in hand and the time during which it is desirable that the mixture shall remain unset. In the case of the setting being required to take place after three or four hours the barytes and silicate of soda may be applied and the Portland or Roman cement blown or dusted on. The composition may be applied to metal surfaces and is said to preserve them from rusting. Alum alone or with silicate of soda may also be applied to ordinary cement to increase the resistance of the cement to the action of water.—H. B.

## X.—METALLURGY.

*Observations on the Constitution of the Rock Matrix of Platinum.* S. Mennier. Comptes rend. 1894, 118, 368—369.

The author draws attention to the agreement of a note by Inostranzeff (Comptes rend. January 29, 1894) with an earlier publication of his own, in regard to the resemblance between the irregular and often filiform occurrence of native platinum in its matrix and that of metallic minerals in meteorites. This resemblance he attributes to identity in the conditions of formation. The platiniferous rock cannot

have been produced entirely by fusion, as the magnesium silicate rock would fuse before the platinum was even softened; whilst, if sufficiently heated, the silicates would ultimately enclose spheroidal grains of platinum, and would exhibit an entirely different fracture from that of the present rock. He holds that the metal has been deposited in fissures in the original peridotite by the interaction of hydrogen and platinum chloride vapour at a temperature far below fusing point. His own experiments point to this conclusion, and further account for the occurrence of limonite, magnetite, or chromite, by supposing them to be formed through the action of water upon iron and chromium (similarly deposited) at the time that the peridotite became changed into serpentine. These theories, while combating the idea that meteorites are necessarily formed by igneous fusion, are directly concerned with the origin of the fundamental rocks of the earth.—W. G. M.

*The Metallurgy of Lead.* J. B. Hannay. Mining Journal, 64, 428—425 and 452—453.

(A paper read before the Institute of Mining and Metallurgy.)

I. *Reactions of Lead and Sulphur.*—The author decides against the existence of subsulphides of lead, showing that they are mixtures of Pb and PbS, which can be fractionated by successive fusion and partial solidification.

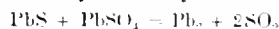
II. *Specific Gravity of Lead Sulphide.*—This was found to be 7.766 for both the native and artificial varieties. It was found necessary to make the determinations in benzene, as water oxidises finely-ground galena even in the cold.

The specific gravities of fused mixtures of PbS and Pb, in proportions equivalent to Pb<sub>2</sub>S<sub>3</sub>, Pb<sub>3</sub>S<sub>2</sub>, Pb<sub>4</sub>S, Pb<sub>5</sub>S, and Pb<sub>6</sub>S, gave numbers very close to the means of the specific gravities of the constituents.

III. The method of analysis was to add in small quantities at a time, the galena, finely powdered, to a mixture of nitric acid and potassium chlorate at 50° C. After evaporating to dryness, pure sodium carbonate and a little water were added, and the mixture was boiled for a few minutes. After cooling, pure sodium bicarbonate was added and the whole allowed to digest for a quarter of an hour. It is claimed that by this treatment all the lead remains in the insoluble part, whilst the sulphur as sodium sulphate is left in solution.

IV. This division of the paper contains a series of investigations to test the truth of the reactions commonly said to underlie the theory of lead smelting.

(a.) The reaction represented by—



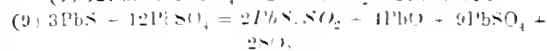
is first investigated. The experimental method ultimately adopted was, to add cautiously one substance in the solid state to the other, fused in an atmosphere of nitrogen, and to correct and investigate the solid and gaseous products.

It was found that the equation given did not represent the whole of the reaction, as lead sulphide appears to form a volatile series of compounds with SO<sub>2</sub>, CO<sub>2</sub>, CO, and H<sub>2</sub>O. These products are colourless gases at and above the temperature at which hard glass tubing softens, but are decomposed on cooling into PbS, and the gas combined with it. The SO<sub>2</sub> compound appears to be PbS.SO<sub>2</sub>.

Lead sulphide by itself was shown to be perfectly non-volatile at a white heat in an atmosphere of nitrogen.

Liquid SO<sub>2</sub> under pressure does not form the compound with PbS, but the reaction which appears to take place, is—  
 $4\text{PbS} + \text{SO}_2 = 2\text{PbS.PbO} + \text{S}_2$

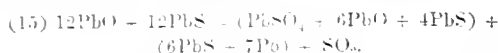
The reaction between PbS and PbSO<sub>4</sub> varies according to the temperature and the proportions used. The extremes of the reaction appear to be—



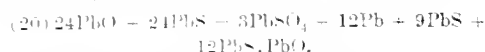


When the heat is pushed high enough, the silica of the porcelain-crucible used begins to react, and the results are very complicated.

(b.) The author next examines the reactions between litharge and galena, and finds that the equation usually given, viz.:  $\text{PbS} + 2\text{PbO} = \text{Pb}_2\text{S} + \text{SO}_2$  does not represent the reaction. The mutual action of these substances also varies with the quantities and temperatures used. In this case the extreme reactions are—



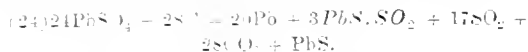
The matter on cooling separates into two layers, the upper one being the  $(\text{Pb}_2\text{SO}_4 + 6\text{PbO}) + 4\text{PbS}$  and the lower  $(6\text{PbS} - \text{Pb}_2\text{S})$ . The other extreme of the action is—



The smelters' grey or black slags consist of the mixture on the right-hand side of equation (20) without the lead.

The compound  $\text{PbO.PbS}$  is rapidly oxidised by air to a mixture of  $\text{PbO}$  and  $\text{PbSO}_4$ ,  $\text{SO}_2$  being given off.

(c.) The action of carbon on sulphate and silicate is examined. The silicate is not reduced by carbon at any temperature, but the sulphate reacts thus—



(d.) The action of heat on lead sulphite is shown to vary with temperature, and may be caused to produce either volatile  $\text{PbS.SO}_2$  or a mixture of  $\text{PbS}$ ,  $\text{PbO}$  and  $\text{PbSO}_4$ .

V. The operations of smelting lead ores are shown by the results of these investigations to be the alternate formation of mixtures of lead and lead sulphide followed by the liquation of the more fusible lead. The explanation is found for the practice of adding lime at a certain stage, viz., to effect the decomposition of the silicate of lead; and the existence of the volatile lead compounds with  $\text{SO}_2$ , &c., is shown to account for the formation of the obnoxious lead fume.

VI. A new method of smelting lead ores is proposed, by blowing air through melted galena in a basic-lined converter. The volatile compound  $\text{PbS.SO}_2$  will be formed, and can be condensed in the form of either  $\text{PbSO}_4$  or  $\text{PbS}$ , and mixed with a subsequent charge. It is claimed that the process is capable of great variation according to the product required.—J. M.

#### Alloys of Iron and Nickel. F. Osmovl. Comptes rend. 113, 532—534.

THE author has examined a series of nickel-iron alloys (prepared by R. A. Hadfield) of the following compositions:—

Mark.	Carbon.	Si licon.	Manganese.	Nickel.
A	0.19	0.31	0.79	0.27
C	0.13	0.23	0.72	0.94
E	0.19	0.20	0.65	3.82
G	0.17	0.28	0.68	7.65
J	0.23	0.24	0.93	15.48
K	0.19	0.27	0.98	19.64
L	0.16	0.39	1.00	24.51
M	0.14	0.28	0.86	29.07
N	0.15	0.31	1.08	49.65

The three "points of transformation" noted by Teheroff as  $a_1$ ,  $a_2$ , and  $a_3$ , respectively, were found at the following temperatures:—

	$a_1$	$a_2$	$a_3$
Steel containing 0.16 per cent. of carbon, but not nickel.	600° C.	700°—750° C.	820° C.
A	635—640	695—715	765—775
C	645—625	685—645	745—755
E	550—565	635—645	
G		595—545	
J		120°—130°	
K		65°—85°	

With L the transformation was not complete at the ordinary temperature. No point of any kind was noticeable with M; with N a feebly marked point was observed at 340°—380°, apparently due to the nickel, and not to the iron.

As regards magnetic qualities, all the alloys from A to L were strongly magnetic, the property being somewhat less marked with the alloys II to L. M was almost non-magnetic, especially at 70°; whilst N showed a slight degree of magnetism, probably because the nickel was in excess. J, K, and L were markedly magnetipolar; L became non-magnetic on heating, so long as it did not cool below 50°; between 50° and 0° it became magnetic, but lost the property on again heating to redness.

The hardness of samples J, K, and L was greatly increased (as tested by a file) during cooling, as the molecular rearrangement progressed. The magnetic polarity and degree of hardness appeared to change simultaneously, both being due to incomplete transformation.—C. R. A. W.

#### Nickel: its History, Uses, and Distribution.

A. G. Charlton. J. Soc. Arts. 42, 496—511.

THE author describes the history of nickel from the earliest period, and in doing so points out that the Bactrian King Euthydenos, 250 B.C., employed coins containing 22 to 23 per cent. of nickel and 77 to 78 per cent. of copper, which closely approximates to the proportion 25 to 75, which experience has taught is the most desirable admixture of these metals for coinage. In these old coins no arsenic is shown by analysis, which debars arsenical ores as being the source of nickel, and points to the use of sulphides, since the silicates could scarcely have been treated at that time.

The commercial use of nickel received its chief impetus only as recently as 1888, when the Canadian Copper Company erected their first furnace, the advantages of the nickel-steel alloy being at about that time investigated in this country by Riley. The results of the latter are to be found in the Journal of the Iron and Steel Institute of May 1889 (this Journal, 1889, 547 and 896).

One by one the objections raised against nickel-steel have now been removed, the effect of cold weather on the plates having been proved to be of no consequence. The toughness of nickel-steel renders it particularly useful for armour-plates, and is illustrated by the fact that blocks cut from some plates at Bethlehem, many of them weighing several tons, cannot by any method yet devised be profitably broken up into sizes suitable for returning to the furnace. Surface hardened plates with the extremely hard exterior, and the tough untreated steel behind, shatter forged steel Holzer projectiles, made of highly carbonised steel containing 0.8 to 0.94 of carbon and 0.94 to 2 per cent. of chromium, which have hitherto proved irresistible. Nickel steel is practically incorrodible and can be advantageously made in the basic open hearth furnace.

Whilst a thousand tons of nickel flooded the market in the early part of the century, over ten million pounds, that is about

five times the quantity, was produced in 1891, the excess probably having been used in nickel steel. There is considerable hesitation in adopting a new material in such cases as for boiler plates, bridge building, and marine engineering, where the metal is subjected to extremes of temperature, but extensive experiments are being carried out, and last year it was decided to place sections of nickel-steel propeller shafting in two of the United States warships. Using this stronger steel will warrant boring out the shaft, materially lessening the weight, whilst preserving its efficiency, and, moreover, such cored shafting can be hollow forged when the hole is large enough to admit a mandril. If nickel-steel can be applied to the construction of boilers, it will enable their thickness to be reduced one-third. The results of some comparative trials are given in the New York Mining Journal of February 25, 1893, from which the following deductions are drawn:—(1.) Nickel steel has an elastic limit of nearly 31 per cent. more than ordinary steel. (2.) The tensile strength of nickel steel is greater by about 20 per cent. (3.) The ductility of steel is not reduced by the presence of nickel.

Another important channel of consumption is the manufacture of a nickel-copper alloy (Ni 20 per cent., Cu 80 per cent.), for casing bullets to be used with small-bore rifles. This alloy has a higher degree of tenacity than the best brass, combined with a higher coefficient of elongation.

The author then deals with the geological distribution of nickel ores. In describing the Canadian deposits he points out that, according to Browne, the ore frequently loses the character of a copper ore which it possesses at the surface, becoming more and more nickeliferous and less cupriferous as the depth increases. The evidence of this, however, would hardly appear to be sufficiently extended to justify founding any general conclusions of similar enrichments in other cases. The following analyses of the average output in the respective mines show the ratios between the nickel and copper contents of the ore:—

	Copper Cliff.	Evans.	Stobie.
Cu.....	1.31	1.43	1.92
Ni.....	5.57	3.74	2.31

The higher the nickel contents of the pyrrhotite in these ores the lower is the ratio of copper to nickel. This is scarcely chance, but results probably from the relation existing between the small contents of copper and iron held in the silicates of the respective eruptive magmas. In speaking of Garnierite he points out that the deeper brown it is the richer the ore is reported to be. This also points to the fact that the association with iron appears to favourably affect the nickel contents of the ore.

Finally the author deals with the genesis of nickel deposits and concludes that it may in most instances be traced to the ultrabasic rocks and their derivatives, serpentines, and magnesian silicates. It is believed that the nickeliferous rocks came from greater depths within the earth than circulating water is likely to have penetrated; much deeper probably than any vein fissure could have extended to. The eruption of these rocks may probably have brought them within reach of surface agencies, and the subsequent leaching by mineral waters may account for the principal deposits at the disposal of the miner existing in their present form and condition.—A. W.

#### *The Results of Heat Treatment on Manganese Steel, and their Bearing upon Carbon Steel.* R. A. Hadfield. Iron and Steel Institute, Spring Meeting, 1894.

THE author disputes the validity of the  $\beta$ -iron theory in explaining the points of recalescence and the disappearance of magnetism in iron and steel. He believes that it will be eventually found that the disappearance of magnetism in manganese steel is considerably due to a particular kind of carbide of manganese, or carbides of manganese and iron, the properties of which vary according to the thermal treatment.

Considering the large amount of iron present in these steels it might be imagined that this non-magnetisability was not permanent, and the present experiments show that this is to some extent correct. The  $\beta$ - or hard form of iron explanation is hardly possible, for it is found that the same material in each case, possessing practically the same composition, can be made to become magnetic or non-magnetic at will. The author believes that the explanation must be looked for in some other direction than allotropic modifications of iron, whether in respect to manganese steel or quenched carbon steel. The true one will probably be found by examining the various carbides of iron and manganese. Both carbon steel, if rapidly cooled, and manganese steel, when quenched in water or normally, show no recalescence or other critical points, which the author believes is strong proof of the formation of carbides, probably of a much more complex nature than hitherto imagined.

The inconsistency in the  $\beta$ -iron theory pointed out by Wigham, that the permanent magnetism of hard steel, which is supposed to consist of  $\beta$ -iron was not compatible with the non-magnetisability of manganese steel, which also is supposed to contain  $\beta$ -iron, has not been satisfactorily explained. Hardness seems to be quite independent of magnetic characteristics, as very hard carbon steels are magnetic whilst hard manganese steel is non-magnetic, although the latter can now be made to become considerably magnetic. If the presence or absence of these properties can be shown to be independent of the physical property known as hardness, it can hardly be possible to claim that non-magnetic steel is proof of the existence of a non-magnetic  $\alpha$ - or  $\gamma$ -iron. Further, if  $\beta$ -iron can be made magnetic or non-magnetic at will, and of practically the same hardness in each condition, it will be inconsistent to say that the non-magnetic state of iron heated above 740° C. is due to the existence of atoms of  $\beta$ - or hard iron retained in that form even when cooled down. Roberts-Austen recently stated that the author failed to distinguish between the hardness or softness of the atoms and the hardness or softness of the mass. Miller, when speaking of  $\beta$ -iron, says, "We are faced with the following exceedingly difficult proposition:  $\beta$ -iron is as soft as glass and brittle,  $\alpha$ -iron soft and tough. This being so, carbon is no directly hardening constituent." The author believes that advance must now come from the fuller study of micro-structure and analytical methods. He agrees with Osmond that carbon at high temperatures possesses "a particular chemical activity"; not, however, in producing  $\beta$ -iron, but in forming carbides, whose properties as yet are imperfectly understood.

The author then describes his own experiments. A piece of non-magnetic manganese steel was cemented in the ordinary manner and afterwards magnetised, when it retained a considerable coercive force. After being kept several months this coercive power was lost, showing it to have been only feeble. The effect of forging and water-quenching on this magnetic material was to render it equally as non-magnetic as in its original state. A bar of manganese steel was cemented and became magnetic. Half the bar was heated, forged, and water-quenched and became non-magnetic, the other half being left in the cemented condition, thus producing a combination of magnetic and non-magnetic qualities on the same bar. The author thinks that the change is a question of continuous heating (probably changing or decomposing the particular form of carbon or carbide) rather than an increase or decrease of carbon, such as is produced by cementation or annealing. To test this, a sample was placed for 50 hours in an annealing furnace, and was then found susceptible to magnetic influence. Apparently the heat treatment must be continuous through a considerable time and accompanied by very slow cooling. This is evidenced by the fact that merely heating manganese steel to a very high temperature for a brief period two or three times will not change its non-magnetic character.

The author then tried the effect of decarbonising manganese steel. The term is often a mis-nomer, since so-called decarbonisation of cast iron to produce malleable castings does not reduce the amount of carbon, the change of state of metal being due to a change of the carbon. Manganese seems to retard this change, and it would seem that

carbide of manganese under these conditions is not readily decomposed or dissociated. A thin bar of manganese steel was decarbonised from 1.08 to 0.06 per cent. of carbon, and was then found to possess considerable magnetic susceptibilities. Unlike the cemented samples, however, when heated, forged, and water-quenched, the decarbonised metal in no case again became non-magnetic. The magnetism was persistent, although apparently no change in the chemical composition had occurred, except as regards carbon. The suggestion thus presents itself that the magnetic qualities of manganese steel are considerably influenced by carbide of manganese. This is supported by the fact that the susceptibility of "Low" manganese steel is twice or three times that of *spiegel*, although the amount of manganese is practically the same in both cases. The conditions in which carbon exists in steel are becoming of more and more technical importance, and methods of analysis have yet to be devised to properly estimate them. A sample of cast iron containing 3.07 per cent. of graphite was, after quenching, found to contain only 1.74 per cent. of graphite and 0.83 per cent. of combined carbon, the remainder, 0.62 per cent. being unaccounted for, and consequently present in some other form not easily detected by ordinary analytical methods. No carbon could be lost by merely heating, since a fortnight's previous annealing of the same sample still left the total carbon at 3.07 per cent. The removal of carbon therefore is not necessary to produce a soft and tough casting; all that is required is the transference from the hardening to temper or graphitic temper carbon. Taking these changes of malleable cast iron and manganese steel into consideration, "does it not seem unnecessary, in order to explain the hardness produced in steel by water-quenching, to call into play a hypothetical explanation that iron is changed from the  $\alpha$ , or soft state, which all admit, into the supposititious, or  $\beta$  hard form."

A bibliography of the literature on the question is appended.—A. W.

*The Physical Influence of Elements on Iron.* J. O. Arnold. Iron and Steel Institute, Spring Meeting, 1894.

The object of this research was to gain, if possible, decisive answers to the two questions: (1) Is the influence of elements on iron governed by the periodic law as enunciated by Roberts-Austen? (2) Is there an intensely hard allotropic modification of the metal iron as stated by Osmond? The author attacks strongly the  $\beta$ -iron theory, and gives results of a large number of experiments, from which he argues that it cannot be accepted. The theory is attacked by the preparation of a series of alloys of practically pure iron with appreciable quantities (1 to 2 per cent.) of the various elements, the preparation of which was beset with numerous difficulties. A consideration of the results shows that the alloys, without exception, are harder than pure iron. Copper, the atomic volume of which is practically identical with that of iron, and which, according to the periodic law, should have little or no effect, was found to considerably harden the iron, much more so even than nickel, which is alleged to rank next to carbon in hardening power. Phosphorus also, which should theoretically have a softening influence, also appears to have a hardening effect. There is likewise a great difference in the effects upon iron of the two closely related elements, arsenic and phosphorus. Three hardened bars, supposed to contain elements more or less powerful in their  $\beta$ -iron-producing properties, all bent double without any sign of fracture. Further, when the three elements supposed to be most efficient in producing  $\alpha$ - or soft iron were tried, the bars began to break. The author allows to  $\beta$ -iron theorists that the sulphur alloy is the softest of the series, but he combats nearly every other conclusion. "The inconsiderable nature of the abrasion hardness produced in the whole of the other alloys may be exemplified by the fact that after the most drastic hardening they all filed with ease, and could be turned and drilled without difficulty (except perhaps the phosphorus alloy) with ordinary shop tools."

The author speaks of the three points of arrestation during the heating and cooling of irons by the terms Ar 1, which is the lowest temperature (650° C.), Ar 2, the intermediate temperature (750° C.), and Ar 3 the highest

(850° C.). Ar 2 was originally regarded by Osmond as probably the retarded termination of the point Ar 3. The author believes that Ar 3 marks the formation of a sub-carbide of iron, whilst the point Ar 1 is due to the combination of the elements to form the normal carbide,  $\text{Fe}_3\text{C}$ . He finds that the absorption of Ar 3 into Ar 1 is coincident with the practical disappearance of the sub-carbide. He admits that the hypothesis in respect to Ar 3 is negatived by the alleged fact that electrolytic iron also develops this point in its most marked degree, but he maintains that the evidence in support of the latter fact is very feeble. He cannot state absolutely that chemically pure iron only shows the point Ar 2, because such a metal has yet to be prepared, but there is evidence of considerable weight against the appearance of Ar 3.

A general review of the recalcrescence results in reference to irons containing about 0.1 per cent. of carbon and 1.5 per cent. of the alloying element shows:—(1) the position of Ar 1 on heating is distinctly raised by the presence of silicon, chromium, arsenic, phosphorus, sulphur, aluminium, tungsten, and manganese, and less decisively by nickel. On cooling, the point is lowered by tungsten, manganese, and nickel, and raised by silicon, arsenic, phosphorus, chromium, and slightly by aluminium and sulphur; (2) Ar 2 is the only truly reversible point, and is singularly constant in its position; (3) Ar 3 is entirely eliminated by silicon, aluminium, phosphorus, and arsenic. Tungsten much diminishes its intensity, but does not greatly affect its position. Manganese, chromium, nickel, and copper lower it to or below Ar 2, whilst the action of sulphur is dubious.

Finally, the two original items of inquiry are replied to as follows:—(1) "The experimental evidence has proved beyond doubt that the ultimate physical influence of elements on iron is not in any way governed by the periodic law." (2) "That no element except carbon has (*per se*) the power of conferring upon quenched iron the property of abrasion hardness to any extent worthy of consideration."

A written contribution to the discussion by Osmond defends the position of the latter. He says that point Ar 2 marks the appearance of magnetism and is due to an allotropic change in the iron itself. All that is changed since 1890 is that points Ar 2 and Ar 3 are seen to be definitely separated, and that instead of a single allotropic modification of iron, there are at least two; in other words iron is in the  $\alpha$  state below Ar 2, is in the  $\beta$  state between Ar 2 and Ar 3, and in the  $\gamma$  state above Ar 3. He maintains that Arnold, while combating the atomic volume theory, supports it by his own experiments, and says that it is proved (1) That hydrogen, carbon, manganese, nickel, and copper lower Ar 3; (2) That carbon, manganese, and nickel lower the double point Ar 3, 2, when they are present in sufficient quantity; and (3) That tungsten, aluminium, silica, arsenic, and phosphorus tend, as the quantity in which they are present increases, to obliterate Ar 3, without modifying its position. All these facts are in accordance with the law of atomic volume, and some of them are even borrowed from Arnold's results. The law of Roberts-Austen is a limited one. "It assumes that the foreign body preserves in solution its natural and individual state, and does not form chemical compounds with the solvent. The starting point at which these compounds cease to intervene is very variable. In alloying iron with such high proportions of these bodies as 1.50 per cent. Mr. Arnold often exceeds these limits, as is abundantly proved by his own investigations on the structure of his alloys, especially in the case of phosphorus, of arsenic, and of sulphur. He therefore seeks under these conditions a verification where one was impossible."—A. W.

*The Walrand-Legnèsel Process as applied to Steel Castings.* G. J. Snelus. Iron and Steel Institute, Spring Meeting, 1894.

The process has for its object the production of steel castings suited to a general foundry, and consists in adding at the end of the ordinary blow a definite quantity of melted ferro-silicon, then making the after-blow, turning down when the extra silicon is burnt out and adding the final ferro-manganese, aluminium, &c., as circumstances require.

The object of adding the ferro-silicon is to increase the temperature, and thus to keep the metal fluid, by the oxidation of the silicon during the after-blow. The use of more siliceous pigs to commence with, and also the addition of ferro-silicon to the metal during the ordinary blowing have both been tried for increasing the temperature, but neither have proved satisfactory, because, in the first place the heat produced by the burning of the silicon is largely carried out of the converter, and in the second, an excess of silicon used in these ways is difficult to be got rid of before all the carbon is burnt. The use of spiegeleisen, with an after-blow of two minutes to burn out the manganese, and thus increase the temperature, was tried by the author ten years ago, and the metal was certainly found to be more fluid and freer from gas cavities, but the difference was not so great as to warrant continuing the process. The advantages of silicon over manganese for this purpose are, that it generates more heat unit for unit, and the flame indication when to terminate the after-blow is better marked.

The advantages of adding the ferro-silicon for the after-blow are; (1) Ordinary Bessemer pig-iron with two or three per cent. of silicon can be used, thus ensuring a steel comparatively free from silicon; (2) The combustion of the added silicon produces such a large amount of heat at the right time, and so rapidly, that the metal becomes very fluid; (3) As the silicon burns to a solid it leaves the metal comparatively free from gas, and the steel is sound and free from gas cavities; (4) In consequence of the metal being so fluid and already free from oxide of iron, the ferro-manganese, or other substances added, such as aluminium, are more effective and remain in the final steel; and (5) that in consequence of the great fluidity of the metal much more time and facility is given for casting operations.

The author gives the results of analyses and mechanical tests of metal produced by this process and shows the cost. He also works out by heat units the result that the temperature of the bath is increased by about 240° C. in two minutes by the burning of the silicon alone in the after-blow.—A. W.

#### *The Capacity and Form of Blast Furnaces.* W. Hawdon. Iron and Steel Institute, Spring Meeting, 1894.

AFTER dealing with the functions of the various parts of the blast furnace, the author proceeds to point out the advantage of an upper bosh, which can sustain the weight of a large portion of the materials above it, thus leaving that immediately below more loose and open, by which means the escape of the gases is rendered more easy and the working of the furnace freer. It is said that such a furnace has the benefit of the free working of a low furnace, combined with the efficiency of a high one. The author then proceeds to give figures obtained by him with a furnace constructed on these lines, by which it is shown that the output is increased, the fuel consumed materially decreased, although the weight of ironstone is slightly increased, and the grade of iron produced is much higher, showing the very regular working obtained. The improvement is not claimed as entirely due to the upper bosh, but to the combined effect of a somewhat large well, the low bosh, and the narrowing of the part above the bosh.—A. W.

#### *The Utilisation of Blast Furnace Slag.* A. D. Elbers. Eng. and Mining J. 1894, 392—393.

THE author traverses the dictum that blast furnace slag is useless for manurial purposes, on the ground that only the acid slags are unattacked by weathering, and that slags which are fairly basic decompose readily in the soil yielding silica and lime as plant food, and—it is said—serving to fix ammonia and nitric acid reaching the soil by means of rain. Another use for slag, which is of permanent importance, is the manufacture of slag wool for which a growing demand exists. The presence of sulphur in blast furnace slag is a hindrance to this and other applications, e.g., the preparation of slag cement, and the elimination of

the sulphur is therefore a point to be aimed at. The refining of blast furnace slag by converting it into slag wool and thus removing a good deal of ferruginous matter, has been attempted for the production of a ceramic raw material, but the process is at present in the experimental stage. The composition of refined slag corresponds approximately with the formula  $12Ca(Mg)O \cdot 4Al_2O_3 \cdot 7SiO_2$ . Its melting point is nearly as high as that of orthoclase, but is lowered by the addition of quartz. The comparatively low content of alumina of refined slag, causes the ceramic mixtures made by its aid to be more nearly transparent than those of the usual constituents. In consequence of their properties the value of refined slag in the ceramic industry arises from the fact that vitrification can be effected at a moderate temperature, and with a low proportion of vitrifying ingredients, e.g., felspar, and that the product is noticeably translucent. The author states that porcelain clays are often less plastic than is desirable, and it is consequently difficult to add to them sufficient vitrifying material to yield a good ware without making the previous work of moulding and modelling unduly difficult, so that a substance like refined slag, capable of acting as a vitrifying constituent without being used in excessive amount, and thus detracting from the plasticity of the raw pottery, is of especial value. Another suggested use for refined slag is in the manufacture of glass, as a substitute for a portion of the silica and lime.—B. B.

#### *Laboratory Notes.* J. S. de Benneville. J. Amer. Chem. Soc. 1894, 16, 66.

See under XXIII., page 667.

#### *Experiments in the Analysis of Pig Copper, Brasses, and Bronzes.* J. S. de Benneville. J. Amer. Chem. Soc. 16, 133.

See under XXIII., page 667.

#### *Determination of Nickel in Nickel Steel.* E. D. Campbell. J. Amer. Chem. Soc. 1894, 16, 96.

See under XXIII., page 665.

#### *Notes on the Determination of Nickel in Steel.* J. Westesson. J. Amer. Chem. Soc. 16, 110.

See under XXIII., page 666.

#### *Experiments on the Estimation of Graphite in Pig-iron.* F. L. Crobough. J. Amer. Chem. Soc. 16, 104.

See under XXIII., page 665.

#### *The Analysis of Steel.* H. K. Bamber. Iron and Steel Inst. Spring Meeting, 1894.

See under XXIII., page 665.

#### *The Determination of Phosphorus in Steel by Dudley's Method.* O. S. Doolittle and A. Eavenson. J. Amer. Chem. Soc. 16, [1], 234.

See under XXIII., page 666.

#### *The Volumetric Determination of Phosphorus in Steel.* C. B. Dudley and F. N. Pease. J. Amer. Chem. Soc. 16, [4], 224.

See under XXIII., page 665.

*The Determination of Phosphorus in Steel in presence of Arsenic.* J. O. Handy. J. Amer. Chem. Soc. **16** [4], 231.

See under XXIII., page 666.

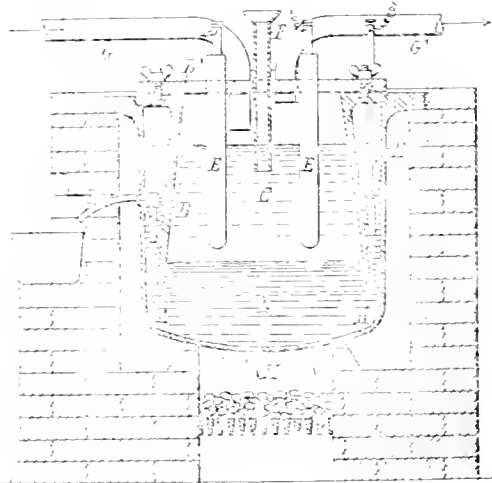
*The Determination of Phosphorus in Steel.* C. B. Dudley and F. N. Pease. J. Amer. Chem. Soc. **16**, [4], 217.

See under XXIII., page 665.

### PATENTS.

*Improvements in the Production of Chlorine and Refined Lead, and in the Recovery of the Silver the Lead contains.* F. M. Lyte, London. Eng. Pat. 7264, April 8, 1893.

LEAD chloride is formed from yellow lead oxide (preferably) or from lead oxychloride, by grinding with an excess of aqueous hydrochloric acid, in a pan having a granite bottom and stoneware sides; or enamelled metal pans may be used, an example of which is shown, in which stone mullers are carried round by cross-arms and stay-ropes of a frame revolving on a centre, and resting at their outer ends on rollers on a circular rail supported by adjusting screws to regulate the grinding action. Silver chloride may be dissolved out of the product by strong hydrochloric acid or solution of a suitable alkaline earthy chloride; and on filtering the warmed solution through spongy lead, silver is deposited. Or the argentiferous lead chloride may be fused over lead, the chlorides being used in successive charges until the lead is sufficiently enriched for recovery of the silver by cupellation.



Crude lead may be oxidised in a basic-lined Bessemer converter, in which a high temperature is attained, and the silver becomes concentrated in the small proportion of lead that escapes oxidation. The last traces of silver may then be recovered from the litharge and fume in the manner first described.

Lead chloride may be purified by solution in hot water and crystallisation, the same water or mother-liquor being repeatedly used. The chloride is then washed, dried, fused, and electrolytically decomposed in the fused state, to obtain lead and chlorine, in the apparatus represented above.

The iron pan A set over the furnace M, contains lead, L, into which the earthenware or plumbago bell B dips, the lead being melted to receive it, and then allowed to set, while the bell is charged with lead chloride by removing the cover B', and afterwards with successive additions through the inlet F. E E are carbon anodes, each hollowed to receive a fusible metal core, preferably of lead, connected to the conducting wires. H is the cathode terminal dipping into the lead L. As the molten lead increases by decomposition of the chloride, it runs off by the pipe D. The

apparatus shown is intended for connection in series, the chlorine entering from another cell by the pipe G and having outlet by the pipe G'. It is a feature of the invention, that the air in the cell should be displaced by a non-oxidising gas, as chlorine or nitrogen, in order to avoid any formation of lead oxide in the process.—E. S.

*An Improved Process, and Furnaces used therewith, for Copper Smelting and Refining.* A. O. Vicenna. Vallenar, Chili. S. America. Eng. Pat. 7667, April 14, 1893.

In this process, by means of an improved compound furnace, sulphurous copper ores may be reduced to copper bars in one heat by a single and continuous operation. The first part of the furnace consists of a cupola furnace supplied with the usual blast or tyvere pipes. Into this furnace the softer and more easily reduced ore is placed together with coke or other fuel. It is rapidly smelted and then discharged into an auxiliary reverberatory hearth. Here the dust ores and siliceous ores are added and the iron is slagged. But little additional heat is here required, and this is supplied by auxiliary furnaces. The slag is removed in the usual way, and the molten copper sulphide, settling at the bottom, passes on into secondary hearths. Here the matte free from slag is exposed alternately to a desulphurising and oxidising current of air, heated by special pipes sunk into the sides of the hearth.

Before the matte gets cool and begins to redden, it may be reheated by opening dampers, and any remaining iron and impurities removed by the addition of a siliceous ore to form a slag. The bath in these secondary chambers may be thus alternatively reheated and treated with siliceous ores, or oxidised by the admission of air for desulphurisation until the copper, in a single heat, from its first smelting is reduced to fine bars. If desired, the ordinary poling with green timber may be carried out in the secondary hearths. The auxiliary hearths and grates may also be used with an ordinary reverberatory furnace in place of the cupola, though in this case the copper pyrites ore must be previously calcined. It is stated that poor ores, which previously could not be treated at a profit, may be smelted in this combined furnace to advantage.—A. W.

*An Improved Process for the Preparation of Iron or Steel Plates for Nickel or other Plating.* C. T. J. Oppermann, London. Eng. Pat. 7791, April 17, 1893.

By this process iron or steel plates are prepared for a polished plating of nickel or other metal, without rendering them too brittle to be afterwards worked and bent into any desired shape. Ordinary steel or iron plates (known as tin plates) are given an extra coating of soft metal, such as tin, an alloy of tin and aluminium, or an alloy of tin and lead, thus protecting them so that the succeeding rolling does not unduly harden the iron or steel. This extra coating is obtained by dipping in a bath of the soft metal in a molten state, after which the plates are rolled between polished rollers and polished by a revolving mop charged with crocus or tripoli powder.

The plates are then cleaned by means of a mixture of lime and water, rinsed with cold water, and suspended in the electro bath of nickel, &c. The electro-plated articles are dried in sawdust and polished by the revolving mop, which is this time charged with dry lime.—A. W.

*An Improved Method of Recovering Tin from Tin Plates, Tin Boxes, and other Tin Waste.* C. W. Kriens and J. R. Harper, both of London. Eng. Pat. 9257, May 9, 1893.

In this process the tin plates or boxes are placed in a solution of caustic soda of sp. gr. 1.200, containing about 15 per cent. of soda, which is contained in iron tanks. The tin is all removed in about 48 hours if cold; in from four to six hours if the solution is boiled. When saturated with tin, the solution is evaporated to dryness, and roasted or calcined, while charcoal is added in sufficient quantity to

carbonise the whole mass. From the residue the alkaline salts are washed out, reacidified with milk of lime, and used for a fresh operation. The tin is recovered as an insoluble oxide or carbonate. The sheet iron is not affected and can be further utilised.—A. W.

*Improvements in and relating to Pickling Iron and Steel Plates and other Articles previous to Tinning or Galvanising.* H. J. Kirkman, Swansea. Eng. Pat. 10,463, May 27, 1893.

In pickling iron or steel plates and other articles with sulphuric acid as preparative to galvanising, a stated proportion of sodium chloride or other equivalent chloride, or of hydrochloric acid, is added in order to hasten the pickling process. The pickling liquid may be used until there is no longer sufficient sulphuric acid present to decompose the chloride formed in the process.—E. S.

*An Improved Metallic Alloy.* D. W. Sugg, London. Eng. Pat. 10,608, May 30, 1893.

Fifty parts of copper, 40 parts of zinc, and aluminium in the proportion of two and a half per cent. of the whole are taken. This is one example, but others may be obtained by varying the amounts of copper and zinc to the same proportion of aluminium.

The mode of preparation of the alloy varies:—For a hard metal, the copper and aluminium are first mixed to form a copper alloy and the zinc added in small pieces during continuous agitation of the molten mass. This gives a reddish alloy that takes a high polish. For a ductile metal the zinc and aluminium are first mixed and the copper then added. This gives an alloy resembling brass. In both cases the metal is claimed to be non-oxidisable, proof against sea water, and, to a large extent, against acids.—A. W.

*Improvements in Steel Founding.* J. G. McRoberts, St. Louis, U.S.A. Eng. Pat. 2617, July 8, 1893. (Under international Convention.)

In steel founding about twice as much heat is required as in iron founding and in consequence shrinkage strains have to be provided for. This excessive heat also compels the employment of dry sand moulds, which are objectionable for many reasons. They are more expensive than green sand moulds. They do not yield as the steel contracts, thus giving rise to checking and cracking, especially with structures with projecting shoulders, U-shaped cross-sections, &c., and, hence, many useful forms cannot be made in cast steel.

In carrying out this process the advantages of dry sand and green sand moulds are combined. The molten metal is introduced into the moulds through a gate of substantially dry sand, but the mould itself is constructed of green sand, which becomes skin dried by the heat given off by the advancing metal before the metal actually comes into contact with the surface of the mould. Wherever cores are required, or where the molten current enters the mould and impinges as a stream upon it, dry sand is employed to prevent abrasion. Wherever resistance is offered to the shrinkage of the metal in cooling, likely to cause a rupture in the casting, the mould is of green sand, which is yielding. By this means the metal is introduced into the mould in such a manner as to prevent the formation of scars, the carrying along of foreign matter caused by the abrasion of the gate, and the casting itself is free to contract on cooling.

A further characteristic is introducing the metal into and through the mould, so that it shall not abrade any opposing part of the mould walls, by pouring it down through a gate of dry sand into a well from which it passes into the mould itself, either horizontally, or substantially horizontally, and upward. The green sand walls of the mould become skin-dried in advance of the progress of the metal, and being vented freely the moisture and gases generated in the mould are enabled to escape without interfering with the metal or the casting.

Instead of using sand any other suitable materials may be substituted, that is, refractory material, which will not abrade when molten metal is passed over it and which will not lead to the generation of steam, for making the gate, and any material of the yielding nature described for the mould itself. Steel castings made in these wet sand moulds are superior to those made in dry sand, and there is the further advantage that they may be allowed to remain in the mould for several hours, thus producing a much tougher casting than is possible with dry sand moulds, where the casting must be shaken out right away. Also checking and rupturing of the casting on shrinking are avoided, so that "reinforcements" on the casting are no longer necessary and the expense attendant on their removal is obviated.

—A. W.

*Improvements in Baths for Toughening, Strengthening, and Hardening Iron and Steel.* F. G. Bates, Philadelphia, U.S.A. Eng. Pat. 1244, January 19, 1894.

The patent refers to improved liquid baths for treating low grade steel or malleable iron for the purpose of increasing fineness of grain of the metal, improving its tensile strength, toughness, ductility, &c., or when desired rendering it harder. The bath consists of a brine of both alum, and common salt, to which brine is added oil, benzene, or other liquids, calculated to liberate hydrocarbon gas at high temperatures, and also certain salts as occasion may arise. Thus for strengthening and toughening the metal, salts of aluminium, potassium and sodium and lime are used. The hydrocarbons raise the percentage of carbon. Phosphate of lead and arsenic increase the hardness. Ammonium salts are employed to liberate ammonia gas.

The articles to be treated are heated to bright redness, placed in the bath, and allowed to remain until gas ceases to be evolved or until they are cold. Examples of the proportions in which the above salts are used are given.

—A. W.

*A New or Improved Solder for Soldering Aluminium, Aluminium Alloys, and other Metals.* O. Nicolai, Wiesbaden, Germany. Eng. Pat. 4980, March 9, 1894.

This process consists in the use of a solder for soldering aluminium of a compound of cadmium with one of the halogens, the chloride or iodide giving favourable results. The solder is conveyed on to the part to be soldered by a brush moistened with spirits of wine and the soldering pipe is then used.—A. W.

*Process for Soldering Aluminium.* E. Singer, F. Donat, and P. Kirchheim, all of Chemnitz, Saxony. Eng. Pat. 5530, March 16, 1894.

The process for soft soldering aluminium consists in dipping the parts to be soldered into a mixture of Russian tallow and Provence oil. After treating with this mixture they are joined together by means of the soldering iron with ordinary plumber's soft-solder or preferably with perfectly pure English tin. It is claimed that the parts thus soldered can be bent as well as hammered without separating, and without the solder flaking off.—A. W.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

*The Use of Electrolysis in Technical Chemical Processes.* J. W. Langley. J. Amer. Chem. Soc. 16, 49—56.

THE electrical smelting of aluminium in the United States is carried on only by the Pittsburgh Reduction Co., and there under the Hall patents (This Journal, 1891, 432, and 1892, 124). The statistics regarding the installation differ in some respects from those quoted in this Journal (*loc.*

cit.): the current density is 7,000 ampères, and the yield of aluminium 1 lb. for 18.1 EHP hours. This is equivalent to a current of 1,926 ampères at 7 volts (the voltage required when the process is in full operation) acting for one hour; hence theoretically the deposit of aluminium should be 1.43 instead of 1 lb.; the reduction efficiency of the process is therefore 70 cent. The alumina dissolves to the extent of 25 per cent. in the fused cryolite, apparently without heat evolution or effervescence, forming a colourless solution, and during the electrolysis no appreciable loss of fluorine or fluoride occurs. That the alumina alone is decomposed, is explained by the low voltage required to electrolyse it, which may be calculated by dividing the number of heat units evolved in the formation of 1 gm. (not one molecule) by Thompson's number (22,900) representing the calorific equivalent of the volt: for NaF this is  $109,700 \div 22,900 = 4.8$ ; for  $Al_2F_6$  it is  $50,000 \div (6 \times 22,900) = 4.0$ ; and for  $Al_2O_3$  only  $388,929 \div (6 \times 22,900) = 2.8$ .

The *Moebius Process* for refining gold and silver is now carried out as follows by the Pennsylvania Lead Company of Pittsburgh: a current of 180 ampères and 1½ volt is passed through dilute nitric, free from sulphuric and hydrochloric acids, contained in a tank divided up into 70 compartments arranged in electrical series, each compartment measuring 18" x 24" x 17" deep and having three anodes and four cathodes 10" x 20" x 1½". The silver anodes contain 1 per cent. of impurities consisting of copper, lead, bismuth, and a little gold, and are suspended in bags; the cathodes are thin plates of fine silver.

In each compartment the average consumption of nitric acid (36 B.) is 2 lbs., and the average yield of silver (inclusive of stoppages) is 470 oz. per 24 hours. This is equivalent to nearly 1 ton per diem in the whole series or to 84 per cent. of the theoretical yield with uninterrupted current. The silver is practically 1,000 fine; but care must be taken to prevent the co-deposition of copper; there is no practical difficulty, however, in this respect.

Edson, in the *Electrical Engineer* describes the complete disinfection of the sewage from the village of Brewsters by running into the sewers sea water which had flowed in a gentle stream between 7 electrodes, viz., 4 carbon cathodes 12" x 12" x 1" and 3 platinum-coated copper anodes, through which a current of 700 ampères and 5 volts was passing. The sea-water thus treated equalled in strength a 1 per cent. solution of chloride of lime, the ratio of the cost per gallon of the two disinfectant liquids being 1:140 in favour of the electrolytic process.—W. G. M.

## PATENTS.

*Improvements in the Extraction of Chromium by Electrolysis, applicable also to the Production of Chromium Alloys.* E. Placet and J. Bonnet, Paris, France. Eng. Pat. 6751, March 30, 1893.

THESE improvements on Eng. Pat. 22,855, 1891 (this Journal, 1893, 18) relate to methods of obtaining chromium and alloys of chromium by electrolysis from the "watery" and fused electrolytes. For the first, they take 10 to 15 grains of bisulphate of potash, 100 grains of chrome alum, and 100 grains of water; heat the mixture until the salts are dissolved, and then electrolyse, when the chromium is said to deposit immediately. The strength of the bath is maintained by means of chrome alum alone, or by means of a concentrated solution of chrome alum and alkaline bisulphate, an overflow pipe being provided. Chlorate of potash and boric acid, benzoic acid, or other analogous salt, is sometimes added to assist the deposit of the metal. For the fused electrolyte, the bisulphate and chrome alum are heated together, and, instead of the potash salt, sulphate or bisulphate of soda or ammonia may be employed as a flux, or the phosphates, borates, chlorates, silicates, chlorides, fluorides, &c., alkalis or alkaline earths, or a mixture of the same; and in place of chrome alum, oxide of chromium or other salts of chromium, which will easily fuse on contact with alkaline earths. With the electrodes may be incorporated chromium compounds previously fluxed with

different fluxes and reducing agents, and, instead of carbon, different metals may be employed as electrodes to obtain alloys, and reducing gases and vapours may be admitted to the crucible by a blast-pipe or through hollow electrodes.

—J. C. R.

*Improvements in Secondary Batteries or Accumulators.* A. E. Peyrusson, Limoges, France. Eng. Pat. 11,122, April 1, 1893.

THE positive electrode is constructed of thin laminae soldered to a central core, from which—in the illustration, where the core is vertical—they radiate, being each, in addition, soldered to end discs threaded on the core. Insulating discs of larger diameter form the two ends, and serve to keep the electrode from the surrounding cylindrical negative electrode. Or thin discs threaded on the central core and soldered to it may take the place of the radial laminae.

The negative electrode is of thin vertically corrugated material, rolled several times round with the layers in contact to form a cylinder. The several layers are united by soldering to stout end-rings; and the whole is slit longitudinally at intervals to allow free circulation of the electrolyte.—E. T.

*Improvements in the Production of Chlorine and Refined Lead, and in the Recovery of the Silver the Lead contains.* F. M. Lyte, London. Eng. Pat. 7264, April 8, 1893.

See under X., page 644.

*Improvements in Electrical Accumulators or Secondary Batteries.* P. Sekoop, Zürich, Switzerland. Eng. Pat. 7,711, April 11, 1893.

THIS patent is for methods of causing the electrolyte to circulate through the cell. In one case the electrodes—which are all made to fit tightly at their sides to the containing vessel by suitable means—alternately reach to the bottom of the cell, and terminate some distance above the bottom. By a suitable pump the electrolyte is caused to enter at one end, and zigzags under and over the alternate plates to the other end.

A cell is also described, consisting of horizontally-placed perforated steel plates, which act as cathode, while insulated rods of copper or copper amalgam pass loosely through the perforations and form the anode. The electrolyte in this case enters at the bottom by a centrally-placed tube, and is withdrawn at the top of one side.—E. T.

*Improvements in and connected with Tanks for Electrical Decomposition of Chloride of Sodium or Potassium in Solution.* J. C. Richardson, London. Eng. Pat. 12,837, June 30, 1893.

THIS invention relates to electrical decomposing apparatus wherein there is no diaphragm between the electrodes to prevent re-combination of the products of decomposition, disturbance at the cathode by hydrogen being prevented chemically. But, as in the case of decomposing common salt solution to obtain chlorine gas and soda solution, the former product may set up some disturbance in escaping from the solution, or struggling to get to the surface from the lower portions of the anodes, it is proposed to arrange horizontally between the electrodes a screen of strips of slate or glass, or other uninfluenced material, which may overlap each other, like the slats of a Venetian blind, with spaces between them for the passage of the current. An arrangement of this description is shown, and another, in which the screen takes the shape of V-troughs placed below the anodes, which will prevent solution and downward pressure of gas towards the soda solution at the bottom, and also catch particles of the material of which the anodes are composed, should any become detached during the progress of the electrolysis.—J. C. R.



*Improvements in and Relating to Electric Furnaces.* H. H. Lake, London. From A. C. Girard and E. A. G. Street, Paris. Eng. Pat. 13,340, July 8, 1893.

See under H., page 626.

*An Improved Manufacture of Carbon for Electrical Purposes.* H. H. Lake, London. From A. C. Girard and E. A. G. Street, Paris, France. Eng. Pat. 13,339, July 8, 1893.

EXPERIMENTS made some time ago by Despretz and Berthelot with the object of studying facts relating to the sublimation and fusion of carbon demonstrated that, at high temperatures, carbon of different kinds passes into the graphitic state. The present invention relates to its industrial application for the manufacture of graphitic carbon on a commercial scale, and the application of such carbon to electrical purposes, notably for lighting, electrolysis, and batteries. Carbons manufactured in the ordinary manner are subjected to such a temperature as will soften, weld, fuse, or sublime the same, and they are thus transformed (either completely or in part) into graphitic carbon or graphite. The necessary heat is obtained by making the carbon part of an electric circuit with suitable moving connections as in treating rods or plates, or the requisite temperature may be arrived at by an arc playing over the surface of plates giving them a certain depth of graphite; or granular or powdered carbon may be similarly treated by passing between electrodes at a rate of flow allowing it to be brought up to the requisite temperature, and such graphitic carbon may be used in the manufacture of carbon by agglomeration or in telephones or microphones. Crude carbon may be treated, first distilling the volatile products contained therein. Eng. Pat. 13,340, 1893, is referred to as describing suitable apparatus in which to heat the carbon by an electric arc (see previous abstract).—J. C. R.

*Improvements in Electrodes and Cells for Secondary Batteries or Accumulators.* E. H. Wheeler, East Molesey. Eng. Pat. 22,912, November 29, 1893.

EACH electrode is composed of two perforated plates united at the edge to form one compound one, the perforations being conical with the bases inwards, so that the active material retained in the space between the plates, and in the perforations themselves, cannot fall out. The containing vessels are formed with grooves not reaching to the bottom. On the shoulders thus produced ledges formed on the plates rest, and thus support the latter away from the bottom of the cell.—E. T.

*Improvements in Galvanic Batteries.* A. J. Boulton, London. From C. Cudell, I. Cudell, and M. Cudell, Aix-la-Chapelle, Germany. Eng. Pat. 3749, February 21, 1894.

THE one electrode is in the shape of a cylindrical vessel of carbon or "brownstone," and is furnished with a glass or stoneware ring at its upper end. The other electrode is of perforated zinc and is attached to a cover. This cover has lugs, which, travelling in inclined grooves in the stoneware ring, enable the cell, with the help of rubber, to be sealed, while allowing gas to force its way out if the pressure rise sufficiently inside the cell.—E. T.

*Improvements in Electrodes for Arc Lamps.* E. O. Clark, New York, U.S.A. Eng. Pat. 4114, February 27, 1894.

THIS invention relates to electrodes for arc lamps provided with protective coverings to prolong the life of the electrodes and prevent useless dissipation of the electrodes or their coatings. The carbon electrodes are coated as usual with a film or covering of copper, and over this a coating of a metal differing in nature from copper, such as zinc and nickel, applied mechanically, chemically, or by electro-deposition, for which in practice excellent results are claimed, in the shape of longer life, not flaking away, dissipated, nor come to a sharp point in burning, but uniformly consumed.—J. C. R.

*Improvements in producing Nitric Acid and Metals from Nitrates.* J. D. Darling and H. C. Forrest, Philadelphia, U.S.A. Eng. Pat. 5808, March 20, 1894.

THE invention consists in electrolysis fused sodium nitrate or potassium nitrate to obtain nitric acid and metallic sodium or potassium. A double cell, preferably of aluminium, is employed, having a removable, insulated cover, to which is fixed centrally a cylinder with funnel mouth, long enough to dip into the fused salt and fitting into the top of the partition wall which divides the two cells. This wall has one or more openings to admit communication between the cells. As the nitrate is consumed, a fresh supply is introduced to the cylinder in such manner as not to admit air. The cover carries also two electrodes (one for each cell) of carbon or platinum, the vessel itself being connected to the negative pole of the source of electricity. Each electrode is surrounded (except at the bottom) by a cup, inverted, having many openings inclined upwardly towards the inside; and is carried by a clamp secured to the cover, and having a transverse and vertical passage leading through its upper part. The cups are insulated from the cover and clamps, to which they are secured.

To conduct the process, sodium nitrate, for instance, is fused in the vessel, the cover carrying the electrodes, &c., is adjusted, and the cover and vessel are connected to the opposite poles of a generator of electricity. The nitrogen peroxide and oxygen eliminated pass from the inverted cups to a series of Woulff's bottles, in which nitric acid is formed. The sodium first liberated reduces the neighbouring nitrate to nitrite and oxide, which latter dissolves in the sodium nitrite; then, both are decomposed, and the sodium set free floats on the surface of the fused salt, and may be withdrawn through an aperture closed by a plug in the side of the vessel.—E. S.

*Improved Method and Apparatus for Manufacturing Caustic Soda.* T. Craney, Bay City, U.S.A. Eng. Pat. 6426, March 30, 1894.

See under VII., pages 636 and 637.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*Solubility Tests of Dark Mineral Lubricating Oils.* Holde. Mitt. d. Tech. Versuchsanst. 11 [5], 261—272.

See under XXIII., page 668.

*A New Method of Analysing Fats and Resins.* P. C. Mellinney. J. Amer. Chem. Soc. 16 [4], 275.

See under XXIII., page 668.

*The Iodine Number of Rosin.* P. C. Mellinney. J. Amer. Chem. Soc. 1894, 16, 56.

See under XXIII., page 668.

*A Rapid and Accurate Method of Determining the Moisture and Oil in Linsed Cakes and other Feeding Stuffs.* A. P. Aitken. J. Amer. Chem. Soc. 16, 114.

See under XXIII., page 669.

## PATENTS.

*Improvements in or Relating to the Treatment of Oils for Refining, Bleaching, and Boiling the Same.* W. N. Hartley, Dublin, and W. E. B. Blenkinsop, Wandsworth Common. Eng. Pat. 9315, May 9, 1893.

RAW linsed oil is claimed to be refined, bleached, and boiled to any desired degree of drying power, density, or viscosity at one operation. This oil should be previously cleared from



muilage and insoluble matters, either by storage in tanks, or by filtration or treatment in a centrifugal machine, or by any other convenient method; preferably mechanically at a temperature not exceeding 50° F., although slightly higher temperatures may be employed successfully. The oil is heated to 190–250° F., and air or oxygen blown through until all moisture is expelled, so that a sample remains clear and bright after cooling. A manganese salt of a fatty acid easily soluble in the oil is then added, either as a solid or in solution; preferably a strong solution of manganese linoleate is employed, the solvent being turpentine, a fatty acid, or oil, such as will form a liquid which will readily mix with the oil to be treated. About one volume of solution containing one-fourth its weight of manganese linoleate is added to 400 of oil, either during blowing, or during a temporary discontinuance of blowing, the current of air or oxygen being resumed immediately, so as to prevent the separation of insoluble matters, and about 15,000 cubic feet of air, or a corresponding quantity of oxygen, being blown through per hour per ton of oil. About an hour or less suffices to bleach the oil when at a temperature of 190° F.; the temperature may then be allowed to fall to 170° F., and more manganese linoleate solution added, say 1 to 1½ volumes per 100 of oil, the blowing being then continued until the desired viscosity is obtained. Or the oil, freed from moisture as described, may be cooled to 170° F., and treated with the full quantity of manganese linoleate at once, air being blown through until the required amount of bleaching and viscosity are attained; about 1½ hour's blowing generally suffices, it is said, to obtain a good pale boiled oil when operating with 5 tons of raw oil, the exact time being variable, according as the air current is greater or less than 15,000 cubic feet per ton per hour, according to the proportion of manganese salt used, and so on. If the oil originally used is not free from muilage, &c. it must be filtered after blowing to render it bright. The appliances used in carrying out the process are those described in the previous patent, No. 11,629 of 1890: one vessel being therein stated to refine, bleach, and boil oil at one operation, yielding a pale boiled oil of uniform quality and free from the disadvantages attending the presence of lead, always contained in oil boiled by means of lead compounds.—C. R. A. W.

*Apparatus for Extracting Substances by Volatile Solvents.* J. Meikle, Glasgow. Eng. Pat. 10,701, May 31, 1893.

See under L., page 622.

*Soap Cakes and Bars.* H. Rimmer, Sandside, Ulverston. Eng. Pat. 5225, March 13, 1894.

Soap tablets and bars are stamped in suitable dies so as to give a corrugated surface to one or more faces; the object of this is to enable the small remaining portions of similar used tablets to be completely used up by pressing them into the interstices of the corrugations so as to fill them up, and thus economise these remaining fragments. Instead of corrugations, variously formed projections or recesses may be stamped, or designs or letters may be employed with the same object.—C. R. A. W.

*Improvements in Apparatus for Extracting Fat and Glue.* F. J. Michalski, Brooklyn, New York, U.S.A. Eng. Pat. 5821, March 20, 1894.

THE process is designed for the continuous extraction of fat and glue from bones or other substances by heating them under pressure with a volatile solvent which is subsequently distilled off. The plant itself may be briefly described, in principle, as resembling a composite Soxhlet's extractor on a large scale.—A. G. B.

### XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

#### (A).—PAINTS, PIGMENTS.

*Spontaneous Combustion of a Lamp Shade.* A. Dupré. Times, May 21, 1894.

See under Analyt. and Scient. Notes, page 670.

#### PATENTS.

*Improvements in the Manufacture of Paint.* W. H. Hyatt, Stockwell, Surrey. Eng. Pat. 9194, May 8, 1893.

THE addition of one-half to one per cent. of borax or other borate to lead sulphate ground in oil, is claimed to enhance the value of the latter as a pigment, rivalling white lead both in drying and covering power.—F. H. L.

*An Improved Paint or Paint-like Composition.* W. C. Nangle, London. Eng. Pat. 11,727, June 14, 1893.

GAS-TAR, cow-dung, oak-bark, sulphur, rosia, mica, and "euphorbium" are all boiled up together, ground and thinned down. The resulting mixture is claimed to be "heat resisting, anti-corrosive, and anti-fouling," as a paint or composition.—F. H. L.

*Improvements in the Manufacture of Lead Pigments.* H. R. Gregory, Wandsworth. Eng. Pat. 13,558, July 12, 1893.

LEAD sulphate—the volatilised variety by preference—is boiled with about 10 per cent. of caustic soda until the liquid is no longer alkaline to test paper, and the resulting mixture washed and dried. As an oil pigment, it is claimed to have the body and opacity of Dutch white lead.—F. H. L.

*Improvements in Compositions or Paints for Preserving Ships' Bottoms and other Structures.* J. W. and S. J. Blake, Gosport. Eng. Pat. 1301, January 20, 1894.

THIS composition contains *inter alia* barytes, quassia, mercuric chloride, and arsenic; copper sulphate being added when it is to be used on wood.—F. H. L.

*An Improved Preparation of Colouring Matters for Dyeing, Painting, Drawing, and other Purposes.* E. Nienstaedt, L. Goldmark, and C. Herrmann, New York, U.S.A. Eng. Pat. 5078, March 10, 1894.

STICKS of wood, glass, or metal, about the size of ordinary matches, are dipped in a strong solution of any dye thickened with dextrin, &c., so as to become coated therewith. They are dried, and for use are simply immersed in the desired solvent. In the case of insoluble colours, the sticks are coated as before, and in use are rubbed down in the water in the usual manner.—F. H. L.

#### (B).—VARNISHES, &c.

##### PATENT.

*A New Material for Making Varnish, Cement, Waterproofing Material, and the like.* J. Renison, Glasgow. Eng. Pat. 9930, May 18, 1893.

THIS consists of the substance known as "Albane," which is produced by the spontaneous or artificial oxidation of gutta-percha, dissolved in a suitable solvent.—F. H. L.

## (C).—INDIA-RUBBER, &amp;c.

## PATENT.

*Improvements in Treating or Preparing Gutta-Percha and Rubber for Various Purposes.* R. Hutchinson, Cowlaire. Eng. Pat. 12,266, June 22, 1893.

This is a simplification of the process described in Eng. Pat. 17,739, 1892 (this Journal, 1893, 936), the inventor having found that by boiling together the gutta-percha or rubber with wool-grease and carbonated or caustic alkali, the former is able to remove the "lanichol" (lanolin) from the grease, and that therefore it is unnecessary to extract the lanolin in a separate operation as previously carried out.—F. H. L.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

*Extraction of Tanning Materials.* Schroeder and A. Bartel. Dingler's Polyt. J. 1894, 291, 259—263.

The authors have already shown (Dingler's Polyt. J. 239, 1893) that by the use of an appropriate quantity of water

neatly all the tannin in a tan-stuff can be extracted by a few hours' digestion, and that a prolonged treatment extracts little more tannin, but deteriorates the quality of the extract by rendering soluble much of the not tannin which would otherwise remain unattacked. The method adopted was to extract the finely divided tan-stuff with 1 litre of boiling water for two hours in an extraction apparatus and to follow this by a second and third extraction with 1 litre of boiling water, each extending over 18 hours.

In the new series of experiments here recounted an amount of water relatively greater than that previously used, and three periods of two hours each, were employed. Each extraction was made with 1 litre of water, but the weight of tan-stuff extracted was diminished, in the case of quebracho from 20 to 10 grms., in the case of oakwood from 30 to 25 grms., and in the case of valonia from 10 to 7 grms.; 10 grms. respectively of myrabolans and sumach, and 20 grms. respectively of oak bark and pine bark were used as before.

The following table shows the results obtained; the rows numbered I. indicate the composition of the bark as determined by the extract made in the first two hours with the first litre of water; the rows IIa. show the composition of the extract made in the second two hours with the second litre of water, the rows IIIa. giving the same values for the third extraction; the rows IIb. and IIIb. show the composition of the bark as determined by the total extraction made by 2 litres in four hours and 3 litres in six hours respectively:—

Material.	Water.	Tannin.	Not Tannin.	Extract Ash.	Insoluble Matter.	Total Extract.	Proportion of not Tannin to 100 of Tannin.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
<b>Oak bark:</b>							
I.....	13.00	11.52	6.22	0.97	68.29	18.71	54.0
IIa.....	..	0.58	0.09	0.00	..	..	..
IIb.....	13.00	12.10	6.31	1.06	67.53	19.47	52.1
IIIa.....	..	0.37	0.16	0.06	..	..	..
IIIb.....	13.00	12.47	6.47	1.12	66.94	20.05	52.1
<b>Pine bark:</b>							
I.....	14.50	13.13	9.22	0.75	62.40	23.10	70.2
IIa.....	..	0.73	0.52	0.06	..	..	..
IIb.....	14.50	13.86	9.74	0.81	61.09	24.41	70.3
IIIa.....	..	0.38	0.56	0.06	..	..	..
IIIb.....	14.50	14.24	10.30	0.87	60.09	25.41	72.3
<b>Valonia:</b>							
I.....	14.50	28.26	8.21	2.24	46.74	38.76	20.2
IIa.....	..	1.56	0.19	0.07	..	..	..
IIb.....	14.50	29.82	8.45	2.31	44.92	40.58	28.3
IIIa.....	..	0.92	..	0.06	..	..	..
IIIb.....	14.50	30.74	8.45	2.37	43.94	41.56	27.5
<b>Myrabolans:</b>							
I.....	13.00	35.44	15.28	2.54	33.74	53.26	43.1
IIa.....	..	0.39	0.11	0.11	..	..	..
IIb.....	13.00	35.83	15.39	2.65	33.13	53.87	42.9
IIIa.....	..	0.22	..	0.04	..	..	..
IIIb.....	13.00	36.05	15.39	2.69	32.87	54.13	42.7
<b>Sumach:</b>							
I.....	12.00	21.39	12.21	3.52	50.88	37.12	57.1
IIa.....	..	1.09	0.27	0.32	..	..	..
IIb.....	12.00	22.48	12.48	3.84	49.20	38.80	55.5
IIIa.....	..	0.55	..	0.18	..	..	..
IIIb.....	12.00	23.03	12.48	4.02	48.47	39.53	54.2

Material.	Water.	Tannin.	Not Tannin.	Extract Ash.	Insoluble Matter.	Total Extract.	Proportion of not Tannin to 100 of Tannin.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Quebracho:							
I. ....	14.50	22.46	1.71	0.28	61.05	24.45	7.6
IIa. ....	..	1.56	..	0.05	..	..	..
IIb. ....	14.50	24.02	1.71	0.33	53.44	26.06	7.1
IIIa. ....	..	0.97	0.05	0.13	..	..	..
IIIb. ....	14.50	24.03	1.76	0.46	58.29	27.21	7.0
Oak-wood:							
I. ....	13.00	7.68	1.91	0.27	77.14	9.86	24.9
IIa. ....	..	0.90	0.08	0.03	..	..	..
IIb. ....	13.00	8.58	1.90	0.30	76.13	10.87	23.2
IIIa. ....	..	0.37	0.02	0.01	..	..	..
IIIb. ....	13.00	8.95	2.01	0.31	75.73	11.27	22.5

A comparison of these results with those obtained in the previous experiments, shows that it is the duration of the extraction process which is responsible for the high proportion of not-tannins found in those experiments; thus, if the quantity of not-tannins in the first two hours' extraction be termed 100, the proportion of not-tannins found at the end of 6 hours' and of 98 hours' extraction becomes:—

	Six Hours.	Ninety-eight Hours.
Oak-bark.....	109	243
Pine-bark .....	112	228
Valonia .....	102	206
Myrabolans .....	101	136
Sumach .....	102	165
Quebracho .....	103	268
Oak-wood .....	105	420

It will also be noted that the quantity of water used has little influence on the proportion of not-tannins extracted.

Prolonged digestion does not appreciably increase the percentage of tannin extracted, so that it may be generally stated that the most profitable method of making extracts is to digest the tan-stuff with a sufficient quantity of water for a short period.

The extraction of a tanning material for analysis is, in the author's experience, best effected by allowing the weighed quantity of powdered material to soak in water in the extraction flask for half a day, and then to complete the leaching with one litre of boiling water in two hours. The size of the flask is a detail to be considered; it should be within the limits of 100—200 cc. capacity when 7—20 grms. of tan-stuff are to be extracted. Although the extraction of the tannin is not complete when the above directions are followed, yet what is more important is secured, namely, constancy of results. In practice it matters but little whether the percentages returned are too high or too low, provided they are constant among themselves.—A. G. B.

*Canaigre as a Substitute for Barks in Tanning.* United States Consular Reports, 1894, 44, 679.

Dry canaigre-root (this Journal, 1892, 622) contains from 20—40 per cent. of tannin according to age, the two-year-old roots containing more than the one-year-old. The fresh root contains about 66 per cent. of water. 112 lb. of the dry roots yield 45 lb. of extract containing 66 per cent. of tannin; the residue is said to be equal in value to wheat-

straw as cattle-food. The following analyses are by Schroeder:—

	I.	II.
Water.....	11.00	14.69
Tannin.....	34.91	27.72
Not tannins.....	18.14	9.40
Extract ash .....	1.97	2.13
Insoluble matter.....	33.98	46.06
	100.00	100.00

Canaigre-tanned leather is soft, bright, and orange-coloured, and is recommended for uppers, saddlery, and fancy leather. There is a good demand for canaigre, which may soon replace gambier.—A. G. B.

#### *The Origin of the Acid Fermentation in Tanning Liquors.*

F. H. Haeflein. Dingler's Polyt. J. 1894, 291, 186—190 and 209—214.

THESE investigations were made with an extract of pine-bark (20 grms. of bark per litre). It was found that the production of acid in such an extract is due to the growth of an organised ferment, which was cultivated and isolated. The fermentation follows the usual course, gradually waxing and waning. A detailed account of the experiments conducted for determining what constituent of the bark is converted into the acid, the nature of the gases evolved, and the influence of temperature and light, is given. The following conclusions are drawn:—

(1.) The formation of acid in a pine-bark liquor is due to a fermentation.

(2.) The ferment is a bacterium, which may be termed *bacillus corticalis*; it is not identical with the *bacillus lacti*, although it ferments milk-sugar and glucose, with the production of a soluble acid and of the same gases which it evolves from pine-bark liquor.

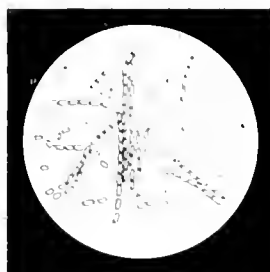
The microscopical appearance of the bacillus is shown in the figure (amplification not stated).

(3.) The constituents of the bark which are fermented are the saccharine substances (Fehling reducing compounds). The fermentation does not occur in pure tannin solution.

(4.) The products of the fermentation are a soluble acid and a mixture of much hydrogen and little carbon dioxide. In one experiment the evolved gas contained 94.6 per cent. of hydrogen and 5.4 per cent. of carbon dioxide; no estimation of the carbon dioxide remaining in solution is quoted.

(5.) The fermentation will not take place below 6° C.; the most favourable temperature is between 30° and 40° C.

(6.) Light hastens the fermentation.



A similar fermentation was observed in extracts of oak-bark, mimosa, and sumach, but not in extracts of quebracho and myrabolans.—A. G. B.

*Spray-tanning.* P. F. Reinsch. Dingler's Polyt. J. 1894, 292, 21—23.

Of the many attempts which have been made to improve the tanning process, having for their object a more rapid output and a more complete utilisation of the tanning material, none has proceeded on any plan other than that of immersing the hide in the tanning liquor.

"Spray-tanning" (*Bieselgerlung*) consists in causing the tanning liquor to flow down each side of the vertically suspended hide, stretched by its own weight; the liquor is received in a tank and pumped back into the reservoir from which it flows.

The plant necessary for the process consists of a number of frames, from which the hides are suspended by hooks, a reservoir supplying pipes which carry equidistant jets, so arranged that the liquor may flow on to each side of the hide, and a tank to receive the partly-spent liquor. To secure equable tanning the liquor must be constant in rate of flow and in strength; the first desideratum is secured by keeping a constant level in the reservoir, the second by adding strong liquor to maintain the specific gravity of that pumped back into the reservoir at the same figure.

The duration of the tanning is alleged to be reduced by this process, for heavy leather, to one-eighth or even one-tenth of the time expended in the usual layer process, and it is claimed that the product is equal in quality to that turned out by the old method.

It will be observed that in spray-tanning both hide and liquor are exposed to the air. Whilst this is of no moment when the process is used for mineral tannage, it is not permissible when tannin itself is used, for the tannin speedily suffers oxidation and is lost so far as the tanner is concerned. This difficulty is by no means insurmountable, inasmuch as the maintenance of an atmosphere of carbon dioxide around the hides is not difficult; all that is required is a box which shall be airtight at the bottom and sides, with a pipe for admission of the gas into the lower part; according to the author the gas will remain unmixed with air for weeks, even if the box be not covered.

A noteworthy economy effected by spray-tanning is that handling and plunging are not necessary; another is the complete utilisation of the tannin, which is most effectively extracted from each drop of liquor as it trickles over the hide. It is also in favour of the new process that it requires a smaller ground space and a less expensive installation than are required for pit-tanning. If a comparison of the two processes be made on the basis of the space necessary for laying away 600 hides, it will be found that the space for pit-tanning will be  $\frac{1000}{37}$  times that necessary for spray-tanning, or  $\frac{1000}{9}$  times if the latter process be conducted in two tiers. The cost of 28 pits is given as 4,480 marks, that of an equivalent spray apparatus as 2,000 to 3,000 marks.

Spray-tanning was specially devised for the author's ferric oxychloride and salt method of tanning; the following figures are claimed to be representative:—

Time occupied in tanning by

	Tanning in Pits.	Ferric Oxychloride in Pits.	Ferric Oxychloride in Sprays.
	Days.	Days.	Days.
Light leather .....	50	11	7
Heavy leather .....	140	28	24

#### COST OF TANNING MATERIAL FOR 1 KILO. OF RAW HIDE.

	Pfennige.
Bark .....	107
Chromium compounds .....	15
Ferric oxychloride and salt .....	5

#### LABOUR BILL FOR 100 KILOS. OF RAW HIDE.

	Marks.
Bark tannage .....	21—10
Chromotannage .....	20
Ferric oxychloride spray-tannage .....	8

Iron-tanned heavy leather is asserted to compare favourably with the best bark-tanned heavy leather. The grain is particularly durable, and the resistance to water is considerably greater than that of bark-tanned leather. For belts the iron-tanned leather is more valuable than bark-tanned, as it does not deteriorate so rapidly when exposed to dry or moist heat.—A. G. B.

#### PATENTS.

*Improvements in the Treatment of Loom pickers, Leather, and other similar Articles with Fatty Matters, and Apparatus for Effecting the same.* R. T. Gillibrand, Darwen, Lancaster. Eng. Pat. 11,248, June 8, 1893.

The various articles named in the title are enclosed in a vessel, which is then made vacuum; the oil or fat is allowed to flow in and pressure is applied.—A. G. B.

*Improvements in Tanning.* C. Heinzerling, Frankfurt-on-Main, Germany. Eng. Pat. 12,849, June 30, 1893.

The following chromium compounds are applicable in this process:—Chromic chromate,  $\text{Cr}_2(\text{CrO}_4)_3$ , prepared by dissolving chromic hydroxide (1 mol.) in chromic acid (3 mols.); "bichromate monosulphate of chromium,"  $\text{Cr}_2(\text{CrO}_4)_2\text{SO}_4$ , made by dissolving chromic hydroxide (1 mol.) in chromic acid (2 mols.) and sulphuric acid (1 mol.); "basic chromate sulphate of chromium,"  $\text{Cr}_2\text{CrO}_4\text{SO}_4$ , made like the preceding, but in the proportion of 1 mol. : 1 mol. : 1 mol.; "chromate disulphate of chromium,"  $\text{Cr}_2\text{CrO}_4(\text{SO}_4)_2$ , formed by dissolving chromic oxide (1 mol.) in sulphuric acid (2 mols.) and chromic acid (1 mol.); aluminium chromate, made by dissolving aluminium hydroxide in chromic acid; chromic thiosulphate, from the decomposition of chromium salts by thiosulphates; chromic sulphite, made either by decomposing chromium salts with neutral or acid sulphites, or by saturating a solution of sulphurous acid with chromic hydroxide; chromous chloride and chromous sulphate, made by reduction of the corresponding chromic salts with zinc dust or iron filings; chromium thiocyanate, formed by decomposing barium thiocyanate with chromic sulphate.

The following are also used:—Ferrous, aluminium, zinc and manganese thiosulphate, formed by decomposing barium thiosulphate with the sulphates of these metals; ferrous, zinc, manganese, and sodium hydrosulphite (hyposulphite), formed by the action of the metal on sulphurous acid or sodium bisulphite.

The hides, prepared as usual, are first laid in a dilute solution ( $\frac{1}{2}$  per cent.) of the above-mentioned compounds of chromic acid and chromic oxide, and then the solution is successively strengthened (up to 10–12 per cent. or even 15–20 per cent. for heavy leather). The duration of the process may vary from 8–14 days for uppers to 5–6 weeks for sole-leather. Uppers are washed after the tanning, but sole-leather should be dried without washing. Soft, pliable upper leather is advantageously tanned first in compounds of chromic acid and chromic oxide, and then in a solution of chromium thiosulphate or sulphite, for which the thiosulphate or hydrosulphites mentioned above may be substituted. When the last-named salts are used the metals contained in them are deposited as oxides in the fibre of the hide, and thus both a tanning and a colouring effect may be produced. Eleven claims cover the above points.—A. G. B.

*Improvements in Apparatus for Extracting Fat and Glue.* E. J. Machalski, Brooklyn, New York, U.S.A. Eng. Pat. 5821, March 20, 1894.

See under XII., page .

## XV.—MANURES, Etc.

*Influence of Potassium Salts on Nitrification.* J. Dumont and J. Crochetelle. Comptes rend. 117, 604–606.

In a previous communication (Comptes rend. 117, 670) the authors showed the favourable effect exercised by various potassic manures on nitrification in cleared lands rich in humus and lime. They have now continued their experiments on soils rich in humus and silicates, but poor in lime. Here, too, the addition of amounts of potassium carbonate up to 4.5 per cent. increased the rate and amount of nitrification, but, on the other hand, potassium sulphate had little or no influence. The following table gives the results found:—

NITRIC ACID FORMED IN 20 DAYS IN 1,000 GRMS. OF HEATH SOIL.

K <sub>2</sub> CO <sub>3</sub> added.	Milligrams of Nitric Acid.	K <sub>2</sub> SO <sub>4</sub> added.	Milligrams of Nitric Acid.
Per Cent.		Per Cent.	
0	24	0	25
0.1	47	0.5	54
0.2	65	1.0	19
1.0	94	1.5	24
1.5	156	2.0	19
2.0	188	2.5	20
2.5	238	3.0	27
3.0	313	3.5	25
3.5	282	4.0	20
4.0	384	4.5	20
4.5	438	5.0	18
5.0	467	..	..
6.0	375	..	..

If, when potassium sulphate is used, chalk is also added, the results are completely changed, nitrification being much increased. Thus, with 2.5 grms. of chalk added, 0.5 per cent. of potassium sulphate increased the nitrification (which, without any manure, was 25 mgrms.) to 75 mgrms., 2 per cent. to 129 mgrms., and 4 per cent. to 163 mgrms.

There can be little doubt that the effect of the potassium sulphate in the presence of calcium carbonate, either natural or added, is due to the formation of potassium carbonate by double decomposition, and not to the direct action of the sulphate.

The authors' results show that in the cultivation of soils rich in humus, nitrification is much assisted by potassium carbonate (in the form of non-lixiviated ashes, for instance) or of potassium sulphate, but that where the latter substance is used, chalk must be added if the soil is poor therein. The quantities of manurial material desirable are, of course, much smaller than the maximum proportions employed by the authors.—L. T. T.

*Influence of Iron on the Vegetation of Barley.* P. Petit. Comptes rend. 117, 1105–1107.

BARLEY was grown in sand (boiled with hydrochloric acid till all iron was dissolved out), part being watered with ferrous sulphate solution and part with ferric sulphate solution, potassium nitrate, calcium sulphate, and potassium phosphate being also supplied. The ferric sulphate was found to act as a poison; but the ferrous sulphate had no such effect, being absorbed to such an extent that the ashes of the stems and leaves contained 10 times as much iron as was present in plants grown similarly, but not treated at all with ferruginous substances; a slight increment in nitrogen was also produced. Apparently, therefore, the general opinion is incorrect that ferrous salts lose their noxious qualities in soils when they become peroxidised.

—C. R. A. W.

*The Utilisation of Blast-Furnace Slag.* A. D. Elbers. Eng. and Mining J. 1894, 392.

See under X., page 643.

## PATENTS.

*Improvements in Apparatus for Separating, Classifying, and Purifying Minerals and other Materials.* A. F. Beyer and A. G. Beyer. Paris. Eng. Pat. 3678, February 20, 1894.

See under I., page 623.

*A Liquid for Use as a Fertiliser and Insecticide, and for other Agricultural and Horticultural Purposes, applicable also as a Deodoriser and Disinfectant.* S. Elliott, Newbury, Berks. Eng. Pat. 8910, April 7, 1894.

THE soluble and solid constituents of the smoke from coal or wood are retained by water in the manner already described (this Journal, 1890, 273). The liquid so obtained is available for the purposes mentioned above. Instead of using the smoke from ordinary furnaces, a special fire may be employed to produce smoke and gases from properly selected materials.—N. H. J. M.

## XVI.—SUGAR, STARCH, GUM, Etc.

*The so-called "Turkish Honey."* A. Fajano. Chem. Zeit. 1893, 17, 1826.

"TURKISH honey" is an important article of commerce in Austria. It has a white colour, in a fresh state is dry, hard, and brittle, but deliquesces on standing. It has a pleasant taste, but leaves behind it a bitter and slightly burnt flavour, which is attributable to alum.

Its analysis gave the following result:—Water, 7.97 per cent.; invert sugar, 56.78; cane sugar, 31.02; ash, 0.31; insoluble in 80 per cent. alcohol, 3.92, which was composed of gummy matter, 68.09 per cent.; alum, 27.80; calcium sulphate, 4.08.

It is prepared by partially inverting cane sugar with acid at 80°–90° C., neutralising the product, and adding a decoction of the root of *Saponaria officinalis*. The mixture is heated for some time, with constant stirring, when a slight caramelisation takes place. Natural honey is then added and the product cooled. The addition of the decoction of *Saponaria* alters the consistency of the finished preparation.—J. G. W.

*Advantages of Heating Diffusion Juice with the Steam arising from another Diffusion.* M. Melichar. Zeit. Zuckind. Bohm. 1894, 18, 301–306.

The author calculates that as a direct source of heat 51.4 kilos. of steam are required per 100 kilos. of beet-roots, from the diffusion to the boiling process; whereas, when steam arising from the liquor is utilised, 45 kilos. of steam are needed. This saving of 6.4 kilos. of steam per 100 kilos. of roots is reduced to 5 kilos. in actual practice. The author therefore considers that the gain is not sufficient to warrant the use of the process, when the losses due to decomposition of the sugar and to the reduction in value of the products due to their increased darkness, are taken into account.—A. R. L.

*Note on the Solution of Indian Gums by Means of Hydrogen Peroxide.* H. Koechlin. Bull. Soc. Ind. Mulhouse, 1894, 36–37.

By heating together 250 grms. of crushed Indian gum, 1,000 cc. of water, and 50 cc. of hydrogen peroxide (12 vol.) for two hours to 80° C., a solution is obtained, which, however, turns wool yellow on steaming. This would appear to be due to a loss of oxygen, and can be corrected by adding bisulphite to the gum solution. Woollen fabrics are turned yellow on steaming in consequence of the oxidation of the sulphite compound, and this is more noticeable in Mather and Platt's large continuous steamer than in ordinary vats, owing to the quantity of air present in the apparatus. It can, however, be remedied by passing through sodium bisulphite. The author remarks that insoluble gums contain a product soluble in water, which is more oxygenated than the rest, and he proceeds to point out an analogy which exists between gums and several vegetable products containing simultaneously two or more substances having different amounts of oxygen. Of these he mentions madder, which contains alizarin and purpurin, logwood and Brazil-wood contain hematin and an insoluble resin, cinchona yields quinine and cinchonine, and nuxvomica and opium also contain different products. In cochineal, according to Schützenberger, there are two substances containing different quantities of oxygen, one gives a carmine on alumina and the other a ponceau on tin. The author also mentions as one of his analogies that blood contains white and red corpuscles. The note concludes with a recipe for a thickening from linseed prepared by boiling for one hour and then filtering 500 grms. of linseed, 1,000 cc. of water, 250 grms. of acetic acid, and 8–10 cc. of hydrogen peroxide (12 vols.). The quantity of hydrogen peroxide depends upon the linseed, and it is important to determine the exact quantity, as an excess causes a complete solution. The seed, after having been treated as described, is dried and the oil extracted in the usual manner.—T. A. L.

*Determination of the Crystallisable Sugar in the Beet.* H. Pellet. J. Amer. Chem. Soc. 16 [4], 266.

See under XXIII., page 669.

#### PATENT.

*Improvements in Apparatus for Creating a Vacuum in Vacuum Pans.* P. Pié, Lima, in the Republic of Peru. Eng. Pat. 4906, March 8, 1894.

See under I., page 624.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*Occurrence of Raffinose in Wheat Germ.* F. Schulze and S. Frankfurt. Ber. 27, 1894, 64–65.

The precipitate obtained by adding strontium hydroxide to the alcoholic extract of wheat germ, in the manner described (this Journal, 1894, 533), is decomposed with carbonic anhydride and the filtrate evaporated to a syrup, the latter being treated with successive small portions of boiling alcohol to remove the saccharose. The residual syrup is dissolved in water and absolute alcohol added to the concentrated aqueous solution, when raffinose is precipitated; it is purified by repeating the last-described operation several times. Richardson and Crumpton (Ber. 19, 1180) conjectured the presence of raffinose in wheat germ, but did not succeed in separating the sugar.—A. R. L.

*The Manufacture of Prepared Cereals.* J. Barrington White. Trans. Inst. of Brewing, 7 [2], 143–156.

The author is strongly of opinion that the prepared form of raw (unmalted) grain is the only one that can be satisfactorily employed by the brewer. The use of untreated grain in the brewery involves the erection of costly plant ("converters"), in which the preliminary gelatinisation of the "grits" is effected. Serious stoppages in the working may, and do, arise owing to a breakdown of the raking machinery, clogging of the pipes and valves, &c., due to the stiffness of the mash. The author states that 50 per cent. of prepared grain may be employed without difficulty in point of drainage, &c. Of the various forms of prepared grain the author prefers the "golden-flaked malt" manufactured from yellow European maize, since it gives "greater palate, fullness, brilliancy, and stability" than either flaked rice or white American maize (this Journal, 1894, 410). The average composition of golden-flaked maize is as follows:—Starch, 83.67; cellulose, 1.64; fat, 0.89; albuminoids, 8.27; ash, 0.45; moisture, 4.37 per cent.; and extract, 109.21 lb. per qr. of 336 lb. Of the total albuminoids, only a small amount is soluble in water.

Subsequently the author describes the manner of manipulating flaked grain in practice, and considers its influence on the composition of the wort. As regards the latter point the changes involved consist essentially in an increase in the percentage of dextrin and malto-dextrin, and a reduction in the amount of soluble albuminoids (as compared with all malt wort). To these facts the author attributes the increased fullness, conditioning power, and stability obtained by the use of flaked malt.

The frequent instability of pale all-malt beers, arising from the enforced use of pale malt—often under-reared and unsound—may be avoided by employing a darker, better-cured malt in conjunction with flaked grain, which yields a practically colourless extract.

In conclusion the author enters into some details concerning the extra profit obtainable by the use of prepared grain, and states that the material may be successfully employed in the brewing of every class of beer.

In discussion it was pointed out that the flavour and aroma of barley-malt were essentially due to the process of kilning to which it was subjected, and that in this respect flaked malt stood on the same level, since it was submitted to a similar torrefying process. At the least, flaked malt must be superior to any form of untreated grain. In regard to extract, the author said that failure to obtain full yield was quite contrary to his general experience; but that in the case of grits, the large percentage of water (13–14 per cent.) they contained would explain deficiency. A question as to the suitability of the grains for cattle-feeding was answered in the affirmative.—H. T. P.

*The Capacity of Yeast to Live in Media containing Hydrofluoric Acid.* E. Sarel. Comptes rend. 113 (1894), 253–255.

The author's experiments lead him to the following conclusions:—A yeast which has become injured to a medium

rich in hydrofluoric acid, yields, when cultivated in a medium containing less hydrofluoric acid, the more active cells, according as the original culture-medium contained more hydrofluoric acid. A yeast having become once inoculated to a certain quantity of hydrofluoric acid, retains this property. The author has obtained yeast which withstood finally 1 gram of hydrogen fluoride per litre, or six times as much as at the commencement.—A. R. L.

*Contribution to the Study of Yeasts.* P. Hautefeuille and A. Perrey. Comptes rend. 118, 589—591.

THE authors have studied the yeasts occurring during the fermentation of the various wines of Nuits and Beaune. These yeasts, with few exceptions, may be classed in three groups—(1) apiculate yeasts, occurring at the beginning of fermentation and often carrying on the fermentation to the end; (2) ellipsoidal yeasts, which appear after the fermentation has progressed for some time, are very active and rapidly (*i.e.*, in about 48 hours) entirely replace those of group 1; (3) ellipsoidal yeasts, occurring in neutral or but slightly acid must, which resemble surface yeasts in behaviour and vary in colour from rose to dark wine-red. The yeasts of this last class appear very active, but except in one instance were always accompanied by those of class 1 or 2. They are noticeable by the presence, in the vitreous protoplasma, of a spherical and highly refractive globule, which, small at first, rapidly increases and is still plainly visible in the old and granular cells. If the fresh cell is placed on a piece of moist chalk this globule increases in size till it nearly fills the cell, becomes covered with a membrane (often coloured), and the protoplasma gradually becomes differentiated into numerous minute spores, which are eventually set free by the rupture of the membrane. In nutritive material multiplication of these yeast cells generally takes place by budding, but in one case the authors observed reproduction by budding and by spores taking place side by side.—L. T. T.

*On the "Cassage" of Wine.* A. Bonffard. Comptes rend. 1894, 118, 827—829.

THE author has investigated the cause of this malady, which exhibited itself among wines from the vintage of 1893, displaying the following symptoms:—On exposure to the air the previously clear wine became covered with a thin, iridescent film of colouring matter which gradually increased in amount and finally fell to the bottom as a reddish-brown precipitate, leaving the wine considerably lighter in colour and of a yellowish hue.

Pasteurisation and the addition of sulphurous acid are effectual means of preventing this malady, pasteurisation effecting even the re-solution of the precipitate and improving the colour of a wine which was already affected.

The preventative action of pasteurisation would point to the disease being the result of bacterial action, but its action upon a wine already affected would indicate that the action was a chemical one upon the colouring matter of the wine. This latter is supported by the facts that filtration through a Chamberland filter, and treatment with sulphurous acid or mercuric chloride do not prevent the disease.

Sulphurous acid added to the extent of 0.03 gram per litre prevents "cassage" but its action is explained by its avidity for oxygen.

The author is of opinion that the prevalence of this disease among wines of the 1893 vintage is due to the precocious maturity of the grapes produced by the extreme meteorological conditions which obtained then, whereby the colouring matter of the grape was rendered particularly liable to precipitation.—J. G. W.

*The Chemical Action of Abrastol (Calcium Naphthol Sulphonate) on Wine.* Scheurer-Kestner. Comptes rend. 1894, 118, 74—76.

ABRASTOL (or asaprol), the calcium salt of  $\beta$ -naphthol sulphonic acid (this Journal, 1894, 534 and 548), is being employed as a preservative in the manufacture of wine, and

is replacing "plastering." The absence of any unfavourable physiological action and the small quantity requisite for the purpose are greatly in its favour. It has, however, been asserted that abrastol in the presence of the principal constituent of wine, gives rise to the production of free sulphuric acid. This statement the author, from experiments made with cream of tartar and with wine, proves to be erroneous, the products of the interaction of cream of tartar and calcium naphthol sulphonate being calcium tartrate, potassium naphthol sulphonate, and naphthol sulphate.—J. G. W.

*The Flavour of Whisky, as Influenced by the Materials used and by the Processes of Manufacture.* J. A. Nettleton. Trans. Inst. Brewing, 7, 167—195.

FOR the purposes of this paper the author classifies whiskies into (1) pot-still whiskies and (2) patent-still whiskies.

*Patent-still Whiskies.*—In patent-still whiskies only very minute quantities of flavouring constituents are present. The higher alcohols amount to only one-fourth to one-fifth, the compound ethers to about one-half to one-third of the average quantities present in pot-still whiskies. Furfural is absent and the amount of free acid is scarcely appreciable. Of the higher alcohols present, the larger portion is propyl alcohol, the one most closely resembling ordinary alcohol. In the patent stills the separation of the constituents of the wort is so complete that in one distillation the spirit leaves the lowest spirit plate almost constantly at a strength of 67.68 over proof, or containing about 93 per cent. of absolute alcohol. The spirits obtained with patent stills are very pure, being, for instance, well below the Swiss or Ruse's standard, which fixes 0.12 on the proof gallon as the maximum percentage of secondary constituents allowable, but they are comparatively flavourless. Large quantities of patent-still spirit are used for blending, and in many patent-still distilleries German yeast is the main product. The flavouring or secondary constituents are, however, present in the original wort, and during distillation collect in large quantities in the various "feints" vessels. The author suggests that with care and judgment, some of these flavouring constituents could with advantage be diverted into the spirit receiver, and a superior flavoured whisky be thus obtained.

*Pot-still Whiskies.*—These whiskies embrace (a) all-malt whiskies, usually styled Scotch, and (b) malt and grain whiskies, usually styled Irish, though some of the Irish whiskies are all-malt whiskies. The Scotch whiskies may further be subdivided into (1) Highland, (2) Lowland, and (3) intermediate whiskies. The wort of Highland whiskies is prepared very similarly to a brewer's wort, but at a lower initial and final temperature, and attenuates from a gravity of 1,050 down to that of water. There are two main distillations, namely, of the wash into "low wines," followed by the collection of "fine spirit." The collected spirit has an average strength of 15 per cent. over proof. The malt for Lowland whisky is usually a mixture of foreign and home-grown barley, dried at a moderate or even low heat. If peat is used it is only sparingly, the malt having little of the peaty flavour. The wort is made as in the Highlands, but the attenuation is more rapid, being effected in 48 hours. There are three main distillations—the wash into low wines; these into weak and strong feints; and the strong feints into spirits and feints. The spirits are collected at from 40° to 60° over proof. In the "intermediate" whiskies (amongst which some of the finest may be classed), pure water and superior, well-cured malt entirely free from peaty flavours are used. A mixture of double and treble distilling is often employed. In the Irish whiskies the waters used vary greatly, some being stream or soft, others well or hard water. The mashing material consists of a mixture of moderately dried malt and a large quantity of kilned grain—barley and oats. Lower initial heats are used in mashing; the wort collected ranges in gravity from 1,040 to 1,050 and is attenuated down to 1,000. The stills are heated by furnaces, and there are three main distillations, the spirit being collected at 45° to 50° over proof.

In Irish distilleries where raw grain is used kilning is often dispensed with if the grain is dry enough for grinding.

Thorough kilning has, however, undoubtedly beneficial effects. It diminishes mould, ensures a sounder wort, and one less susceptible to bacterial fermentation, and therefore tends to reduce the amount of secondary products. Highly-dried malts are found to give worts which, after attenuation, are less acid and from which the first distillate is distinctly purer. Even with maize in patent still distilleries kilning is advantageous.

Where raw grain is used this might be advantageously mashed at 170°–180°, the mash temperature lowered to 140°, and the fine malt then added.

The main principles governing the quantity and quality of the flavourings are: the quality of the extract present in the wort, the type or species of yeast used, the temperatures employed throughout the process, and the duration of fermentation. The finer shades of flavour possessed by the original mashing materials are distinctly discernible in the extract, and though they blend during fermentation with other flavours not previously existent, they yet impart a distinct character to the product. The various species of yeast each tend to create certain and peculiar flavouring bodies. High fermentation temperatures produce rapid and exhaustive attenuation, but are also responsible for the powerful bacterial fermentation which accompanies the main process, and which contributes more than anything else to the production both of desirable and undesirable flavours. Prolongation of the time of fermentation at high temperatures causes a rapid increase of acidity at the expense of the alcohols, and a simultaneous production of several distinctive flavours.

The patent or Coffey still eliminates alike good and bad flavours. Pot-stills only partially eliminate the secondary products, this elimination varying greatly in degree at different distilleries. There is still amongst distillers a preference for furnace-heated stills. The method of heating governs the flavour of the distillate in several ways. In steam-heated stills no new flavours are likely to be created, and greater control is possible over rate of distillation and separation of fractions. The furnaces are more likely to cook the wash and partly burn the sedimentary matter. The presence of furfural in pot-still whisky is attributed to this action, together with the high drying of the malt. The higher percentage of this alcohol in Scotch than in Irish whiskies is due to the higher kiln-drying and the double instead of treble rectification. The author believes that fine flavours may be obtained by employing a furnace for the wash-stills, but steam-jackets or coils for the low wines and feints still.

The esters, to which undoubtedly the special flavour and aroma are largely due, are chiefly found in the low wines, feints, and foreshots. Of the low boiling esters, ethylic, propylic, and isopropylic acetates have been recognised in whiskies. There is some evidence that in all-malt whisky there is an excess of esters over the quantities found in malt and grain whiskey. In the collection of the distillates cloth-filtering is advisable. The cloth filters are found to be covered with a mineral and oily deposit, and the oils so retained are the heaviest and probably least desirable of the esters present.

The processes taking place during maturation of spirit are still far from fully known. Bell's analyses, whilst showing a reduction of furfural, seem against the generally received theory of the oxidation of the higher alcohols, as he found severely any reduction of these in the fully-matured spirit. Allen's analyses, on the other hand, tend to show a consistent reduction of the higher alcohols during ageing.

In the discussion on the paper Dr. Sykes pointed out that in choosing the mashing temperature the distiller had to keep in view the preservation intact of the diastase in the mash; distillers, with very few exceptions, depended on brewers for their yeast. Both Bell and Allen in their analyses used modifications of Berthelot's method for estimating the esters, but Allen first removed the furfural. He believed the furfural present in spirits was derived from the gum in the mash. Lintner, jun., has shown that a gum is present in malt and barley, which on hydrolysis splits up into galactose and xylose. This latter, if distilled with

hydrochloric acid, yields about half its weight of furfural, and the small amount of acid in the mash probably acts to a limited extent in the same way. —L. T. P.

## PATENTS.

*Improved Method and Apparatus for Collecting the Carbonic Acid generated during the Fermentation of Beer or Wine.* K. Michel and M. Frank, Munich, Germany. Eng. Pat. 10,263, May 24, 1893.

FERMENTATION is effected in a tun of cylindrical or other form provided with an air-tight cover which is fitted with a pressure-gauge, the necessary taps, valves, &c., and a man-hole. In practice, in order that the quality of the beer may be unimpaired, the man-hole is left open for the first 2–3 days after pitching and the wort aerated at intervals in the usual way. When fermentation is in full swing, the man-hole is closed and the carbonic acid drawn off automatically under a pressure of one atmosphere or more. When the evolution of gas begins to sensibly slacken, the fermenting vessel is again opened and the beer finished in the usual way. The carbonic acid thus obtained is stated to require no purification before being compressed or otherwise utilised. —H. T. P.

*An Improved Method of and Means for Utilising Spent Hops for Food and Litter.* E. O. Greening, Lee; and E. W. Greening, Thames Ditton. Eng. Pat. 10,941, June 5, 1893.

THE inventors chief claim is the utilisation of spent hops as a food or litter.

Hop waste, obtained from breweries, is first dried by storing the hops in stacks, constructed with hollow centres from which the air is exhausted by an exhaust fan or similar apparatus or source of draught such as a heated chimney. When the mass is dry and cool, the seed is separated by winnowing, preferably by means of a combined sifter and wind current winnowing machine. The seeds are then cleaned from dirt and fibre, and may be sold either whole, or ground into meal, or pressed into cakes, or they may be mixed with other feeding materials. The fibre makes a good litter of considerable commercial value.

—L. de K.

*An Improved Apparatus for Airing or Rousing Brewers' Wort or other Liquids.* F. R. Stone, Liverpool. Eng. Pat. 11,936, June 17, 1893.

THE improved rouser consists of a covered spoon-shaped vessel, perforated with holes, which is attached to the discharge pipe of a suitable pump; the whole being fixed permanently, or temporarily—as may be desired—to the side of the fermenting vessel. The wort being drawn up by the pump, falls from the rouser in thin spreading streams, and is thus aerated. —H. T. P.

*Improved Method of and Apparatus for making Vinegar and Maturing Spirits.* R. H. Leaker, Bristol. Eng. Pat. 15,475, August 15, 1893.

THE object of this invention is to accelerate the process of vinegar making, and to prevent as far as possible the loss of the volatile bodies, such as aldehyde, ethers, &c., produced during acetification, on which the aroma and market value of the vinegar depend. The apparatus employed consists of a closed rectangular chamber, of which the upper portion is fitted with a series of inclined shelves or frames covered with some kind of woven material. The shelves slide in grooves, and are inserted through openings in the side of the vessel, so arranged that any one shelf may be withdrawn for cleaning purposes, &c. without disturbing the rest. The lower part of the chamber acts as reservoir for the wash. By means of a pump and sprayer the wash is continuously discharged in a fine spray on the surface of the uppermost shelf, from which it gradually percolates over and



through the other shelves, thus exposing a large surface to evaporation. An air-blower is also provided and serves to circulate filtered or unfiltered air, mixed with a portion of the vapour generated from the alcoholic wash through the apparatus. The bulk of the volatile vapour is, however, continuously drawn by suction through a condensing worm attached to the cover of the chamber, the condensed products being returned to the wash reservoir. In addition to the above, attenuating coils are provided, for the purpose of regulating the temperature of the wash; and a steam pipe passing through the bottom of the chamber, by means of which the whole apparatus may be sterilised or dried, prior to recharging. Drawings of the apparatus accompany the specification.—H. T. P.

*Improvements in the Process and Apparatus used in Brewing.* A. Zimmer, Paris, France. Eng. Pat. 23,998, December 13, 1893.

THE malt, instead of being merely crushed as ordinarily, is separated by a complete process of "high-milling" into malt-flour, and certain sub-products, such as husk, germs, &c. For brewing beer, the sub-products are mashed with water, and the wort rich in diastase thus obtained employed to saccharify the main product (the flour). The main wort, if turbid, may be centrifuged; it is then boiled with hops, cooled to 50° C. and passed through a centrifugal machine, in order to separate the suspended matters, and aerate it prior to fermentation. A detailed description accompanied by drawings, is given of an improved centrifugal machine, by using which, the violent frothing obtaining in the older forms, is avoided.—H. T. P.

*Apparatus for Distilling and Sterilising Water.* J. Nagel, Chemnitz, Saxony. Eng. Pat. 2588, February 6, 1894.

See under I., page 423.

*Improved Process for the Manufacture of Beer.* J. C. Pennington, Paterson, New Jersey, U.S.A. Eng. Pat. 3642, February 20, 1894.

THE object of the process is to brew beer absolutely free from disease germs. To this end the wort is completely sterilised by intermittent heating; a pure yeast culture used for pitching, and fermentation effected in a closed vessel provided with a safety valve, &c. Finally the beer is stored in sterilised vessels.—H. T. P.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY AND WATER PURIFICATION, DISINFECTANTS.

### (A).—CHEMISTRY OF FOODS.

*The Determination of Nitrates in Potable Water.* A. H. Gill. J. Amer. Chem. Soc. 16, 122-132.

See under XXIII., page 663.

*The Determination of Nitrates in Potable Water.* A. H. Gill. J. Amer. Chem. Soc. 16, 133.

See under XXIII., page 664.

*A Rapid and Accurate Method of Determining the Moisture and Oil in Linseed Cakes and other Feeding Stuffs.* A. P. Aitken. J. Amer. Chem. Soc. 16, 114.

See under XXIII., page 669.

*Detection of Salicylic Acid in Food.* K. P. McElroy. J. Amer. Chem. Soc. 16 [3], 198.

See under XXIII., page 670.

### PATENTS.

*Improvements in or Relating to the Preparation of Substitutes for Coffee.* H. Trillich, Munich, Germany. Eng. Pat. 8726, May 1, 1893.

THE inventor claims to have made an improvement in preparing coffee substitutes from cereals, malt, &c. by impregnating the unroasted materials with extract of coffee shells or a decoction of cocoa shells, fruit or leaves of kola nut, or leaves of tea or maté. In using barley, a very good product is obtained when 100 kilos. of the grain are treated with 500 grms. of extract of coffee shells dissolved in three litres of water. The form of the cereals may be chosen in whole grains, hulled grains or grains in a coarsely or finely disintegrated condition. Chicory, figs, pears, and leguminous matters in general, may also be used.

The impregnated material is then roasted in the usual manner. The flavour will, of course, depend on the choice or proportion of the ingredients of the extract.—L. de K.

### (B).—SANITARY CHEMISTRY AND WATER PURIFICATION.

#### PATENTS.

*A New Compound or Mixture for Purifying Sewage and Foul Water or Liquid.* G. Whalley, Chorley, Lancashire. Eng. Pat. 9359, April 28, 1894.

THE mixture consists of ferric oxide (1 part), bauxite (1 part), and brown vitriol (2 parts). It is heated with sulphuric or hydrochloric acid, a small quantity of nitric acid being added if necessary. The mixture is employed in proportion to the nature of the sewage, &c. to be purified. The effect is to cause quick precipitation of the organic matter present in the water.—N. H. J. M.

*An Improved System for the Purification and Filtration of Sewage, Foul Water from Sewers, Manufactories, and other Sources.* G. B. Sharples, Manchester. Eng. Pat. 12,265, May 5, 1894.

A SYSTEM of continuous flow precipitation tanks is employed, combined with perforated slabs carrying suitable filtering media or chemicals, or both. By means of suitable divisions across the tanks, the water can be made to pass upwards or downwards as may be required.—N. H. J. M.

### (C).—DISINFECTANTS.

#### PATENTS.

*A Liquid for use as a Fertiliser and Insecticide, and for other Agricultural and Horticultural Purposes, applicable also as a Deodorizer and Disinfectant.* S. Elliott, Newbury, Berks. Eng. Pat. 8910, April 7, 1894.

See under XV., page 652.

*Improvements relating to the Burning of Sulphur for Disinfecting Purposes.* H. H. Lake, Southampton Buildings, Middlesex. Eng. Pat. 4395, April 14, 1894.

THE apparatus consists of a sulphur candle, made of a block of sulphur with a wick, enclosed in a container made of some porous material, such as paper. This rests on a perforated metal cap placed in a metal cylinder, into which a suitable quantity of water is introduced. The water prevents the fire from being transmitted to surrounding objects, and is gradually and constantly supplied to the sulphur. On reaching the heated sulphur, the water is vaporised and forms sulphurous acid with the sulphurous oxide.—N. H. J. M.

*Improvements in Disinfectants.* I. S. McDougall and J. T. McDougall, London. Eng. Pat. 11,679, April 14, 1894.

IRON salts are rendered more effective as disinfectants by the addition of aluminium or magnesium salts. Ferrous and ferric chlorides and sulphates, or ferric nitrate or acetate may be used singly or mixed. To increase both disinfecting and precipitating power an aluminium salt such as sulphate (40 parts) is mixed with the iron salt (60 parts). To increase the hygroscopic effects a magnesium salt is added in amounts varying from 10 to 20 per cent.—N. H. J. M.

*Improvements in Sulphur Candles and their Holders.* C. T. Kingzett, Chislehurst, Kent. Eng. Pat. 11,807, April 14, 1894.

THE difficulty of igniting an ordinary mass of sulphur is entirely overcome by using a wick of cotton, or asbestos impregnated with sulphur (finely divided sulphur or sulphur dissolved in carbon bisulphide may be employed), or a wick with a mixture of sulphur and some oxidising agent as potassium chlorate. Instead of a wick, a fuse consisting of a mixture of sulphur and potassium chlorate, sodium nitrate, or permanganate, may be employed. With the wick or fuse the candle will burn until all the sulphur is used up.

The candle may be burnt in a double cylindrical holder containing water or any other medicament, such as Sanitas oil. The evaporation of the water renders the sulphurous anhydride much more effective by securing more complete contact between the sulphurous anhydride and the articles which require to be disinfected.—N. H. J. M.

*Improvements in Insecticides and Fungoid Exterminators.* W. S. McDougall and J. T. McDougall, London. Eng. Pat. 11,944, April 14, 1894.

PRECIPITATED sulphur prepared by any well-known process is employed. Sulphur in this very fine state of division is much more efficient and can be distributed over a much greater space than flowers of sulphur.—N. H. J. M.

*Improvements in Fumigants.* W. S. McDougall and J. T. McDougall, London. Eng. Pat. 12,406, April 14, 1894.

THE improvement consists in employing meal-flours as binding materials in the production of fumigants. For fumigants for use on vegetable growths and for disinfecting purposes respectively, the following mixtures are employed:—(1.) Tobacco extract in powder (4 parts), middlings (4 parts), water (2 parts). (2.) Sulphur powder (5 parts), common quality of wheat-flour (3 parts), water (2 parts). The ingredients are mixed into a dough, and dried. Saltpetre may be added to render the product self-consuming when ignited.—N. H. J. M.

## XIX.—PAPER, PASTEBOARD, Etc.

*Spontaneous Combustion of a Lamp Shade.* A. Dupré, Times, May 23, 1894.

See under *Analyst and Solent Notes*, page 670.

*The Causes of the Difficulty of Sizing Paper in the Hot Season.* E. Muth. *Dingl. polyt. J.* 290, 261.

THE often-observed difficulty of sizing paper during hot seasons is due to the resin soap used for this purpose. In consequence of the higher temperature of the air, the pulp in the beaters often reaches a temperature of 25°C., and under these conditions, favoured by the violent agitation of the whole mass, the exceedingly fine globules or flakes of resin formed by the action of the alum or aluminium sulphate upon the resin soap, instead of immediately filling up the pores of the fibre and incrusting it, unite to larger aggregates of a sticky character, and which, therefore, are no longer capable of serving the purpose of sizing, but may even cause the paper to adhere to, and tear upon, the calender bowls. As it is scarcely practicable to reduce the temperature of the contents of the beaters, the only remedy is to shorten the time occupied by the sizing operation as much as possible, and to allow, during hot seasons only, from 15 to 20 minutes for the sizing, instead of the usual 45 or 60 minutes. It is also necessary to dry the paper upon the machine very gradually. With these precautions the sizing of paper will be found to offer no difficulty during hot weather.—C. O. W.

## PATENT.

*Improvements in Artificial Whalebone for Dresses and Corsets.* E. Tourrette, Brussels, Belgium. Eng. Pat. 20,708, November 1, 1893.

VEGETABLE parchment possesses none of the drawbacks proper to other whalebone substitutes, and has been found to improve in quality under the influence of the temperature of the human body; it therefore forms the claim of this patent.—A. G. B.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Stability of a 1 per mille Solution of Corrosive Sublimé exposed to the Atmosphere.* Taurat. *Comptes rend.* 117, 1081—1082.

ACCORDING to Leo Vignon, a solution of corrosive sublimate in distilled water containing 1 per mille of dissolved salt, is quickly altered by exposure to air, a white precipitate being rapidly formed, and the solution impoverished to about half the original strength at the end of a week. The author finds that this effect is due to free ammonia in the air, and that if ammonia be absent, no precipitation or loss of strength occurs.—C. R. A. W.

*Cinchonifine.* E. Jungfleisch and E. Leger. *Comptes rend.* 119, 535—538.

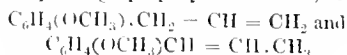
CINCHONIFINE is isomeric with cinchonine, being produced therefrom by heating to 120° in presence of sulphuric acid mixed with its own weight of water. Several alkaloids are thus formed; cinchonifine is found amongst those which either does not dissolve; and from amongst these it is distinguished by being insoluble in 50 per cent. alcohol. It melts at 273°C., or some 1° above the melting point of cinchonine. It is dextro-rotatory, the rotatory power

increasing with the concentration of the solution. It forms two well-marked classes of salts, for the most part readily soluble in water, and crystallisable; several of these are described; also the chloromethylate, mono-iodomethylate, di-iodomethylate, mono- and di-iodethylates, and the mono- and di-bromethylates. (This Journal, 1894, 58).

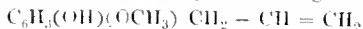
—C. R. A. W.

*Essence of Tarragon; its Transformation into Anethol.* E. Grimaux. Comptes rend. **117**, 1089—1092.

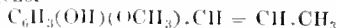
The essential oil contained in Tarragon is stated in various textbooks to consist almost entirely of crystallisable anethol, although this statement is not substantiated by any direct observation. Gerhardt found that it is a fluid, and only isomeric with anethol, although giving the same oxidation products. The author finds that anethol is entirely absent, but an isomeric oxidised substance,  $C_{10}H_{12}O$ , *estragol*, is present, boiling at  $216^{\circ}.5-212^{\circ}$  (uncorrected), or about  $16-17^{\circ}$  lower than anethol. This body does not solidify at  $-43^{\circ}$ ; its specific gravity is  $0.9325$  at  $15^{\circ}$ ; and the index of refraction,  $nd = 1.523$ . When heated on the water-bath for 24 hours with three or four times its volume of concentrated alcoholic potash, estragol is largely converted into the higher-boiling isomeride anethol, melting at  $21.5^{\circ}$ . The original estragol, accordingly, appears to be an allyl derivative of phenol, whilst anethol is the corresponding propenyl derivative, respectively,



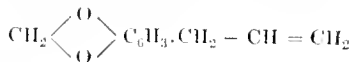
since the relationships between the two (as to difference in boiling point, about  $16^{\circ}$ , and transformation of lower-boiling isomeride into the other by heating with potash) are the same as those subsisting between eugenol—



and iso-eugenol—



between the corresponding methyl eugenol and methyl iso-eugenol; and between safrol—



and iso-safrol—



Eykman's *chavicol* (from betel nut), on the other hand, is probably a stereo-chemical isomeride of anethol.

—C. R. A. W.

*A New Source of Rhodinol.* P. Monnet and Ch. Barbier. Comptes rend. **117**, 1092—1094.

The essential oil of pelargoniums, cultivated in Algeria and the south of France, contains a considerable proportion of *rhodinol*,  $C_{10}H_{14}O$ , the odorous constituent of oil (otter) of roses. By fractional distillation *in vacuo*, a portion boiling at  $123-125^{\circ}$  under a pressure of 14 mm. is isolated; this is heated in closed vessels to  $140^{\circ}$  for eight hours with its own weight of acetic anhydride; the product is washed with alkali to remove acetic acid, and finally with water, and again fractionated *in vacuo*; the portion passing at  $128^{\circ}-129^{\circ}$  under 14 mm. pressure is pure rhodinol acetate, identical in all respects with that from oil of roses. On saponification by alcoholic potash it furnishes rhodinol, boiling at  $124-125^{\circ}$  under 14 mm. pressure, identical with that obtained from rose oil, not only as regards physical and organoleptic properties, but in its chemical reactions; by oxidation it forms an aldehyde, *rhodinol*,  $C_{10}H_{16}O$ , which combines with bisulphites, hydroxylamine, and phenylhydrazine, and is oxidised by ammoniacal silver solution to *rhodinolic acid*,  $C_{10}H_{16}O_2$ . Rhodinol combines with bromine to form a tetrabromide,  $C_{10}H_{14}Br_4O$ ; chlorine forms a liquid product,  $C_{10}H_{14}Cl_2$ , from which a terpene is obtainable, boiling at  $177-178^{\circ}$ , and forming a tetrabromide, melting at  $123^{\circ}-124^{\circ}$ .—C. R. A. W.

*Presence of Camphene in Spike Oil.* G. Bouchardat. Comptes rend. **117**, 1094—1096.

Oil of spike (*Larandula spica*) contains a small proportion of a camphene, isolated by distillation, whereby 9 grms. of liquid passing at  $155-160^{\circ}$  and 15 grms. passing at  $160^{\circ}-165^{\circ}$  were obtained from 4 kilos. of oil; the monochlorohydrate,  $C_{10}H_{17}Cl$ , obtained from these fractions was solid and levorotatory, whereas the hydrocarbon itself was dextrorotatory. Alcoholic solution of potassium acetate decomposes the hydrochloride from camphene at  $80^{\circ}$ , but has practically no action on that from turpentine; the hydrochloride obtained from spike oil was almost entirely decomposed in this way, solid camphene in an imperfectly pure condition being isolated from the product of the action.

—C. R. A. W.

*Synthetic Borneols.* G. Bouchardat and J. Lafont. Comptes rend. **118** (1894), 248—250.

Bertram and Walbaum (J. prakt. Chem. 1894) have obtained an isomeride of natural borneol by heating camphene at  $50-60^{\circ}$ , with a mixture of glacial acetic acid and sulphuric acid. The authors have succeeded in preparing a similar compound from terebenthene. The following method of preparing camphene borneol is more rapid than that of Bertram and Walbaum (*loc. cit.*):—Equal parts of camphene and glacial acetic acid are mixed with one-third the weight of sulphuric acid diluted with acetic acid. The reaction is moderate and instantaneous, the chief product being the acetate of the borneol together with a small quantity of a new compound of camphene, and the borneol having the formula  $C_{20}H_{32}O$ , and boiling at  $187^{\circ}$  under a pressure of 20 mm.

Camphene borneols are more readily soluble in light petroleum spirit than terebenthene borneols and the natural compounds, they form smaller crystals than the former and sublime readily. Camphene borneols are also less stable than their isomerides, inasmuch as they decompose when heated a little above  $250^{\circ}$ ; on oxidation the same camphor is obtained that results from other borneols.—A. R. L.

*The Odour of Benzoic Acid with Remarks on other Odorous and Inodorous Bodies.* J. Passy. Comptes rend. 1894, **118**, 481.

See under Analyt. and Scient. Notes, page 670.

## PATENTS.

*Improvements in the Manufacture of Orthohalogen-phenol, and in the Production of Pyrocatechine therefrom.* H. Baum, Frankfurt-on-the-Main, Germany. Eng. Pat. 12,942, July 1, 1893.

The employment of phenol for the manufacture of pyrocatechin (pyrocatechol) has hitherto been regarded as a technical impossibility owing to the difficulty of brominating or chlorinating it so as to form the ortho-halogen-phenol. If bromine vapour or chlorine is admitted into phenol heated to  $150-180^{\circ}$  C. ortho-bromo or ortho-chloro-phenol are formed in abundant quantities, and are practically pure. These products, purified in a manner described in the specification, on fusion with caustic potash at a high temperature, under pressure, yield pure pyrocatechol.—J. C. C.

*Process for Obtaining an Iron Albumen Derivative from Animal Organs.* B. Wilcox, London. From C. F. Boehringer and Soehne, Mannheim, Germany. Eng. Pat. 16,589, September 4, 1893.

This is a process for obtaining an iron albumen derivative from animal organs, such as the liver, spleen, and kidneys, and consists in gradually heating the reduced or minced organs with water to boiling point, and extracting and mixing the solution with dilute acid until a brownish yellow precipitate forms.—J. C. C.

*An Improved Manufacture of Artificial Musk.* W. Mallman, Roermond, Holland. Eng. Pat. 1018, February 24, 1891.

A mixture of valerate of lime and toluylate of lime is distilled in the dry state. The heavy ketone is separated from the distillate by washing repeatedly with dilute soda solution; this ketone is redistilled, and the products distilling up to 235° to 245° C., are finally nitrated, forming trinitro-iso-butylketone. In this process the inventor obtains a large yield and but little by-product, and also the product produced has a more pleasant and lasting odour than the known trinitrobutyltoluene.—J. C. C.

*Improvements in Photographic Developers.* C. D. MacL, London. From The Actien Gesellschaft für Anilin-Fabrikation, Berlin, Germany. Eng. Pat. 11,872, June 16, 1893.

By introducing further substituting groups, viz., the amino or hydroxyl group into the benzene nucleus of the para-phenylenes, their developing properties are enhanced to such a degree, that the products may be used in the presence of carbonates or sulphites of alkalis, even with bisulphites.

Aminopara-phenylenediamine, oxypara-phenylenediamine or aminopara-toluenediamine are the developers claimed.

—J. C. C.

*An Appliance for Washing Photographic Plates and Prints.* W. Thomson, Manchester. Eng. Pat. 12,734, June 29, 1893.

The inventor claims a method of washing photographic plates or prints by a continuous current of pure water, and the use of a moveable partition as a means of lifting the plates as well as for directing the flow of the water. For details of this appliance the original specification must be consulted.—J. C. C.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

### PATENTS.

*Improvements in or Relating to the Manufacture of Films for Photographic Purposes.* T. H. Blair, Paris. Eng. Pat. 3504, March 14, 1893.

In this process the long slab or table which is usually employed in the manufacture of the body of the film is dispensed with. Instead of it the patentee uses a slowly revolving drum with a slightly roughened surface to receive the substance forming the substratum of the film. This substance is supplied by means of a hopper with a narrow opening of about the ultimate breadth of the film. From the drum the material is carried over bands, rollers, &c., until sufficiently dry. After that it enters a dark chamber, where it is dipped in the sensitive emulsion, then cooled by a blast of cold air, and finally wound on a reeling apparatus consisting of a shaft provided with an apron, upon which slats or bars with projecting portions at the ends are fixed in such a way as to prevent the second layer of film and apron from touching the former, and allow sufficient air space. The film is thus held under suitable tension until it is dry and ready for use.—H. S.

*Improvements in or connected with the Preservation of Photographs Produced on Slate for Decorative or Artistic Purposes.* W. and H. Jones, Bangor, North Wales. Eng. Pat. 10,685, March 31, 1893.

THE part of the slate upon which the photograph is to be produced, is first enamelled white or tinted by a process of slate enamelling. The photograph is then produced on the part so coated, and is then covered with a very thin coating of very pale polishing copal varnish. The slate is then stoved and afterwards polished with very soft rotten stone.

—J. C. C.

*Improvements in the Manufacture of Sensitive Plates and Films for Photographic Purposes.* C. E. Pettitt, Torquay. Eng. Pat. 8956, May 4, 1893.

THE object of this invention is to diminish or prevent halation and solarization. A colored medium is interposed between the sensitive emulsion and its support, so as to obstruct or absorb the actinic rays in their passage to and from the reflecting surface of the support.

The colored medium may be prepared in several ways; for instance, it may be gelatin, albumin, or other vehicle colored with a blue, yellow, or red of such density that it will prevent halation and yet not unduly interfere with the printing property of the negative. Or the colour of the medium may be of a distinctly non-actinic character and may be removed after exposure by chemical means.

—J. C. C.

*Improvements in the Manufacture of Orthoholographenol, and in the Production of Pyrochekine therefrom.* H. Baum, Frankfort-on-the-Main, Germany. Eng. Pat. 12,942, July 1, 1893.

See under XX., page 658.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*Researches on the Manner of Combustion of Ballistic Explosives.* P. Vieille. Comptes rend., 1894. 118, 346–347, and 458–461.

At present, the probable behaviour of a new powder when fired in any service weapon cannot be accurately foretold from its physical characteristics or its chemical composition: experiments must be made with it in a known arm, and these experiments are especially laborious and costly in the case of slow-burning powders which require the use of heavy charges. Piobert experimented in 1833 on the rate of combustion of powders under normal pressure; but his belief that the rapidity of combustion is unaffected by pressure has long been considered untrue, while the hypothesis that the combustion of the material proceeds in parallel layers, under pressure as well as in the open air, although accepted for powders of high density and affording the *raison d'être* of large grain explosives, does not perfectly stand the test of exact experiment. The author has therefore sought to imitate the conditions obtaining in the gun at the moment of discharge by observing the behaviour of the powder when exploded in a closed vessel under a pressure of several thousand atmospheres. In this studying, however, the development of pressure by combustion in a closed and perfectly resisting vessel, where the pressure at any given moment depends only upon the fraction of the charge which has been burnt, the conditions are not exactly those met with in the gun where the movement of the projectile allows expansion to take place and thus affects the specific heats of the gases.

In burning large grain powders, if combustion proceed by surfaces parallel to the original surface of the grain, the quantity burnt at any moment should be a function of the size of the grain and the rapidity of combustion, and a knowledge of the quantity burnt, calculated from the elements of time and pressure, determines the rapidity of combustion; and these elements added to the energy of the substance determined either theoretically or by pressure measurements, completely evaluate the powder for ballistic

purposes. Since by combustion in a closed chamber, pressure at any instant depends upon the fraction ( $x$ ) of the charge burnt, the formula—

$$P = \frac{f \Delta}{1 - d \Delta}$$

may be exchanged for—

$$x = \frac{1}{1 + \frac{1 - a \Delta}{1 - \frac{\Delta}{\delta}} \cdot \frac{P_0 - P}{P}}$$

where  $P_0$  represents the final maximum pressure,  $\Delta_0$  the mean density of the products of complete combustion,  $P$  the pressure at any moment,  $a$  the volume, and  $\delta$  the density of the explosive. From the curve expressing the relation of  $P$  and  $t$ , both  $x$  and the rapidity of combustion may be found. But the application of this method depends upon the accuracy of the hypothesis of

combustion by parallel layers at high pressures. Experiments were therefore made both with the older black and cocoa powders, and with the newer colloidal gelatinised cotton explosives, by burning in the closed vessel equal charges of grains, geometrically similar but of unlike sizes, cut from the same explosive by means of a saw. If the combustion of the powder proceed by parallel layers, the pressure should vary as a function of time, and hence with the ratio of similarity existing between the sizes of the grains. The following table of results shows however, that for black and brown powders the ratio of similarity does not accord with the ratio of time nor with the inverse ratio of  $\frac{dP}{dt}$ . It would appear that for these powders the law of parallel surfaces does not hold good, but that as the curves of each pair of experiments are practically identical, the powders are disaggregated at very moderate pressures. The colloidal powders on the contrary by the agreement of the different ratios show that they follow the law of combustion by parallel surfaces.

Nature of Powder.	Density of Charge.	Maximum Pressure in Kilos. Per Sq. Cm.	Ratio of Similarity of Charges.	Time observed, in Thousandths of a Second.	Time Ratio.	$\frac{dP}{dt}$ Maximum in Tons per Second.	Inverse Ratio of $\frac{dP}{dt}$ .
<i>Black or Brown.</i>							
French 1. ....	0.4	1,600	2.0	( 7.64 ) ( 7.95 )	0.9	( 498.0 ) ( 450.0 )	0.9
" 5. ....	0.6	8,000	3.7	( 10.56 ) ( 8.86 )	1.2	( 212.5 ) ( 206.8 )	1.0
German prismatic brown PRH .....	0.6	3,000	3.2	( 23.75 ) ( 16.55 )	1.4	( 220.3 ) ( 267.6 )	1.2
Chinese prismatic brown.....	0.6	3,000	2.0	( 15.88 ) ( 16.04 )	1.0	( 254.4 ) ( 248.8 )	1.0
<i>Colloidal.</i>							
Pure cotton powder (German type).....	0.2	2,400	4.0	( 18.55 ) ( 17.5 )	1.3	( 249.0 ) ( 191.0 )	1.5
Nitrated cotton powder (French industrial BN) .....	0.2	1,600	2.0	( 41.95 ) ( 19.52 )	2.1	( 68.2 ) ( 139.8 )	2.0
Nobel's ballistite, Bilbao .....	0.2	2,600	3.0	( 18.60 ) ( 17.16 )	2.6	( 201.1 ) ( 55.4 )	2.8
English cordite type .....	0.2	2,600	2.0	( 5.50 ) ( 1.83 )	3.0	( 658.7 ) ( 238.6 )	3.6

—W. G. M'

#### PATENTS.

*Improvements in or relating to Explosives.* E. Kabin, Prague, Austria. Eng. Pat. 11,502, June 12, 1893.

This invention consists essentially in the employment of the nitrates of amido-compounds, especially of those of aniline and teluiline, mixed with ammonium nitrate.

—W. M.

*Improvements in and Apparatus for Igniting Blasting Charges.* C. Roth, Hennickendorf, Germany. Eng. Pat. 23,417, December 5, 1893.

The object of this invention is to ignite blasting charges without danger of producing explosions of firedamp, the production of a flame outside of the hole being dispensed with. Certain gases or liquids are caused to pass through a fine-bore leaden tube, leading to the cartridge, whereby they are brought into contact with substances of such a nature that the action of the gases or liquids generates sufficient heat to cause the explosion of the cartridge. In practice this invention may be carried out when detonators are required by placing some finely-ground lead peroxide in the front end of the detonator above the fulminate. This peroxide is in contact with the open end of a thin lead, rubber, or papier maché tube of a sufficient length to ensure

the safety of the person firing the charge, and communicating at its other end with a gas-generating apparatus. When sulphurous acid is turned on it drives the air out of the tube and detonator through a small tube or passage provided in the material employed for tamping, and, coming in contact with the lead peroxide, generates sufficient heat to explode the detonator. In a similar manner antimony and chlorine, spongy platinum or platinised asbestos and hydrogen may be employed. Another method of working consists in placing a small quantity of a mixture of potassium chlorate and sugar in the detonator or charge and forcing sulphuric acid along a leaden tube which is in contact with the mixture, when explosion takes place.

Details and drawings of the several methods of carrying out this invention are given in the specification.—W. M.

*An Explosive Composition.* A. Maurette, Toulouse, France. Eng. Pat. 4626, March 5, 1894.

"This invention consists in a composition of materials for use as an explosive":—Saltpetre 640, powdered wood-charcoal 50, wood-ashes 55, chlorate of potash 5, and flowers of sulphur 25 parts.—W. M.

*An Improved Compound for use in the Manufacture of Matches.* H. Priester, Lauenburg, Pomerania. Eng. Pat. 5939, March 21, 1894.

THE inventor claims "the utilisation of magnesium either alone or alloyed with other metals to form combustible compounds capable of being applied by dipping," matches coated in this way giving a coloured light and scattering sparks when burning. It is stated that a much more brilliant result is obtained when the magnesium is alloyed with copper, calcium, or zinc and then added to one of the known firing mixtures.—W. M.

## XXIII.—ANALYTICAL CHEMISTRY.

### APPARATUS, ETC.

*Some Laboratory Apparatus.* A. Bidet. Comptes rend. 1894, 118, 478—481.

**SIPHON LEVEL REGULATOR.**—This is an S-shaped tube (Fig. 1) with a side tube S sealed on a little above the lower bend. The long arm O is inserted in the apparatus

Fig. 1.

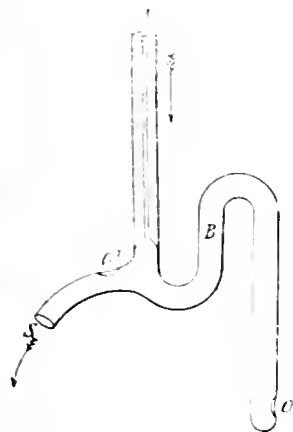
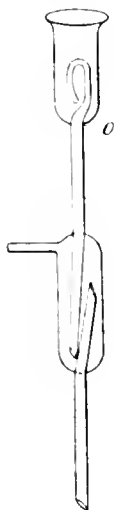


Fig. 2.



in which the constant level of liquid is required, and the hole O is placed at the side of the tube to prevent air from the boiling water passing up into the siphon tube. Water flows continuously through A, and may then pass through either S or B, the tube BO forming a siphon which may act in either direction according as the level of the water in the apparatus to be supplied is above or below that for which the instrument is regulated. The hole O, by free admission of air, serves to prevent the tube S from acting as the limb of a siphon.

**SAFETY TUBE ARRANGED TO PREVENT SPLASHING.**—This apparatus (Fig. 2) serves for introducing liquids into a vessel and for leading off gases by the side tube. Regurgitation quietly transfers the liquid in the middle cylinder into the upper funnel through the recurved tube at O, whence it returns after the pressure has again become normal. The lower tube is adapted to a tubulure by a cork with one perforation.

**INTERNALLY-COOLED CONDENSER.**—In this instrument (Fig. 3) the vapours entering by the upper tube are condensed in the annular space surrounding a water-cooled glass

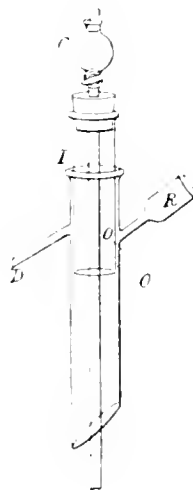
tube, sealed into an outer vessel at its lower end, and the condensed liquid passes away through T. An outside water-jacket may be adapted to this, so that a double cooling surface is presented for condensation.

Fig. 3.



**COMMUTATING ARRANGEMENT FOR CONDENSER.**—By connecting an inverted condenser with R (Fig. 4) and a downward condenser with D, and adjusting the inner circularly-sliding tube I so that the hole O is opposite one or other of these tubes, this instrument allows distillation or cohobation to be carried on at will, or to be effected

Fig. 4.

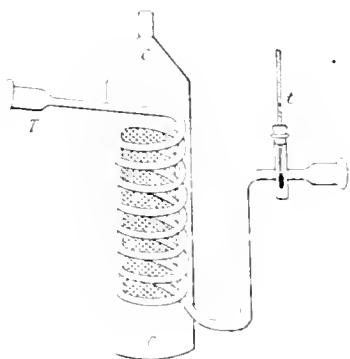


alternately without delay. The tubes are joined by an india-rubber ring. A cork in the inner tube permits the passage of a separating funnel for the introduction of liquids into the distilling flask.

**SUPERHEATER FOR VAPOURS.**—The superheating is effected by passing the vapour through T (Fig. 5) to the copper spiral, which is enclosed in a brass jacket terminating above in an open cone. The spiral is lined with metallic gauze, and beneath it is placed a suitable burner. The

superheated gases pass away over the bulb of a thermometer *t*. With such an instrument 20 cm high a tem-

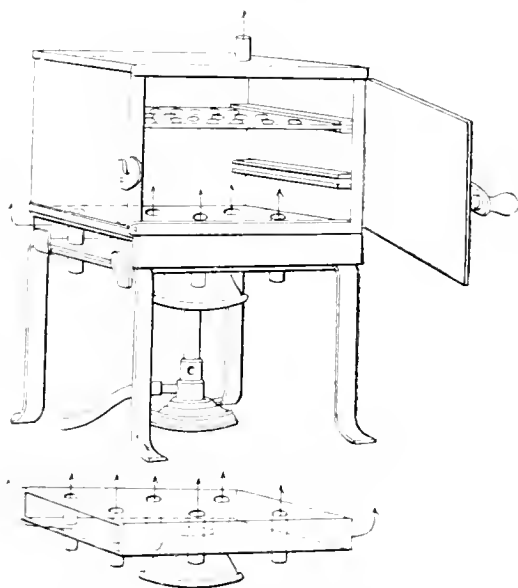
Fig. 5.



perature of 300° C. has been attained in the efflux. With a triple Bunsen burner a still higher temperature could be attained.—W. G. M.

*A New Form of Air Bath.* E. Sauer. J. Amer. Chem. Soc. 1894, **16**, 31—33.

In order to ensure a constant temperature, which shall not be limited by the boiling point of some liquid contained in an outer jacket, the author has devised the form of air bath shown in the figure. The bath has a false bottom, perfor-



ated to allow a series of tubes to connect the inside of the oven with the outside air beneath the apparatus through the real bottom; the latter, in addition to corresponding perforations, has a larger central aperture through which the flame of a Bunsen burner can play upon the false bottom and around the connecting tubes; the products of combustion pass away through the sides of the lower chamber between the two perforated plates, the sides being left open. Evaporation is facilitated by the rapid renewal of the atmosphere within the bath by means of the tubes; and experiments are quoted to show that not only is evaporation rapid, but the temperature at different parts of the bath is very uniform.—W. G. M.

*Dropping Flask for Standard Solutions.* F. Vanderpoel. J. Amer. Chem. Soc. **16** [3], 156—157.

The use of burettes for standard solutions involves serious errors (e.g., such as arise from alteration of temperature), which can be avoided by substituting weighing for measuring. The figure shows a simple form of dropping flask for standard solutions, which has been found convenient. The

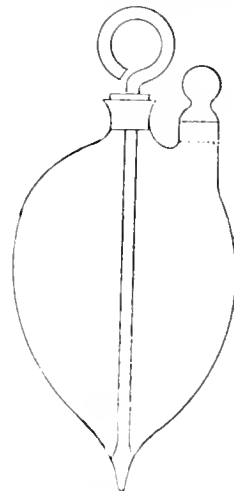
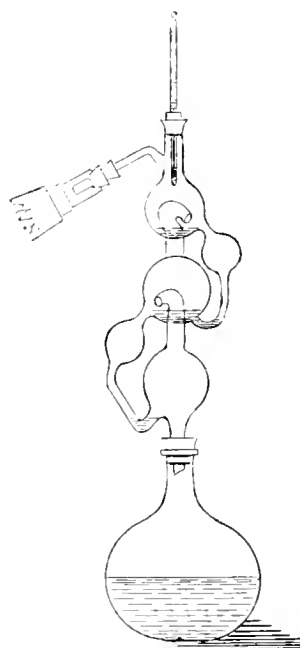


figure is almost self-explanatory. It suffices to say that the central rod is of glass, and passes through a cork in the upper tubulus of the flask, and is ground into the lower exit tube, thus serving to regulate the outflow. The side tubulus is used for filling the flask. The whole apparatus can be slung to a hook on the balance or supported on a light tripod in the manner of a weighing bottle.—B. B.

*Apparatus for Fractional Distillation.* C. W. Volney. J. Amer. Chem. Soc. **16** [3], 160.

The apparatus shown in the figure is similar to an ordinary dephlegmator, save that the central tubes constituting the



passage for the vapour from the flask to the condenser are not trapped by liquid. The author states that in spite of this, fractionation proceeds readily, and that products of nearly constant boiling point are obtained by a single repetition of the distillation.—B. B.

#### PATENTS.

*Improvement for the Determination of Butter Fat in Milk.* C. R. C. Tichborne, Dublin, and C. J. Thompson, Bray. Eng. Pat. 7547, April 13, 1893.

The apparatus consists chiefly of a circular metallic basin, provided with a cover, round the inside of which the graduated milk-bottles are placed in compartments. The basin may be filled with hot water, or the arrangement may be used as a steam-jacket by passing steam through an opening of the top of the cover.

The inventors claim, as chief improvement, the special application of the heat from the top of the apparatus instead of from the bottom of the tank, so as to avoid the chilling of the bottles, and an arrangement by which currents of air produced by the revolution of the machine fall back upon the stems containing the isolated fat. A minor improvement consists in the use of a screw regulating the height of the shaft carrying the frame and holding the bottles.—L. de K.

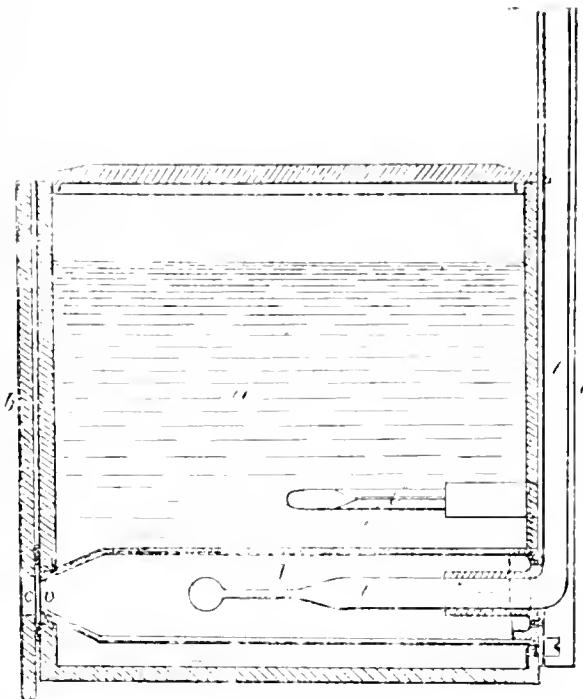
*Improvements in Apparatus for Detecting and Estimating the Proportion of Fire-damp or other Inflammable Gases in the Atmosphere of Mines and other Spaces.* G. Fletcher, Derby. Eng. Pat. 12,261, June 22, 1893.

This is a patent for an instrument based on the known fact that a luminous spiral of wire glows more brightly when the surrounding air contains a small proportion of some inflammable gas. The instrument consists of two identically similar spirals of fine platinum wire. One of these is enclosed in an air-tight tube containing air and having the upper end closed by glass. The other spiral is contained in a similar tube of wire-gauze having the upper end likewise closed by glass. The spirals are observed through a screen of ground glass or other suitable substance. When a current of electricity is passed through both spirals in air they glow equally, but if they are introduced into an atmosphere containing an inflammable gas, the spiral in the open or wire-gauze tube glows the more brilliantly, the brilliancy increasing with increase in the proportion of inflammable gas present. By a pinion wheel fitted with a pointer the spiral in the closed tube may be approached to, and that in the wire-gauze tube reeled from the ground glass disc till the two appear equally luminous. The circle over which the pointer moves is graduated by experiment with measured percentages of gas, and afterwards enables the operator to directly read off the percentage present. Any small storage battery, such as those in use for miners' lamps, may be used as the source of the electricity.—L. T. T.

*An Improved Actinometric Pyrometer.* C. Latarche, Lempère, near Verdun, France. Eng. Pat. 18,652, October 5, 1893.

This instrument consists of a chamber *l* containing a thermometer *t*, and surrounded by a cistern of water *a*, containing a second thermometer *t'*. The chamber *l* is closed except for the circular opening *o*. The instrument is placed against the wall *b* of the furnace in such a way that the opening *o* is against a corresponding opening *c* made for that purpose in the wall *b* of the furnace. The bulb of the thermometer *t* and the inside of the chamber *l* are blackened. The water in the cistern *a* is kept at about the boiling point by the heat from the furnace wall, and the thermometer *t* is also warmed by the heat of this water-jacket. But in addition the bulb of the thermometer *t* receives the heat rays of the furnace through the opening *c o*, and therefore registers higher than *t'*, which of course shows the temperature of the water. This difference, read on the scales at the front *d* of the apparatus, gives an

index of the furnace temperature, the difference being dependent on the size of the opening *c o*, the distance of the bulb of *t* from this opening, and the heat of the furnace chamber. By graduating the instrument with known furnace-heats, and regulating the size of the opening *c o*

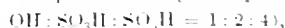


and the distance of the bulb of *t* from that opening, any degree of sensitiveness may be attained, and each degree-centigrade of *t* in excess of *t'* may be made to represent any chosen number of degrees of heat. For moderate furnace-heats the author suggests regulation, so that each degree excess of temperature of *t* represents 10 °C. of actual temperature. For very high furnace-heats it is advisable to reduce the sensitiveness by reducing the size of the opening *c o* by introducing a diaphragm there. The instrument is best hinged to the furnace wall, so that during stoking, &c., it may be swung on one side to prevent as much as possible the accumulation of dust in *l*. The patentee claims that the instrument does not get out of order, gives very reliable results, and that the temperature can at any time be read off at once.—L. T. T.

#### INORGANIC CHEMISTRY.— QUANTITATIVE.

*The Determination of Nitrates in Potable Water.* A. H. Gill, J. Amer. Chem. Soc. **16**, 122–132.

STIMULATED by the unfavourable criticisms of Hazen and Clark (J. Anal. Appl. Chem. **5**, 1) upon the phenol-sulphonic acid process of determining the nitrates in water, as proposed by Sprengel and revived by Grandval and Lajoux, the author has re-investigated the method, which he now considers to be more delicate, trustworthy, and accurate than any process yet published. The best reagent is pure phenol disulphonic acid (probably—



which with nitric acid gives picric acid even in the cold; it is prepared by heating in (not upon) a water-bath at 100° for six hours, a flask containing 3 grms. of pure phenol and 37 grms. of pure sulphuric acid



sp. gr. 1.840); the purity of the product thus obtained has been proved by the analysis of the re-crystallised barium salt. It is apt to crystallise out upon standing, but is readily re-dissolved on warming. Among other reagents, equal parts of phenol and sulphuric acid gave brown colours, which could not be accurately matched; mixtures of the ortho and para-sulphonic acids from a 5 per cent. solution of phenol gave a fairly good, but sometimes reddish shade; while pure ortho-phenol mono-sulphonic acid gave a precipitate with ammonia and a bluish-green coloration. The disulphonic compound in 7.5 per cent. solution yielded a pure yellow, which did not change upon standing. In the tests of the process a standard solution of potassium nitrate containing 0.1 part of nitrogen per 100,000 was employed, and for certain tests a solution of sodium chloride, containing 7 parts of chlorine per 100,000, was also used. 1 cc. of the nitrate solution was diluted to 5 cc., and evaporated in a porcelain dish 2½ inches in diameter; and the residue was moistened with ten drops (0.7 cc.) of the phenol-sulphonic acid, contact being ensured in every part by rubbing with a glass rod. 7 cc. of water was then added and afterwards 3 cc. of ammonium hydrate, the liquid being well stirred after each addition. When the colours could not be accurately compared in the dishes, tubes 1½ inches deep and ½ inch in diameter were employed. Variations in the process are dealt with under several heads:—

*Method of Treatment:* The picric acid reaction gives low results at 0° C., but good results alike at 100° and at the normal temperature. The addition of phenol or of phenol sulphonic acid to the water before evaporation offers no advantages. *Quantity of Acid used:* When little chlorine is present, the amount of acid used is of little consequence, provided that it suffice to cover the residue; but the use of less than 10 drops may cause loss of nitric acid.

*Evaporation:* A slight loss (equal to about 1 per cent. of the nitrogen present) occurs when the evaporation is conducted at 20° over sulphuric acid, or at 100° on a steam bath, if the dish be removed immediately the residue is dry; a greater loss results from the more prolonged evaporation at 65° C. The addition of sodium carbonate before evaporation has no marked effect, while the use of sodium acetate rather increases the loss of nitrogen. A large volume of water evaporated gives lower results than a small volume, but the difference is not great. Heating of the dried residue even to 65° C. causes a distinct loss of nitrogen. *Effect of Chlorine:* In waters containing less than 2 parts of chlorine per 100,000 the halogen exerts no appreciable influence, but with 7 parts per 100,000 the evaporation of a comparatively large volume (10 cc.) at 100° may entail a loss of 30 per cent. of the nitrogen, but this loss may be reduced to 6 or 7 per cent. by using only 1 to 5 cc. of the water and evaporating at 20° over sulphuric acid.

*Comparison with Standards:* The errors due to imperfect matching increase (actually but not relatively) with the percentage of nitrogen, ranging from 0.002 to 0.003 part of nitrogen for a sample containing 0.05 part per 100,000 to 0.005 part for one with 0.1 per 100,000. The readings should be taken rapidly, and the eye occasionally rested by looking intently at a blue surface. The author recommends for waters with much nitrogen the use of a quantity which will give a reading of about 0.07; when the chlorine also is high, evaporation should be conducted *in vacuo* over sulphuric acid, while if it exceed 7 parts per 100,000 it should be removed by silver sulphate free from nitrates. With ground water 10 cc. of a portion, previously decolorised by alumina in the cold, is evaporated on the steam bath, the dish being removed before the disappearance of the last drop of liquid. For the standard, 0.72 gm. of potassium nitrate is dissolved in 1 litre of water, 10 cc. of the solution is evaporated *in vacuo*, treated with acid, and made up to a litre, 10 cc. of this contains 0.1 part of nitrogen per 100,000. Portions withdrawn for test are made alkaline as required. The process does not determine nitrogen in nitrites, since nitrous acid gives nitro-phenol with phenol sulphonic acid, and this is colourless in dilute solutions.

—W. G. M.

*The Determination of Nitrates in Potable Water.* A. H. Gill. J. Amer. Chem. Soc. 16 [1], 193—197. (This Journal, 1894.)

**PART II.—REDUCTION TO AMMONIA.**—The chief objections to any method for estimating nitrates in water, based upon the reduction of the nitrates to ammonia, are caused by (1) the corrections to be applied (*e.g.*, that for free ammonia); (2) the risk of obtaining ammonia from the organic matter present; (3) the variation in the colour produced by the Nessler solution, especially when distillation is omitted. The variation is largely due to the presence of hydroxylamine, which is formed to some extent in place of ammonia. The irregularity of the results of the method is shown in tables given by the authors, in which errors of from 5 per cent. to 20 per cent. of the true quantity of nitrate are quoted. The results are generally lower than those obtained by the phenol sulphonic acid process, which yields figures that are, on the whole, nearer the truth. This holds even in presence of chlorine, in which contingency the reduction process is usually considered preferable. The many processes of reduction by means other than the use of aluminium—*e.g.*, finely-divided iron and an acid—are primarily designed for the analysis of commercial nitrates and not for dealing with such small quantities of nitrates as are found in potable waters. Harrow's method (J. C. S. 1891, Trans. 320; this Journal, 1891, 727) is sensitive to 0.04 part of nitrogen per 100,000, *i.e.*, 20 times less delicate than the phenol-sulphonic acid process. The author states, in conclusion, that improvement in the determination of nitrates in potable waters is to be sought in the formation of organic nitro-compounds of high colouring power and constant composition.—B. B.

*General Method for the Volumetric Determination of Silver in any Form.* G. Deniges. Comptes rend. 117, 1078—1081.

THE volumetric determination of silver by means of an alkaline chloride and potassium chromate as indicator requires an exactly neutral solution. Moreover, the presence of certain organic substances interferes with the accuracy, whilst only substances soluble in water can be treated. The Volhard-Charpentier process with potassium thiocyanate permits of the examination of silver compounds soluble in nitric acid, but is inapplicable in the case of substances not soluble therein, such as silver chloride. On the other hand, since all silver compounds are soluble either in ammonia or in potassium cyanide solution, the following process can be used in all cases. The substance is dissolved in ammonia, or, if not soluble completely therein (*e.g.*, silver iodide), in ammonia together with a sufficient quantity of standard potassium cyanide solution. In many cases the solution is best effected by dissolving in nitric acid and supersaturating with ammonia. A definite quantity of potassium cyanide is then added with a little potassium iodide as indicator, and then decinormal silver nitrate until a permanent turbidity is perceptible. This occurs when the total silver present is faintly in excess of that corresponding with the cyanide as regards formation of argentocyanide of potassium,  $\text{AgCy}$ ,  $\text{KCy}$ ; so that if the cyanide solution employed is standardised in terms of the silver nitrate solution, the silver present in the substance tested is known from the difference between the volume of silver nitrate corresponding with the cyanide added, and that actually employed in the titration. Variations in the quantity of free ammonia present do not affect the result; nor does the presence of caustic or carbonate of potash or soda, or of chlorides, bromides, phosphates, &c. Moreover, pure potassium cyanide solution of about 1 per cent. strength is extremely stable at ordinary temperatures, and also at boiling heat in presence of alkalis; so that a standardised solution suffers no change on keeping for a fortnight. Very satisfactory figures were obtained on making tests with known quantities of silver chloride, bromide, iodide, phosphate, chromate, and sulphide. In this way precipitated silver chloride, iodide, &c., may be volumetrically determined, and thus the quantity of chlorine, &c. present in liquids containing organic matter (*e.g.*, urine,

blood, &c.) may be easily ascertained. Similarly, precipitates (organic or otherwise) containing silver may be readily titrated.—C. R. A. W.

*Experiments on the Estimation of Graphite in Pig-Iron.* F. L. Crobaugh. J. Amer. Chem. Soc. 1894. 16, 101–108. From several experiments with different solvents and details of treatment, it appears probable that the graphite may be estimated in pig-iron with sufficient accuracy for commercial purposes as follows:—Dissolve 5 grams of the iron in 125 cc. of nitric acid (1·135 sp. gr.), and add immediately 12 cc. of a 40 per cent. hydrofluoric acid solution; boil for five minutes after complete solution, and then filter through a double tared filter-paper, wash with dilute hydrochloric acid until all iron is removed, and then with alcohol, ether, and ammonia successively, or with ammonia alone. The filter-paper is dried at a temperature below 100° C. and weighed; it is afterwards ignited, and the weight of the residue is deducted from that of the mixed graphite precipitate. The final residue should be practically nil, owing to the solvent action, first of the hydrofluoric acid, and then of the ammonia. A trace of some colouring substance, probably combined carbon, is yielded to the ammonia wash-waters, but is rapidly removed. The hydrofluoric acid etches the beaker in which the solution is effected to some extent, but no severe corrosion was noticed even after several treatments. The ammonia and the alcoholic and ethereal washings leave a black mirror-like deposit on evaporation, which is soluble in warm dilute ammonia; but that from the ammoniacal liquid will not dissolve in alcohol or ether. The dissolved matter separates in each case in brownish flocks. This is a matter, however, which the author is still investigating. Results are sufficiently concordant.—W. G. M.

*The Analysis of Steel.* H. K. Bunber. Iron and Steel Institute, Spring Meeting, 1894.

THE analysis is as follows:—200 grains of steel drillings are taken, to which is added 1 oz. of distilled water with 2½ oz. of pure nitric acid, sp. gr. 1·42. When the violent action has ceased, it is boiled for some time until all further action ceases, and the steel is completely dissolved. To this there is added 1 oz. of a pure 10 per cent. solution of sodium carbonate. The whole is boiled to remove carbonic acid, evaporated to dryness, and ignited in a platinum vessel at a dull red heat. The red powder obtained is then treated with another ½ oz. of the above sodium carbonate solution, evaporated to dryness, treated with hot distilled water, containing about 0·5 per cent. of sodium carbonate, filtered, and washed carefully with the same. If chromium be absent, the solution may be acidulated and evaporated to dryness to remove silica, and the analysis then carried on by the usual methods. The phosphorus is precipitated as ammonia magnesium phosphate, which takes down the arsenic as ammonia magnesium arsenate. These can be weighed together and the arsenic afterwards separated and determined. If chromium be present it is advisable to add the magnesia mixture with a little extra ammonium chloride direct to the alkaline solution, when the phosphorus and arsenic are separated without any trace of chromium.

By the use of this method the author has in several cases found chromium present when it was not even suspected. Where chromium is present there is generally more or less vanadium or molybdenum. These, together with the phosphorus, sulphur, and arsenic, are converted into sodium salts by this method, the iron being converted into insoluble oxide. If distilled water free from sodium carbonate is used it is impossible to filter off the finely-divided oxide of iron.

—A. W.

*The Determination of Phosphorus in Steel.* C. B. Dudley and F. N. Pease. J. Amer. Chem. Soc. 16 [4], 217–224.

THE authors, needing to know with the utmost attainable accuracy the percentage of phosphorus in three samples of steel, made a comparison between various standard methods

with the aid of several chemists accustomed to steel analysis. Sample 1 was a boiler-plate steel containing about 0·15 per cent. C and less than 0·05 per cent. P; sample 2 was a fire steel with 0·5–0·6 per cent. C and 0·05–0·07 per cent. P; sample 3 was a reliable steel with 1·0–1·2 per cent. C and less than 0·01 per cent. P. Special care was taken in sampling. The results obtained for No. 1 varied from 0·035 per cent. to 0·042 per cent.; from No. 2, 0·015 per cent. to 0·026 per cent.; from No. 3, 0·019 per cent. to 0·033 per cent. For the purpose which the authors had in view, these variations were too great to be passed over, and an examination of the cause of discrepancy was accordingly instituted. It was seen that the acetate method tended to yield lower results than the molybdate process, and the following causes of loss were detected. When the process is worked in the usual manner the amount of iron in the ferric state used to precipitate the phosphorus as ferric phosphate is insufficient to carry the whole of the phosphorus down; the residue left on the filter, after dissolving through the ferric precipitate containing the phosphorus with hydrochloric acid, still gives a reaction with molybdate solution, and is therefore not free from phosphorus; the filtrate from the ammonium magnesium phosphate (citric acid being present) also gives evidence of the presence of phosphorus.

The fact that the molybdate method has a tendency contrary to that of the acetate process, prevents the use of the former as an absolute standard, and has led to the device of the following scheme:—10 grams of the metal are treated in the usual manner for the separation of phosphorus as ferric phosphate, but instead of oxidising only a small quantity of ferrous iron, at least 0·5 gram, is converted into the ferric state, and the basic acetate precipitation aided by the addition of a little ammonium acetate. The precipitate, containing all the phosphorus, is dissolved in moderately strong hydrochloric acid, the solution is evaporated to dryness, the residue treated with 40 cc. of strong nitric acid, and evaporation repeated to expel hydrochloric acid. The residue thus obtained is dissolved in 75 cc. of nitric acid (sp. gr. 1·13), the solution filtered, and precipitated with 75 cc. of molybdate solution, with the usual precautions. The precipitate is collected, well washed, dissolved in 2·5 cc. of strong ammonia, diluted with water, and washed until the filtrate measures about 100 cc. Sulphuretted hydrogen is then passed and the solution slightly acidulated with hydrochloric acid, by which treatment molybdenum sulphide is thrown down. The precipitate is filtered off, well washed with weak hydrochloric acid, and the solution evaporated nearly to dryness, diluted with a little weak hydrochloric acid, filtered if necessary, concentrated to 15 cc., and precipitated with magnesia solution and ammonia, and the precipitate collected in the ordinary way. This method tends to give results very slightly below the truth from the retention of phosphorus in the filtrate from the ammonia-magnesium phosphate, but it is preferable to either of the single processes. It is a modification of Riley's combination method, and has the advantage of eliminating arsenic at the point where the molybdenum sulphide is separated.

—B. B.

*The Volumetric Determination of Phosphorus in Steel.* C. B. Dudley and F. N. Pease. J. Amer. Chem. Soc. 16 [4], 224–230.

THE volumetric method, which consists essentially in reducing the phosphomolybdate precipitate by means of zinc and titrating the reduced solution by permanganate, is largely used in America, and its possible failings have now been studied by the authors. The following questions arise:—(1.) Is all the phosphorus collected in the molybdate precipitate? On this the authors cannot express a personal opinion, but quote Lawrence Smith to the effect that the concentration of the phosphorus in a small proportion of the iron by means of a preliminary basic acetate precipitation is desirable. (See preceding abstract.) (2.) Is the yellow precipitate of constant composition? This the authors answer in the negative, insisting on the need for observing standard conditions, and condemning the use of any ratio

expressing the composition of the yellow precipitate obtained under one set of conditions, for calculating the composition of a precipitate produced under different conditions. (3.) How shall the proper factor for use with permanganate of potash be obtained? The published ratios between molybdic acid and phosphorus vary from 100:1.54 to 100:1.90, and a similar ambiguity exists in the relation of the iron (used to standardise the permanganate) and the molybdic acid after reduction. The authors have endeavoured to settle this point by determining phosphorus in steel gravimetrically and volumetrically, and thus obtaining the ratio empirically. (See preceding abstract.) The values which they prefer are: iron to molybdic acid = 100:90.76, and molybdic acid to phosphorus = 100:1.90. (4.) If the conditions are made constant, will the results obtained be uniform and agree with each other? The authors consider that with identity of conditions the agreement of duplicates by the same chemist should be within 0.002 per cent., and of different chemists within 0.004 per cent.—B. B.

*The Determination of Phosphorus in Steel by Dudley's Method.* O. S. Doolittle and A. Eavenson. J. Amer. Chem. Soc. 1894, 4, 234—247.

The paper is mainly a criticism of that by Dudley (*vs.*), and deals with several points discussed by the latter.

*Ratio between phosphorus and molybdic acid in the yellow precipitate.*—The process chosen by Dudley of comparing gravimetric and volumetric results from the same steel, and thus obtaining an empirical ratio, was adopted. Four steels differing considerably in composition were used and gave ratios varying from 100:1.759 to 100:1.813, the value 100:90.76 being taken for the ratio between iron and molybdic acid. The authors therefore conclude that Dudley's ratio 100:1.90 is erroneous. A direct determination of the same ratio in yellow precipitate obtained from a solution of ferric nitrate containing phosphoric acid, was made by estimating the phosphorus in one weighed portion of a solution of the yellow precipitate, and the molybdic acid in another portion of the same solution. The method used for estimating the molybdic acid consisted in precipitation with lead acetate, the mixed precipitate of lead molybdate and lead phosphate being dried at 130° C., and the phosphate allowed for. The ratio between molybdic acid and phosphorus thus computed is 100:1.797. A repetition of the determination on yellow precipitate actually separated in the course of several steel analyses by Dudley's own method, gave the ratio 100:1.792, confirming the value usually accepted, viz., 100:1.794.

*Ratio between iron and molybdic acid.*—The authors found that the means adopted for reducing the molybdic acid previous to its titration with permanganate had a considerable influence upon the results. The common plan of passing the solution through the so-called "reductor" (a zinc-filled tower), gave results which fluctuated with the condition of the reducing material and the time occupied by the liquid in traversing the apparatus. Merely boiling with excess of zinc gave results appreciably lower than those obtained by the aid of the reductor. The ratio finally adopted by the authors between iron and molybdic acid is 100:89.16, instead of Emmerton's value 100:90.76.

*Effect of temperature on the precipitation of arsenic.*—The authors' results confirm those of Handy (*vs.*), in so far as the mechanical action of the phosphorus is concerned, in carrying down arsenic with the molybdate precipitate. Only when both phosphorus and arsenic are present in considerable amount is precipitation of the latter to be feared. A necessary precaution is to keep the temperature of the solution at the time of precipitation as low as is consistent with the complete separation of the phosphorus. The authors consider 32° C. a suitable limit.

—B. B.

*The Determination of Phosphorus in Steel in the Presence of Arsenic.* J. O. Handy. J. Amer. Chem. Soc. 16 [4], 231—234.

An objection to the molybdate method for the determination of phosphorus in steel is that arsenic may be simultaneously precipitated and reckoned as phosphorus. The author shows that when little or no phosphorus (*e.g.*, 0.002 per cent.) is present, arsenic is not precipitated even if present to the extent of 0.5 per cent. In the event of a larger quantity of phosphorus being determined, arsenic may precipitate, being apparently carried down mechanically, but can be eliminated by solution and re-precipitation. These data apply to the author's own method. (J. Anal. Appl. Chem. April 1892.)—B. B.

*Notes on the Determination of Nickel in Steel.* J. Westesson. J. Amer. Chem. Soc. 1894, 16, 110—112.

The presence of chlorides interfering with the electrolysis of the acetic solution after separation of the iron as basic acetate and necessitating the use of hydrogen sulphide to collect the nickel, the author dissolves 1 gm. of the sample in 1.16 sp. gr. sulphuric acid, to which 5 cc. of weak nitric acid is subsequently added; neutralisation with sodium carbonate and precipitation as basic acetate with the aid of sodium acetate are carried out in the usual way. The precipitation of the acetate is twice repeated, and the collected filtrates are evaporated to 400 cc., the addition at this stage of 10 cc. of strong ammonia, followed by boiling for a few minutes, is found to precipitate the manganese, which does not retain more than 1.5 per cent. of the total nickel present. The ammonia must be added to the hot solution in order to separate the manganese satisfactorily. The filtrate is then ready for electrolysis. Results are very concordant, and the whole analysis may be completed in eight hours, with the aid of eight good Grove's cells, or longer with a less powerful battery.—W. G. M.

*Determination of Nickel in Nickel Steel.* E. D. Campbell. J. Amer. Chem. Soc. 1894, 16, 96—102.

This method is intended to provide a rapid and accurate determination without precipitation of basic acetates or use of hydrogen sulphide. Dissolve 2.2222 grms. of the steel, boiling, in a 500-cc. graduated flask in 20 cc. nitric acid of 1.20 sp. gr., and 5 cc. hydrochloric acid of 1.21 sp. gr.; add 155 cc. sodium phosphate solution (200 grms. of crystallised  $\text{Na}_2\text{HPO}_4$  in 1,860 cc. of water); clear, if necessary, with a few drops of hydrochloric acid, add 25 cc. of 1.04 acetic acid, and then 100 cc. of sodium acetate solution (250 grms. crystallised salt in 820 cc. of water), shake, dilute to 502.5 cc., shake again, and allow to stand for 15 minutes. The ferric phosphate thus precipitated will be free from nickel, but may retain a little copper; it will occupy a volume equal to 2.5 cc. The liquid is passed through a dry filter into a dry beaker; 250 cc. is then withdrawn for further treatment, this being exactly half the solution after removal of the iron precipitate. Boil the 250 cc. and add 20 grms. potassium hydroxide dissolved in 40 cc. of water; boil for five minutes and then maintain nearly boiling until the precipitate has separated well; filter through an asbestos filter with the aid of the filter-pump, and wash. The precipitate contains every trace of copper, manganese, and nickel; it is now redissolved in 6 cc. of hot, strong hydrochloric acid mixed with 6 cc. of hot water, and the filter washed with the minimum of water. The solution and washings are caught in a flask, and should not exceed 50 cc.; to the liquid is added 15 grms. of granulated lead (of such size that it passes a sieve with 20, but remains on one with 40 meshes to the inch), which precipitates all the copper by exchange during a 10 minutes' intermittent agitation at 40° to 50° C. Filter through glass-wool, wash with a little water, evaporate the filtrate down to about 60 cc.; then add 10 cc. of the above-mentioned sodium phosphate solution and ammonia until a precipitate just forms, then add hydrochloric acid until it just redissolves, cool, transfer to a graduated vessel, add 5 cc. of ammonia (sp. gr. 0.90) and dilute to

111.1 cc. After shaking well and standing for 15 minutes, pass through a dry filter-paper, and reserve 100 cc. of the filtrate (equal to 1 gram of the steel) for determination of the nickel. The manganese, with lead from the copper precipitation, is thus separated. The nickel is then determined by electrolysis or volumetrically. For the electrolytic method, the 100 cc. of filtrate is transferred to a 200-cc. platinum dish, and there mixed with 25 cc. of ammonia (sp. gr. 0.90) and 50 cc. of water; after deposition, using a platinum cone as anode, the nickel is washed first with water and then with spirit, and dried 110°. The empty dish should be tared after the removal of the nickel deposit, which is effected by means of 6 cc. nitric acid of sp. gr. 1.2, and subsequent washing with spirit, followed by drying at 110°, in order to imitate as far as possible the conditions under which it was weighed with the nickel. For the volumetric process the 100 cc. of solution is just acidified with hydrochloric acid, and ammonia is then introduced until the deep blue colour of the ammonium nickel salt just disappears, after which an excess of 1 cc. is added, the whole is diluted to 200 cc., and 5 cc. of cupric ferrocyanide indication is run in, followed by standard potassium cyanide (12 grms. to the litre) until the purple colour changes to a clear pale, straw-yellow. After allowing for the cyanide used by the indicator, the nickel percentage is readily calculated. The cyanide solution must be standardised with nickel solution of known strength under similar conditions, as to bulk of solution and ammonia salts, to those obtaining in the analysis. The indicator is made by adding 500 cc. of water containing 2.5 grms. of potassium ferrocyanide, with constant stirring, to a solution formed by dissolving 2.5 grms. of copper sulphate in 25 cc. of water, adding to it ammonium oxalate until the precipitate first formed just redissolves, and then diluting to 500 cc. A precipitate sometimes forms on standing, but is so fine that, if the liquid be thoroughly shaken, it does not affect the accuracy of the estimation. The correction for the indicator is found by titrating 5 cc. to which 200 cc. of water, and six to eight drops of ammonia have been added; it should require about 0.2 cc. of the cyanide solution, of which 1 cc. = 0.0025 Ni. Duplicate results, right to within 0.05 per cent., may be obtained by the volumetric process within three hours of receiving the sample.

—W. G. M.

*Laboratory Notes.* J. S. de Benneville. J. Amer. Chem. Soc. 1894, **16**, 66—67.

In the analysis of ingot copper, the separation of many of the impurities by ammonia is often incomplete, and, moreover, the precipitate tends to pass through the filter: the author therefore dissolves 10 grms. of the metal in nitric acid, evaporates excess of acid, re-dissolves the copper in ammonia, and adds sufficient barium hydroxide to throw down all the minor constituents, the appearance of a scum (of  $\text{BaCO}_3$ ) indicating sufficiency of baryta. The precipitate, which separates well, is filtered and washed with dilute ammonia; it contains all the lead, bismuth, tin, iron, manganese, and antimony present, but not the whole of the arsenic or phosphorus. It may be treated by any known method of separation. The silver, zinc, and cadmium in the filtrate are precipitated by hydrogen sulphide, after decolorisation by potassium cyanide. If the solution of the 10 grms. of copper be diluted originally to 150 cc., but little of the copper ammonium salts separate out, and these dissolve readily in a little warm dilute ammonia, which does not affect the baryta precipitate. With 50 grms. diluted to 500 cc. considerable separation was noted.—W. G. M.

*Experiments on the Analysis of Pig-copper, Brasses, and Bronzes.* J. S. de Benneville. J. Amer. Chem. Soc. **16**, 133—140.

In this paper the author details experiments which he has made in working out his process for the analysis of pig-copper (*vide* preceding abstract). On treating an alloy of tin and copper with nitric acid a slightly excessive percentage of tin was found in all cases quoted, showing that

the metastannic acid was quite insoluble after 12 hours' digestion in the liquid obtained, either by simple dilution to 10 cc. after evaporation to pastiness, by dilution to 150 cc. with 6.92 per cent. nitric acid, by dilution to 10 cc. and with 5 grms. of added ammonium nitrate, by adding 6.52 ammonia, or by adding 5 grms. of ammonium nitrate and 6.52 ammonia. But if phosphorus is present in minute quantities it remains almost completely with the tin, when, however, the proportion of phosphorus to tin is high (*e.g.*, 25:7) much remains in solution, the ratio of  $\text{SnO}_2 \cdot \text{P}_2\text{O}_5$  in the residue averaging, in the case quoted, 1:4. In the case of arsenic, much may remain in solution even when an excess of tin is present. The phosphate and arsenate of tin are not completely decomposed even after a five hours' digestion with alkaline sulphide. When tin is thrown down from cuprous solutions by an excess of ammonia, the precipitate is always contaminated with much copper. Substituting antimony for tin in the experiments, it is found that the separation of antimony is not quite complete either from nitric acid or ammoniacal solution, and that the concentration of phosphorus in the residue is even more incomplete. Referring more directly to his own process, the author finds that while nickel, cobalt, zinc, cadmium, and silver are not at all precipitated, and tin, lead, and iron are completely precipitated either by ammonia or by a mixture of ammonia and barium hydroxide, the phosphorus, which is not separated by the former, is partially precipitated by the latter reagent, and the bismuth, manganese, and antimony which are incompletely thrown down by ammonia, are perfectly separated by the ammoniacal baryta. Arsenic is quantitatively precipitated only in concentrated solutions by the barium mixture, but not at all by ammonia alone. Finally the author finds that the manganese may be separated with sufficiently accurate results by precipitation with nitric acid and potassium chlorate, as in the similar method used for its determination in steel, and that without the removal of the copper from the solution, as had been proposed by Jones.—W. G. M.

*Note on Pemberton's Method of Phosphoric Acid Determination as compared with the Official Methods.* W. C. Day and A. P. Bryant. J. Amer. Chem. Soc. 1894, **16** [4], 282—283.

The authors find that the agreement between the two methods is as close as could be desired. Pemberton's statement that the time required for a single determination is 30—40 minutes from the measuring of the solution for titration, is entirely reasonable.—A. G. B.

*The Determination of Phosphoric Acid.* H. Pemberton, jun. J. Amer. Chem. Soc. 1894, **16** [4], 278—282.

In a previous paper (this Journal, 1894, 176) the author quoted some experiments which showed that 23.2 molecules of  $\text{Na}_2\text{O}$  are required to neutralise an amount of the yellow molybdate precipitate containing 1 mol.  $\text{P}_2\text{O}_5$ . New experiments have shown that this number should be 23 molecules, so that the standard acid for the process should be made by diluting 323.7 cc. of N-sulphuric acid to one litre, and not 326.5 cc., as previously stated.—A. G. B.

*The Chemical and Physical Examination of Portland Cement.* T. B. Stillman. J. Amer. Chem. Soc. **16** [3], 161.

See under IX., page 637.

## ORGANIC CHEMISTRY.—QUANTITATIVE.

*The Titration of Aniline and other Organic Bases with Methyl Orange.* G. Lange. Chem. Ind. **16**, 490.

ATTEN'S experiments on the titration of organic bases with methyl orange are stated by Lange to have been made by

him 10 years ago. The results, however, were found to be satisfactory in the case of quinoline only. The titration of para-quinidine showed only from 97 to 99 per cent., that of aniline from 90 to 94 per cent. of the quantity of base actually present. The bases were dissolved in 25 per cent. strong alcohol; solutions in stronger alcohol could not be titrated at all.—C. O. W.

*The Iodine Number of Rosin.* P. C. Mellbiny. J. Amer. Chem. Soc. 1894, 16, 56--57.

MILLS gives 112.7 as the bromine absorption of rosin, which is equivalent to an iodine absorption of 179, but this does not agree with the iodine numbers of Benedikt (115.7) and Williams (115.3 to 113.3). The author—working with different rosins, using 250 cc. silver a-say bottles with well-ground stoppers, and treating 0.3 gm. of the sample with 10 cc. of chloroform, and an iodine solution containing 25 grms. of iodine and 30 grms. of  $\text{HgCl}_2$  per litre, in a dark place—found an average iodine value of 155.5; but owing to intentional variations in the excess of iodine (equivalent to from 1.9 to 5.2 cc. of decinormal thio-sulphate solution) and in the time of absorption (2 to 52 hours) the actual numbers ranged from 126.7 to 172.6. The greater the excess of iodine and the longer the time of absorption, the higher was the reading. The variations are so great as to disqualify von Hubl's process for use with oils containing rosins.—W. G. M.

*A New Method of Analysing Fats and Resins.* P. C. Mellbiny. J. Amer. Chem. Soc. 1894, 16 [4], 275--278.

THE following process has been devised for determining the amount of bromine which oils and resins can absorb by addition ("bromine addition figure"), and at the same time the amount of bromine which replaces hydrogen ("bromine substitution figure"), in a given time. It depends upon the fact that bromine, in forming substitution products, liberates a molecule of hydrobromic acid for every atom of bromine which replaces hydrogen, whilst in forming additive compounds no hydrobromic acid is liberated.

From 0.25 to 1.00 gm. of the substance is dissolved in 10 cc. of carbon tetrachloride in a 500-cc. bottle. An excess of a solution of bromine in carbon tetrachloride ( $\frac{N}{3}$ ) is added, and the bottle kept in the dark for 18 hours. At the expiration of this period the bottle is placed in ice to form a partial vacuum by cooling the gaseous contents, and a piece of rubber-tubing is slipped over the neck so as to form a well around the stopper. This well is filled with water, and the stopper is carefully lifted, so that the water may be sucked into the bottle and absorb the hydrobromic acid present. When about 25 cc. of water have been added, the bottle is well shaken and 10--20 cc. of a 20 per cent. solution of potassium iodide and about 75 cc. more water are added. The liberated iodine is titrated with  $\frac{N}{10}$  sodium thio-sulphate. The contents of the bottle are transferred to a separator, the aqueous portion separated, and filtered through a cloth. A few drops of thio-sulphate are added to the aqueous solution if it be blue, and the liquid is titrated with  $\frac{N}{10}$  potassium hydroxide solution, methyl orange being used as an indicator. It is better to add an excess of the alkali and to titrate back.

The total bromine absorption per cent. is calculated from the difference between the bromine equivalent to the liberated iodine and the total bromine used. The bromine substitution figure is calculated from the amount of hydrobromic acid found by the titration, and returned as a percentage on the oil taken; twice this value subtracted from the total bromine absorption gives the bromine addition figure.

The following results are quoted:—

	Total Bromine Absorption in 18 Hours.	Bromine Addition Figure.	Bromine Substitution Figure.
W.G. rosin.....	212.70	0	106.35
E. rosin.....	206.50	0	103.25
Second rosin oil (a).....	116.20	0	58.10
" " (b).....	114.70	0	57.35
American raw linseed oil...	102.88	102.88	0
Same oil boiled.....	103.92	103.92	0
White salad cotton-seed oil..	65.54	64.26	0.64
Sperm oil.....	56.60	54.52	1.04

—A. G. B.

*Solubility Tests of Dark Mineral Lubricating Oils.* Holde. Mitt. K. Tech. Versuchsanst. 11 [5], 261--272.

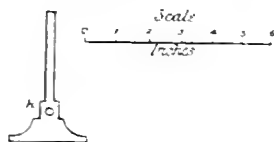
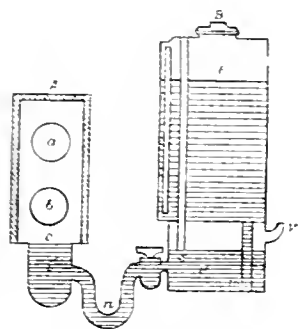
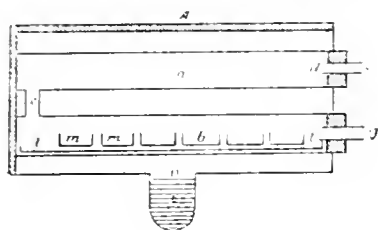
IN a paper published in 1890 (311), the author described how certain dark mineral oils, when treated with petroleum spirit, gave a dark precipitate, soluble in benzene, which he identified as asphalt. In the present paper he further investigates the same subject. He had found that the specific gravity and boiling point of the petroleum spirit used as a solvent, greatly influenced the solubility of the asphalt, and great care had to be devoted to this point, as the previous experiments had shown that the asphalt, that would separate out from these oils, was in solution, or partly so, in the crude petroleum. A number of preliminary tests were therefore made of the solubility of the oils in question, in petroleum spirit. The results of these tests are given in tabular form in the paper.

They showed that the amount of deposit formed depended very largely on the specific gravity of the petroleum spirit used, and on the time the solution was allowed to stand. Oils which showed turbidity after standing for ten minutes, were frequently quite clear at first. Generally speaking, the more asphalt an oil contains the sooner the precipitate shows itself. All the oils experimented on in these tests were clearly soluble in the petroleum spirit used. It may be said that the larger the quantity of solvent added to the oil, the larger the volume of the deposited matter, and that filtering has no perceptible effect on this volume. The author considers that it may be generally assumed that the asphalt exists in a dissolved state in dark mineral oils rich in that substance, and that the dark particles observable in certain oils of this class are probably particles of solid paraffin coloured dark by the oil itself. The influence of the addition of asphalt on the physical properties of the oils, was also investigated, and it was found that it produces a certain thickening of the oil, an increase in the specific gravity, and a strong coloration. The effect of various quantities of asphalt on the viscosity is, however, but small. The addition of asphalt does not injuriously affect the behaviour of mineral lubricating oils at ordinary temperatures. When thin layers of asphalt-containing oils were subjected to a temperature of 100° C. for 10 hours the following effects were noticed. Oils containing but little asphalt, or none, remained liquid; those containing from 1.3 per cent. to 2.3 per cent. became sticky and thick; whilst oils containing 5 per cent. to 6 per cent. were converted into a thick resin-like mass. All these oils, however, remained liquid when the temperature was 50° C. instead of 100° C. For railway purposes, the percentage of asphalt must be small, or the oils are liable to solidify at low temperatures and to dry up at high ones. The author suggests the following conditions:—(1.) The oil should form a clear solution with benzol (which dissolves all the asphalt present). (2.) That petroleum spirit of a sp. gr. of 0.64 should not form a greater quantity

of deposit or precipitate with unfiltered than with filtered oil (thus the presence of suspended particles of asphalt is guarded against); and he is of the opinion that the testing of dark-coloured mineral oils for lubricating purposes, by examining the quantities of asphalt deposited by petroleum spirit, from small samples of both filtered and unfiltered oil, is desirable.—R. B. P.

*A Rapid and Accurate Method of Determining the Moisture and Oil in Linned Cakes and other Feeding-Stuffs.* A. P. Aitken. J. Amer. Chem. Soc. **16**, 114—122.

THE author believes that the discrepancies between different determinations of water in feeding-stuffs are mainly due to the low and comparatively irregular temperatures obtaining in the ordinary water-oven, and to the varying oxidation of oil in the sample. He therefore heats the material in a steam bath in a current of dry coal-gas (or hydrogen). The apparatus is shown in longitudinal and cross-section in the figure. It consists of a box 10 in.  $\times$  4 in.  $\times$  2 in., with a



small well below, and carrying a pair of horizontal tubes, such as are used for telescopes, running lengthwise, one above the other, but connected together at one end of each, which end is otherwise closed; at the top of the box is a  $\frac{3}{16}$ -in. hole (not shown) for the escape of steam. Connected with the box is an automatically-regulated supply-cistern B, so arranged that when the water level in the lower chamber sinks, air is drawn in through *v*, and the bottom of the tube *s* being uncovered, a bubble of air finds its way into the hermetically-sealed top of the upper reservoir, and displaces a corresponding

volume of water, which passes through the tube *a* into the lower reservoir. The sample to be examined (generally 1 gm.) is weighed into an aluminum boat *m*, which, with five similar boats, is placed on a tray *l* and introduced into the lower heating-tube of the steam bath. Coal gas, dried by passing over calcium chloride, is then passed through *d* into the upper tube, where it becomes heated, and then, after traversing the row of boats in the lower tube, is finally led off through *q* to the Bunsen burner *h*, and is there utilised for the generation of the steam required to heat the apparatus. The box is constructed of copper, but is jacketed throughout with  $\frac{1}{2}$  in. asbestos sheeting. The samples are cooled in desiccators and weighed, and may then be used for the determination of oil. The extraction of the oil is effected by ether, the sample being placed in a polished brass tube  $\frac{1}{2}$  in. in diameter and 4 in. in length, contracted at one end to a continuation tube  $\frac{1}{8}$  in. in diameter and 1 in. long. Before introducing the material into this tube, a disc of re-gauze, followed by a double tightly fitting wad of filter paper, should be introduced into the wide tube and pressed down to the contracted end; a second wad of paper only is finally placed above the sample. When oil-cake is being tested the gauze may be omitted. The extraction is effected with boiling ether with the aid of a specially constructed water-bath; this consists of a zinc box, somewhat shallower than the length of the extraction-tube, with a number of vertical copper tubes, running through from top to bottom and soldered in at both ends; each tube has a slight shoulder at the bottom, sufficient to catch and support the wide portion of one of the extraction-tubes, which must just fit into it. A gentle circulation of warm water is maintained in the box with the aid of an upper and a lower lateral tube soldered through one side of the bath. Below this box is a second water-jacketed vessel with a stop-cock at the bottom, and with perforations in its lid corresponding to the tubes in the upper box, through which pass the contracted portions of the extractors; a circulation of cold water is maintained in the jacket around the box, which thus serves to retain the ethereal solution of oil. In use, the tube containing the sample is dropped into one of the tube-sockets of the water-bath and is then filled with ether to within an inch of the top, and firmly corked. The pressure produced on boiling forces the ether through the material; it must then be renewed 10 (or even 20 times) until a drop yields no residue on evaporation. Finally the sample and wads are pushed out on to a watch-glass, the paper is removed, and the material dried at 100° and re-weighed. With very fine powders a half-turn screw-cap with a cork washer is substituted for the cork during extraction, in order to resist the pressure of vapour. The oil extracted is thus measured by loss of weight.—W. G. M.

*Determination of the Crystallisable Sugar in Beetroots.*

H. Pellet. J. Amer. Chem. Soc. 1894, **16** [4], 266—273.

A SUITABLE amount of the rasped pulp is weighed into a flask of a definite volume (26.048 grms., made up to 201.35 cc., are taken for the German instruments), and 5—7 cc. of lead subacetate (30 B.) and a little ether, to reduce the foam, are added; the flask is shaken and the volume made up. The solution is filtered and its rotatory power read. Sources of error are (1) the use of insufficient lead subacetate; and (2) the retention of air bubbles by the pulp, due to insufficient use of ether or insufficient agitation.

THE Lindeboom "sound," devised to extract cylinders of approximately the same weight from the roots, and Hanriot's machine for pulping the cylinders, are described as greatly expediting the work of a laboratory for the analysis of mother-beets. A "continuous tube" for polarimeter readings is used by the author; it consists of an ordinary tube with a side tube at each end; the sugar solution is displaced by water run through the tube, and the water is in its turn replaced by the next sugar solution, the disappearance of striae in the liquid being the indication of complete replacement.—A. G. B.

*Detection of Salicylic Acid in Food.* K. P. McElroy, J. Amer. Chem. Soc. 1894, 16 [3], 198-203.

Fitt. method at one time used by the author for the detection of salicylic acid in tinned vegetables, was carried out as follows:—The contents of the tin were triturated in a mortar, water and a little phosphoric acid being added, the mixture strained through a bag, and the filtrate distilled. The distillate was collected in fractions and tested with ferric chloride solution (5 mgrms. per cc.). As an illustration of the course of the volatilisation of salicylic acid in a current of steam, the following experiment may be quoted: 25 mgrms. of salicylic acid were dissolved in 250 cc. of water containing a little phosphoric acid and distilled, the distillate being collected in 25-cc. portions and the salicylic acid estimated colorimetrically. Fractions 1 and 2 gave a slight but distinct reaction; fraction 3 contained 0.3 mgrm. of salicylic acid; No. 4, 0.1 mgrm.; No. 5, 0.5 mgrm.; No. 6, 0.8 mgrm.; No. 7, 0.9 mgrm.; No. 8, 2.2 mgrm.; No. 9, 3.0 mgrms. The total quantity which distilled over was therefore 8.1 mgrms. A similar method is described by Ince for the examination of wine. 210 cc. of the wine are mixed with 10 cc. of dilute sulphuric acid and the mixture distilled, the distillate being collected in portions of 50 cc. The first portion is thrown away and the salicylic acid estimated colorimetrically in the next two. The result is multiplied by 8 and the product returned as the total content of salicylic acid. After the examination of various samples of tinned vegetables by his method described above, the author repeated the examination, taking the residual contents of the tins, which had, in the meantime, been dried and powdered finely enough to pass a 100-mesh sieve. Portions of this powder were made into a stiff dough with dilute sulphuric acid and extracted with ether. The ethereal extract was evaporated to dryness and the residue taken up with water and distilled. The distillate gave a stronger reaction than did the original trial by direct distillation, and in some cases salicylic acid was detected where it had previously been overlooked. This difference probably arose from the extraction of salicylic acid, in the second series of trials, from the solid part of the samples, while in the first only the juice was used. In some cases the quantity of salicylic acid found was so small that it appeared doubtful whether it could have been added as a preservative. This doubt is, however, resolved by a fact observed by Kolbe (J. Prakt. Chem. 1880, 21, 443, and 22, 112), who found that salicylic acid completely disappeared from water and wine contained in casks, whereas it remained in the same liquids kept in glass vessels. Four months' standing was enough to cause this disappearance in some cases. He proved that wood not only removed salicylic acid, but destroyed it, as the acid could not be recovered from test-cubes of wood immersed in solutions of salicylic acid. It is possible that the woody tissue of tinned vegetables may act similarly. The ordinary method of slaking out with ether is used by the author in the examination of beer and wine. The distillation process is cheaper and quicker than the method of extraction, and the reaction with ferric chloride is sharper, although less salicylic acid is obtained. The reason for this is that less impurity accompanies the distilled than the extracted acid.

The objection raised to the distillation process, that in the process of fermentation (e.g., of beer) certain phenol-like bodies are formed which interfere with the reaction for salicylic acid, would, if substantiated, apply equally to the extraction method. It has also been alleged that so-called "caramel malt" contains a substance capable of giving all the reactions characteristic of salicylic acid, with the exception of the reaction with Millon's reagent. Experiments made by the author on such malts gave negative results.

—B. B.

## ANALYTICAL AND SCIENTIFIC NOTES.

*Spontaneous Combustion of a Lamp-Shade.* A. Dupré, Times, May 23, 1894.

The writer states that on May 10th, a smell of burning being noticed in his house, search at length revealed the fact that a paper lamp-shade in one of the rooms had been

entirely consumed by fire. No damage was done, as no inflammable body was near the shade at the time. For two days prior to the accident the lamp had not been lighted, and there had been no fire in the room. Since morning, when the shade was intact, no one had even entered. The shade, made about a year ago from so-called crinkled tissue paper, consisted of one white and one yellow sheet, gathered into a bunch at the top, and spreading thence over the frame below. On examination, the yellow paper was found to be coloured by chromate of lead, and the author considers that the spontaneous ignition of the shade was due to this body, though he has not yet been able to reproduce the occurrence under experimental conditions. The dangerous paper is readily recognised by setting fire to a piece of it, and blowing out the flame. Should it behave like a piece of ignited touch-paper it is probably a chromate paper, and the writer considers it to be dangerous. Pale green papers, and possibly others, also contain chromate of lead.

—C. G. C.

*The Spontaneous Combustion of Lamp-Shades.* Chemical Trade Journal, 370, 404.

*Appropos* of the observations of Dr. Dupré on this question, Messrs. Crompton and Bros., paper-makers, of Elton, near Bury, Lancashire, write to the *Times*, stating that whilst they do not doubt that the writer of the letter is quite sincere in believing that the lamp-shade in his house fired spontaneously, they cannot believe such a thing to be possible. They state that since reading the letter referred to they have made numerous experiments with many of the various colours of the papers they make, and particularly with the chrome colours, and in no single instance could they get any one of them to fire spontaneously. They had the papers in question heated in a heating-chamber to nearly 200° F., with the result that in every case the papers when taken therefrom remained in their original uninjured condition, notwithstanding their being subjected to the heat named for several hours. They also saturated the papers with chromic acid and dried them, and even after this severe test failed to get them to fire spontaneously at the heat named, or under similar circumstances.

*The Odours of Benzoic Acids, with Remarks on other Odorous and Inodorous Bodies.* J. Passy, Comptes rend. 1894, 118, 481-482.

In a preceding note (Comptes rend. May 1, 1893) the author has shown that in proceeding down the series of normal fatty acids the odour regularly diminishes until, at the 14th term, it permanently vanishes, alike in the acid and in its aldehyde, alcohol, and ethereal salt derivatives. But with benzoic acid it is the acid alone which is inodorous in the pure state. Working with carefully purified benzoic acid prepared by four different methods, he finds that the crystalline acid is quite inodorous, but that if its solution in water or in alcohol be warmed, it at once evolves the characteristic benzylic smell of the other compounds. The same smell is recognisable if a 0.1 per cent. solution of benzoic acid containing 0.6 per cent. of sodium chloride at blood temperature be used as a nasal douche (Aronsohn's method). Inodorous bodies may thus be classed either as those which are always so, and those which under the conditions of the experiment have not passed into the "odorous condition." In its behaviour, benzoic acid is like vanillin, coumarin, and other perfumes, which only emit their characteristic odours at temperatures at which they are appreciably volatile.—W. G. M.



## New Books:

**SYSTEMATIC SURVEY OF THE ORGANIC COLOURING MATTERS.** By DR. G. SCHULTZ and P. JULIUS. Translated and edited, with extensive additions, by ARTHUR G. GREEN, F.I.C., F.C.S. London and New York: Macmillan and Co. 1894. Price 21s.

This is a translation, with very extensive additions, of the work of Messrs. Schultz and Julius, the appearance of which in the original German, was reported in this Journal, 1891, 662. It commences with the editor's preface, in which is set forth the fact that such a work as this would be of inestimable value not only to the technical chemist, but to the dyer, analyst, merchant, patent-agent, &c. For this country and America, the value of the work has been much enhanced by the addition by Mr. Green of about 50 new colours which have made their appearance since the publication of the German edition, and also by the references which have been given to British and American patents whenever these exist.

Page 1 to 57 is occupied by a concise and very instructive account of those coal-tar derivatives and compounds of the fatty series that are largely used in the coal-tar colour industry. This preliminary treatise forms the prelude to the tabular system of colouring matters, which extends from page 60 to page 189. The Translator and Editor's Appendix then follows with 21 new colours, and thereafter follow Tables for the Qualitative Analysis of the Artificial Colouring Matters, the first for Dyestuffs soluble in water and the second for those insoluble. The work closes with two Alphabetical Indexes, the first referring to the pages of the book, the second to the numbers indicating the position in the tables. The book is strongly bound in cloth and forms a volume of large 8vo size.

**JAHRES-RUNDSCHAU ÜBER DIE CHEMISCHE INDUSTRIE UND DEREN WIRTSCHAFTLICHE VERHÄLTNISSE FÜR DAS JAHR 1893, EIN ÜBERSICHTLICH GEORDNETER BERICHT ÜBER DIE FORTSCHRITTE DER CHEMISCHEN GROSS- UND KLEIN-INDUSTRIE, SO WIE ÜBER ANALYTISCHE METHODEN.** Unter Mitwirkung von Fachmännern. Herausgegeben von Dr. ADOLF BENDER. Vienna, Pest, and Leipzig: A. Hartleben's Verlag. London: H. Grevel and Co., 33, King Street, Covent Garden.

This work appears in four parts. Part I., containing 71 woodcuts, price 6s., and treating of Fuel; Metallurgy; Technology of Water; Manufacture of Inorganic Acids, Bases, and Salts; Earthenware; Cements and Artificial Stone; Glass; Manures; and the Apparatus and Machinery of Laboratories and Works. Part II., with 35 woodcuts, price 6s., and treating of Starch, Starch sugar, &c.; Beet-sugar, Cane-sugar, &c.; Milk, Butter, and Cheese; Flour, Bread, Preserves, Spices, &c.; Spirit Manufacture; Brewing, Wine; Sanitation, Disinfection, Medicaments, &c. Part III., with 24 woodcuts, price 3s., and treating of Organic Colouring Matters; Dyeing, Calico-printing, &c.; Tanning; Paper Manufacture. Part IV., with 26 woodcuts, price 3s., and dealing with Ethereal Oils and Resins; Fats and Mineral Oils; Lakes; Explosives; Photography; Adhesives and Cements. The four volumes or parts are bound in paper covers, and are of 8vo size, and each contains its own Table of Contents and Alphabetical Index. The whole collective work may also be obtained at the price of 18s.

**MICRO-ORGANISMS IN WATER.** Their Significance, Identification, and Removal, together with an account of the Bacteriological Methods employed in their Investigation. Specially designed for the use of those connected with the Sanitary Aspects of Water Supply. By PERCY FRANKLAND, Ph.D., F.R.S., and MRS. PERCY FRANKLAND. Longmans, Green and Co., London, and New York, 15, East 16th Street. 1894. Price 16s.

This is a large 8vo volume bound in cloth, and containing 320 pages of subject-matter, illustrated with 28 wood-engravings and 20 plates. The existing literature of the

Bacteriology of Water being both extensive and scattered, the authors have deemed it an urgent necessity to collate and condense this literature. This they have endeavoured to do in the present work, besides making numerous additions in the shape of the results of investigations of their own. A chapter has also been added on the comparatively novel subject of the Bactericidal Action of Light.

The leading themes treated of in the chapters of this work are as follows:—

I. The Sterilisation and the Preparation of Culture Media. II. The Staining and Microscopic Examination of Micro-organisms. III. The Examination of Water for Micro-organisms. IV. The Bacterial Contents of various Waters. V. The Purification of Water for Drinking Purposes. VI. The Multiplication of Micro-organisms. VII. The Detection of Pathogenic Bacteria in Water. VIII. The Vitality of Particular Pathogenic Bacteria in Different Waters. IX. The Action of Light on Micro-organisms in Water and Culture Media. X. An Appendix containing Tabular Descriptions of the various Micro-organisms found in Water. These comprise upwards of 200, and the descriptions thus given include the principal characters of all the micro-organisms, so far as the authors have been able to ascertain, as yet found in water.

**JAHRESBERICHT ÜBER DIE LEISTUNGEN DER CHEMISCHEN TECHNOLOGIE, MIT BESONDERER BERÜCKSICHTIGUNG DER GEWERBESTATISTIK FÜR DAS JAHR 1893.** Von Dr. FERDINAND FISCHER. Mit 200 Abbildungen. Leipzig: Verlag von Otto Wigand. London: H. Grevel and Co., 33, King Street, Covent Garden. Price, 21s.

This general report of the advance and condition of chemical technology for the year 1893, especially in the German Empire, has now appeared from the press. If the magnitude of that advance could receive any due measurement in accordance with the increase of the literature of the subject, the statement would not be without significance, that whereas the volume of Fischer's Jahresbericht issued for 1892, contained 1124 pages of text, the present issue for 1893 contains upwards of 1200 pages. In a work of this kind, one essentially of reference, the indexes must be of the most complete and searching character, and in this 1893 issue of the Jahresbericht we find these extending from page 1210 to 1272, i.e., over 62 pages. The index of authors fills 28, that of subject-matter 24, and the Index of German Patents 9 pages of the large 8vo volume. The text is illustrated with 200 engravings referring to plant and apparatus, and the treatment of the whole subject may be gathered from the following epitome of the sub-divisions of the same:—GROUP I. CHEMICAL TECHNOLOGY OF FUEL (pages 1 to 182). GROUP II. METALLURGY (pages 183 to 369). GROUP III. CHEMICAL MANUFACTURES: INORGANIC (pages 370 to 510). GROUP IV. CHEMICAL MANUFACTURES: ORGANIC (pages 511 to 677). GROUP V. GLASS, POTTERY, and EARTHENWARE, CEMENTS, ARTIFICIAL STONE (pages 677 to 765). GROUP VI. FOODSTUFFS (pages 766 to 973). GROUP VII. CHEMICAL TECHNOLOGY OF THE TEXTILE FIBRES (pages 974 to 1070). GROUP VIII. OTHER ORGANIC CHEMICAL INDUSTRIES (pages 1071 to 1175). GROUP IX. MECHANICAL AIDS AND APPARATUS FOR CHEMISTS (pages 1175 to 1299).

A comparison indicates that the literature of the following branches in the Jahresbericht for 1893 shows a marked increase over that in the Jahresbericht for 1892, which again indicates increased scientific and inventive activity in those branches:—Groups I., II., V., VII., and VIII., the increase of Group VIII. being by far the largest. Group VIII. includes Fats and Lubricants; Fat Acids, Soaps, &c.; Varnishes and Paints; India-rubber; Tanning, Glue, &c.; Manures, &c.; and the Preservation of Wood.



## Trade Report.

## TARIFF CHANGES AND CUSTOMS REGULATIONS.

## CANADIAN TARIFF CHANGES.

(From the Board of Trade Journal.)

A communication has been received at the Board of Trade enclosing copies of the tariff resolutions taking effect from the 27th March last. The following are some of the changes effected:—

Articles.	Rates of Duty.
<i>Alcohol, Spirits, &amp;c.</i>	
Spirituous or alcoholic liquors, distilled from any material, or containing or compounded from or with distilled spirits of any kind, and any mixture thereof with water, for every gallon thereof of the strength of proof, and when of a greater strength than that of proof, at the same rate as the increased quantity that there would be if the liquors were reduced to the strength of proof. When the liquors are of a less strength than that of proof, the duty shall be at a rate herein provided, but computed on a reduced quantity of the liquors in proportion to the lesser degree of strength; provided, however, that no reduction in quantity shall be computed or made on any liquors below the strength of 15 per cent. under proof, but all such liquors shall be computed as of the strength of 15 per cent. under proof as follows:—	
(a.) Ethylalcohol, or the substance commonly known as alcohol, hydrated oxide of ethyl or spirits of wine, gin of all kinds, N.E.S.; rum, whisky, and all spirituous or alcoholic liquors, N.O.P.; amyl alcohol or fusel oil, or any substance known as potato spirit or potato oil, methyl alcohol, wood alcohol, wood naphtha, pyroxylic spirit, or any substance known as wood spirit or methylated spirits, absinthe, arrack or palm spirit, brandy, including artificial brandy and imitations of brandy; cordials and liqueurs of all kinds, N.E.S.; mescal, pulque, rum, shrub, schiedam, and other schnapps; tafia, angostura, and similar alcoholic bitters or beverages.	Per gallon 2 dols. 12½ cts.
(b.) Spirits and strong waters of any kind, mixed with any ingredient or ingredients as being or known or designated as anodynes, elixirs, essences, extracts, lotions, tinctures, or medicines, N.E.S.	Per gallon 2 dols. 12½ cts. and 30° ad val.
(c.) Alcoholic perfumes and perfumed spirits, bay-rum, Cologne and lavender waters, hair, tooth, and skin washes, and other toilet preparations containing spirits of any kind, when in bottles or flasks weighing not more than four ounces each.	Per gallon 2 dols. 12½ cts. and 40° ad val.
When in bottles, flasks, or other packages weighing more than four ounces each.	Per gallon 2 dols. 12½ cts. and 30° ad val.
(d.) Nitrous ether, sweet spirits of nitre, and aromatic spirits of ammonia.	
<i>Animal Products.</i>	
Starch, including farina, corn starch, or flour, and all preparations having the qualities of starch; the weights of the package to be in all cases included in the weight for duty.	Per lb. 1½ cents.
Lard, hard compound, and similar substances, cotton-lene, and animal stearine of all kinds, N.E.S.	" 2 cents.
Tallow, stearic acid.	20° ad val.
Beeswax.	10° ad val.
Candles, N.E.S.	25° ad val.
Soap, N.E.S., pear-line and other soap powders, pumice, silver, and mineral soaps, sapolio, and like articles.	35° ad val.
Soap, common, or laundry, not perfumed.	Per lb. 1 cent.
Castile soap, mottled or white.	" 2 cents.
Glue and mucilage.	25° ad val.

## CANADIAN TARIFF CHANGES—cont.

Articles.	Rates of Duty.
Yeast cakes and baking powders, the weight of the package to be included in the weight for duty.	Per lb. 6 cents.
<i>Chemicals, Oils, and Paints.</i>	
Acid, acetic and pyroligneous, N.E.S., and vinegar, for any strength not exceeding the strength of proof.	Per gall. 15 cents.
For each degree of strength in excess of the strength of proof.	2 cents additional.
The strength of proof shall be held to be equal to 6 per cent. of absolute acid, and in all cases the strength shall be determined in such manner as is established by the Governor in Council.	
Acid, acetic, and pyroligneous of any strength, when imported by dyers, calico-printers, or manufacturers of acetates or colours, for exclusive use in dyeing or printing, or for the manufacture of such acetates or colours in their own factories under such regulations as are established by the Governor in Council.	25° ad val.
Glacial acetic acid or acetic acid exceeding the strength of proof, when imported by druggists and other than dyers, calico-printers, or manufacturers of vinegar or acetates or colours, to be used in their own factories for purposes of manufacture, other than as heretofore excepted from this provision, for the strength of proof.	Per gall. 15 cents.
For each degree of strength in excess of the strength of proof.	1 cent. additional.
Acid, muriatic and nitric, and all mixed acids.	20° ad val.
Acid, sulphuric.	Per lb. 1½ cent.
Sulphuric ether.	" 5 cents.
Acid, phosphate.	" 2 cents.
All medicinal, chemical, and pharmaceutical preparations, when compounded of more than one substance, including patent and proprietary preparations, tinctures, pills, powders, troches, lozenges, syrups, cordials, bitters, anodynes, tonics, plasters, liniments, salves, ointments, pastes, drops, waters, essences, and oils, not otherwise provided for:	
All liquids.	50° ad val.
All other.	25° ad val.
Provided that this item shall not be held to include drugs and preparations recognised by the British and the United States Pharmacopoeia as official.	
Cod-liver oil medicated.	20° ad val.
Oils, essential.	10° ad val.
Pomades, when imported in tins of not less than 10 lbs. each.	15° ad val.
Perfumery, including toilet preparations (non-alcoholic), viz.:—Hair oils, tooth and other powders and washes, pomatus, pastes, and all other perfumed preparations, N.O.P., used for the hair, mouth, or skin.	30° ad val.
Illuminating oils, composed wholly or in part of the products of petroleum, coal, shale, or lignite, costing more than 30 cents per gallon.	25° ad val.
Oils, coal, and kerosene distilled, purified, or refined; naphtha and petroleum, N.E.S., products of petroleum, not elsewhere specified.	Per imp. gallon 6 cents.
Lubricating oils, composed wholly or in part of petroleum, and costing less than 25 cents per imperial gallon.	Per imp. gallon 6 cents.
Crude petroleum, fuel and gas oil (other than naphtha, benzine, or gasoline), when imported by manufacturers other than oil refiners for use in their own factories, for fuel purposes and for the manufacture of gas.	Per gallon 3 cents.
Paraffin wax.	Per lb. 2 cents.
Paraffin wax candles.	" 4 cents.
British gum, dextrine, sizing cream, and enamel.	10° ad val.
Lubricating oils, N.E.S., and axle grease.	25° ad val.
Barrels containing petroleum or its products, or any mixture of which petroleum forms a part, when such contents are chargeable with a specific duty.	20 cents each.

CANADIAN TARIFF CHANGES—*cont.*

Articles.	Rates of Duty.
Linseed or flaxseed oil, raw or boiled, lamp oil, neat-foot oil, and sesame seed oil.	20 <sup>°</sup> <i>ad val.</i>
Olive oil prepared for salad purposes.	30 <sup>°</sup> <i>ad val.</i>
Vaseline and all similar preparations of petroleum for toilet, medicinal, or other purposes.	35 <sup>°</sup> <i>ad val.</i>
Blacking, shoe, and shoemakers' ink, and shoe harness, and leather dressing, and harness soap.	25 <sup>°</sup> <i>ad val.</i>
Ink for writing.	20 <sup>°</sup> <i>ad val.</i>
Blueing, laundry blueing of all kinds.	25 <sup>°</sup> <i>ad val.</i>
Dry white and red lead, orange mineral, and zinc white.	5 <sup>°</sup> <i>ad val.</i>
Ochres, ochrey earths, raw siennas, and colours, dry, N.E.S.	20 <sup>°</sup> <i>ad val.</i>
Oxides, dry fillers, fire proofs, umbers, and burnt siennas, N.E.S.	25 <sup>°</sup> <i>ad val.</i>
Paints and colours, rough stuff and fillers, N.E.S.	25 <sup>°</sup> <i>ad val.</i>
Paints and colours ground in spirits and all spirit varnishes and lacquers.	Per gallon 1 dol.
Turpentine, spirits of.	5 <sup>°</sup> <i>ad val.</i>
Varnishes, lacquers, japans, japan driers, liquid driers, and oil finish, N.E.S.	Per gall. 20 cents and 20 <sup>°</sup> <i>ad val.</i>
Paris green, dry.	10 <sup>°</sup> <i>ad val.</i>
Putty.	15 <sup>°</sup> <i>ad val.</i>

*Minerals.*

Asbestos in any form other than crude and all manufactures thereof.	25 <sup>°</sup> <i>ad val.</i>
Coal, bituminous.	Per ton of 2,000 lbs. 50 cents.
Coal dust.	20 <sup>°</sup> <i>ad val.</i>
Plumbago.	10 <sup>°</sup> <i>ad val.</i>
Plumbago, all manufactures of, N.E.S.	25 <sup>°</sup> <i>ad val.</i>
Electric light carbons or carbon points, not exceeding 12 inches in length.	Per 1,000, 2 dols. 50 cts. and in proportion for greater or less lengths.
Salt, fine, in bulk, and coarse salt, N.E.S.	Per 100 lbs., 5 cts.
Do. in bags, barrels, or other packages, the bags, barrels, or packages to bear the same duty as if imported empty.	Per 100 lbs., 7½ cts.

*Miscellaneous.*

Albumenised and other papers chemically prepared for photographers' use.	35 <sup>°</sup> <i>ad val.</i>
Glass carboys, empty or filled, and phials.	30 <sup>°</sup> <i>ad val.</i>
Common window glass.	20 <sup>°</sup> <i>ad val.</i>
Coloured glass and rolled plate.	25 <sup>°</sup> <i>ad val.</i>
Plate glass, not coloured, in panes of not more than 12 sq. ft.	Per sq. ft. 4 cents.
Plate glass, in panes not more than 30 sq. ft.	" 6 "
" " " 70 sq. ft.	" 8 "
" " " over 70 sq. ft.	" 9 "
When bevelled, in each case.	2 cents extra per sq. ft.
Silvered glass, N.E.S.	30 <sup>°</sup> <i>ad val.</i>
" " bevelled.	35 <sup>°</sup> <i>ad val.</i>
Stained glass windows.	30 <sup>°</sup> <i>ad val.</i>
All other glass manufactures.	20 <sup>°</sup> <i>ad val.</i>
Cement.	Per barrel 40 c.
Plaster of Paris, uncalcined.	15 <sup>°</sup> <i>ad val.</i>
" " calcined.	Per barrel of 300 lb. 10 c.
All manufactures of leather, N.E.S.	25 <sup>°</sup> <i>ad val.</i>
Celluloid in the rough.	10 <sup>°</sup> <i>ad val.</i>
Copper wire.	15 <sup>°</sup> <i>ad val.</i>
Zinc, N.E.S.	25 <sup>°</sup> <i>ad val.</i>

CANADIAN TARIFF CHANGES—*cont.*

Articles.	Rates of Duty.
Phosphoric acid.	10 <sup>°</sup> <i>ad val.</i>
Lead.	Per 100 lb. one.
Chrome steel.	15 <sup>°</sup> <i>ad val.</i>
Nickel plated steel.	10 <sup>°</sup> <i>ad val.</i>

GENERAL TRADE NOTES.

SALT IN THE ISLE OF MAN.

According to Professor Boyd Dawkins, the prospects of the salt industry being established in the Isle of Man are sound, to say the least, and without being unduly sanguine, it may be added promising.

The fact of salt being worked at Preeval, and across the Irish Channel at Carrickfergus, caused geologists, not unnaturally, to wonder whether a submerged bed of salt connected the two districts. If that were so, it would by a natural sequence follow that salt should exist in the Isle of Man. At different times since 1886, Professor Dawkins has been endeavouring to ascertain if geological data could be got together to substantiate this conjecture. From the communication he has made this week to the Geological Society at Manchester, he seems to have been on the whole successful.

His geological investigations tend to show that North-Eastern Island, the north of the Isle of Man, and the English Lake District constitute one vast marine basin, from which the Triassic salt-bearing marls crop out at Barrow, the Isle of Man, and again at Carrickfergus, and are linked together by the deposits submerged in the Irish Sea. Borings have shown that the marls worked at Barrow and Carrickfergus are identical, and quite lately borings in search of coal rather than salt, made in the north of the Isle of Man have revealed such information touching the lay of the salt-bearing marls, as to give rise to the prospect of the salt industry being established in that island.

The bed of salt, as far as it has been investigated, is thinner than that which occurs at Dunerue, near Carrickfergus; but a boring at the Point of Ayre has resulted in the discovery of a bed over 31 ft. thick, the bore hole also intersecting a brine run 2 ft. 6 in. deep. As further confirmation of the possibilities of this industry being developed in the island, the fact may be cited that the sectional details revealed by the boring at the Point of Ayre, agree almost exactly with those which have been recorded about the saliferous marls of Dunerue; from which district, according to the latest statistics of the mineral production of the United Kingdom, close on 55,000 tons of rock salt are produced annually.—*Chemical Trade Journal*.

SUPPLIES OF CACUCHOUC IN BOLIVIA.

The French Chargé d'Affaires at La Paz learns from Santa Cruz that new clusters of *ficus elastica* have just been discovered on the rivers Verde, Tarvo, Itenes, San Martín, Paragua, and their tributaries. From Santa Cruz to Rio Paragua is a distance of 119 leagues, which may be covered in a fortnight. The road is flat and practicable for carts traversing numerous farms. Of this route 22 leagues is through desert country, and it is essential to mark out the ground from a point called Riaves as far as Rineonada, where the first acacias, so very rich in gum, are to be found. Rineonada (on the Rio Paragua) will be the depot for the leaves or sticks of *borrechu*. The route for exporting this product terminates at Puerto Suarez, situated at a distance of 165 leagues. This may be covered in 25 days, through a country almost level and practicable for carts, but with a scarcity of water and pasture land.—*Board of Trade Journal*.

ELEVENTH REPORT OF THE COMPTROLLER-GENERAL OF  
PATENTS, DESIGNS, AND TRADE MARKS; WITH  
APPENDICES FOR THE YEAR 1893. (C.—7386.)  
Price 2½d.

This annual report is issued by the Comptroller of the Patent Office in pursuance of the requirements of the 102nd section of the Patents, Designs, and Trade Marks Act, 1883 (45 & 47 Vict. c. 57).

The following statement shows the total number of applications for Patents, Designs, and Trade Marks, and amount of gross receipts and expenditure in each year, from 1884 to 1893 inclusive:—

Years.	Patents.	Designs.		Trade Marks.	Gross Receipts.	Gross Expenditure.
		Single.	Sets.			
1884	No. 17,110	No. 19,545	No. 238	No. 7,104	£ 103,827	£ 64,123
1885	16,101	20,388	337	8,026	88,509	77,739
1886	17,176	23,717	324	10,677	106,754	*109,567
1887	18,051	25,751	309	12,586	124,279	81,577
1888	19,103	25,923	316	13,315	119,623	83,924
1889	21,009	24,370	335	11,316	172,820	79,886
1890	21,507	22,255	318	10,258	192,006	83,240
1891	22,258	21,673	277	10,787	203,520	*103,180
1892	24,169	19,269	258	9,101	199,859	*96,822
1893	25,120	19,174	306	8,675	174,878	*95,103

\* Including expenditure on account of new offices and buildings.  
—*Ibid.*

#### PHOSPHATE AND ASBESTOS MINING IN CANADA.

The French Consul in Quebec, in a recent report to his Government, says that for the last two years the most important of the mining products of the province—phosphates and asbestos—which used to be exported in vast quantities, have shown a considerable falling off in the quantity produced, so much so that production has almost completely ceased. It is expected that this check in the development of a very important industry will only be of a temporary nature, and that, fresh demands arising, the production will resume its upward course that until recently it has followed for so many years.

Phosphates are found in large quantities in the province of Quebec. Their first working only dates back to 20 years ago, and the production, which was about 4,000 tons in 1878, attained in recent years an average of 25,000 tons annually. The new phosphate beds discovered in Florida, the competition with which has brought about a considerable fall in prices, has rendered the working of Canadian phosphate comparatively unremunerative. The rates quoted on the English market, which were about 5*l.* per ton in 1889, fell to 3*l.*, and thus brought about a complete stoppage in the production in 1893. Canadian phosphates, which are remarkable for their richness, are found under the form of apatite in crystalline masses of a red or green colour. According to official statistical returns the quantity of phosphates exported from Quebec in 1880–81 amounted to 15,701 tons; in 1884–85, 18,984 tons; in 1890–91, to 25,257 tons; and in 1891–92, to 17,243 tons.

Equally with phosphates, asbestos, which has been worked in Quebec since 1878, has given rise to an industry which was rapidly developed to such a point that the production, at first only amounting to a few hundred tons, exceeded 8,000 tons in 1890 and 10,000 in 1891.

Asbestos is a product peculiar to the province of Quebec, which, with Italy, are the only places which supply this article to commerce. Numerous and fresh uses for this material having been discovered of recent years, a peculiar phenomenon was observable; this was that in spite of the considerable increase in the production the prices rose by about 80 dollars a ton, until they attained a rate of 250 dollars

in 1890. Since the latter date, however, prices have again fallen. Asbestos is principally used in various ways in connection with steam machinery and electric lighting. With it are made felts, tissues, packing paper, cements, bricks, &c. This product, which has received the name of chrysotile, a form or serpentine, is found in certain portions of the serpentine rocks of the Eastern Township of Quebec, as well as in some parts of Ottawa county, Quebec. The mining is practically confined to two sections, one at Thetford and the other at Black Lake, the two sections being about four miles apart. The mineral found in these districts is of the very highest quality, while elsewhere there are deposits of a lower grade, which, while not suitable for millboard and steam-packing, answer admirably for cements, paints, &c.—*Ibid.*

#### THE NORWEGIAN WOOD-PULP INDUSTRY.

According to the *Bulletin du Musée Commercial*, the year 1893 was a very favourable one in Norway for manufacturers of wood-pulp, the prices having on the average been four kroner (4*s.* 5½*d.*) higher for wet pulp, and eight kroner (8*s.* 10½*d.*) higher for dry than they were in the preceding year.

Purchasers hastened to place their orders, and at the commencement of the year the quantities sold amounted to 130,000 tons. The demand having again increased, the wood-pulp syndicate realised that the reduction of the production to the extent of 33½ per cent., which was primarily fixed upon, was too great, and at a meeting which was held in May the maximum reduction was fixed at 23½ per cent., but in spite of this prices continued to rise. Towards the end of the year small quantities which were available were sold at 55 kroner (3*l.* 1*s.* 1½*d.*) and 100 kroner (5*l.* 11*s.* 1½*d.*) per ton respectively for pulp wet and dry.

For the current year the greater part of the production is already sold at remunerative prices. The improvement which has manifested itself in the Norwegian wood-pulp industry has led to the very considerable extension of the factories already existing, and to the construction of new works in Sweden and Norway.

The home consumption has remained almost stationary; in England the consumption has increased, and also in France. As regards Germany, this country imported, in 1893, 10,000 tons of Norwegian wood-pulp more than in 1892, but this increase was in great measure due to the dryness of the season last year. At the present time there are 59 wood-pulp factories in Norway; of this number one is engaged in the manufacture of casks, three manufacture cardboard, and ten paper.

The quantity of wood-pulp exported from Norwegian ports, that is to say, the Norwegian product, and a certain proportion of Swedish pulp, amounted in 1893 to 230,000 tons, as compared with 215,000 tons in 1892 and 207,000 tons in 1890. The above observations and figures all relate to mechanical wood-pulp.

As regards chemical wood-pulp, the increase in the demand created in 1892, by reason of the restrictions effected in the rag trade continued in 1893, and the results of that year have been equally favourable to that industry. The prices in 1893 of dry chemical pulp quoted by the Norwegian manufacturers were as follows:—First-class dry sulphite, from 200 to 210 kroner (11*l.* 2*s.* 2½*d.* to 11*l.* 13*s.* 4*d.*); second-class from 185 to 195 kroner (10*l.* 5*s.* 6½*d.* to 10*l.* 16*s.* 8*d.*) per ton. First-class sulphate pulp was quoted at from 180 to 190 kroner (10*l.* to 10*l.* 11*s.* 1½*d.*) and second class from 170 to 180 kroner (9*l.* 8*s.* 10½*d.* to 10*l.*). It is stated that these prices are with difficulty obtained, and the numerous factories recently erected or projected in Norway, Sweden, and America, do not appear to have a very brilliant future before them. In all probability the prices of pulp will, it is said, fall, while those of the raw material will rise.—*Ibid.*

#### THE POSITION OF COLLODION COTTON.

The following letter has been issued from the Home Office:—

April 25, 1894.

Since the Explosives Act, 1875, came into force, the question has more than once been raised as to the position

of collodion cotton, or, as it is sometimes called, soluble gun-cotton; and, as you are probably aware, we have until recently held that such material need not be ranked as an explosive.

Further researches on the subject of the material have, however, disclosed the fact that its solubility may in some cases be associated with a certain explosibility, and under these circumstances it appears to us that soluble gun-cotton does, as a matter of fact, properly fall within the category of explosive as defined by the Explosives Act, 1875, and the Orders thereunder, except when its condition is such as to deprive it of its explosive properties, viz., when it is—

- (a) In solution in alcohol or ether, or
- (b) wet, or
- (c) saturated with methylated spirit and contained in air tight cases.

We have been in communication with the Customs on this subject, and we understand that they are issuing instructions to their officers to that effect. Of course this decision affects the manufacture, storage, and transport of soluble cotton, which does not fall within the exemptions specified above; of course, also, when soluble gun-cotton is intended for use, itself, as an explosive (not merely as an ingredient of an explosive), it necessarily falls within the definition of an explosive, in virtue of section 3. of the Act, irrespective of the condition in which it may exist.

#### SOUTH AFRICAN SUGAR INDUSTRY.

*Deutsches Handels. Archiv*, 1894, 105.

The sugar industry of Natal makes no attempt to compete with the world's market; its scope does not extend beyond the limit of the South African market. The sale of sugar within the colony itself is protected by a tax, which, for refined sugar, amounts to 1*d.* per lb., and for raw sugar to 3*s.* 6*d.* per centner; whilst its sale beyond the limits of the neighbouring States is subject to the taxes there existent. In the territories of the South African Zollverein (Cape Colony, Orange Free State, Basutoland, British Bechuanaland) and in the protectorate of Bechuanaland, these taxes amount to 6*s.* 3*d.* per 100 lb. for both refined and raw sugar. On raw sugar re-exported into those territories of South Africa not belonging to the Zollverein, a reimbursement of 3*s.* per 100 lb. is allowed. An abatement of 5 per cent. is allowed in Cape Colony on all imported sugar for the manufacture of preserved fruits and confectionery. The tax on sugar, whether refined or raw, in the South African Republic is 3*s.* 6*d.* per 100 lb. The effect of heavy taxes on Natal sugar sold in the neighbouring States is that all those interested in the Natal sugar industry are zealous supporters of the extension of the Zollverein.

The average annual production of sugar in Natal during the last 10 years is assessed at 12,000 tons. According to the "*Blaubuch*" it amounted to—

	Tons.
In the financial year 1890—91.....	11,640
" " 1891—92.....	26,319.25

the values of which for the two periods were 25*l.* 5*s.* and 23*l.* respectively per ton. Statistics regarding the amount of sugar exported are only at hand in the case of that exported by sea. The average amount during the last five years was:—Raw sugar, 5,234 tons; molasses, 222 tons. The Customs authorities in Natal divide everything into these two categories, whereas the Customs authorities in Cape Colony differentiate the imported sugar from Natal between raw sugar, to which the chief portion corresponds; also between refined sugar and "crystals." The distinction between raw and refined sugar has only, at present, a significance for the tax administrators of Cape Colony in so far as it touches the transport to territories without the Zollverein. Refined sugar includes only loaf- and cube-sugar. The raw sugar of Natal may be used for consumption, and, as a matter of fact, is exclusively used in Natal. The quantity of imported foreign sugar, as well as that transported into the interior, is very small in comparison

with the quantity of the home product. The average market price of sugar was—

	Per lb.
In the financial year 1890—91.....	3 <i>d.</i>
" " 1891—92.....	3 <i>d.</i>

The sugar landed in the ports of Cape Colony during the last five years was, besides a small quantity of molasses, as follows:—

	Tons.	£
Raw sugar.....	15,158	leaving a value of 226,000
Refined sugar and crystals.....	1,555	29,000

The quantity of imported sugar during late years is steady and has not inconsiderably increased. Besides the above mentioned quantities of sugar, however, there was landed at the ports of Cape Colony during the five years 1888—1892 an average annual quantity amounting to 3,120 tons for transportation to the territories beyond the Zollverein. The amount seems steadily increasing, and reached in the last year of the period named 15,710 tons.

In view of the steadily-increasing consumption of sugar in South Africa, a syndicate has been formed and a limited company floated with the object of constructing a beet-root sugar factory in the district of Swellendam, east of Cape Town. The prospectus was circulated in September 1893. At first, the whole of the required capital, amounting to 39,000*l.*, could not be got together, but the remaining portion was subsequently subscribed in London.

The contract for the furnishing of the factory and the accessory machinery has been given to a German firm. The value of this contract is said to be about 20,000*l.*

—A. R. L.

#### SCANDINAVIA AS A SOURCE OF IRON ORE SUPPLY.

*J. Head. Iron and Steel Institute, Spring Meeting, 1894.*

Although pigs and bars have long been known as Scandinavian products, iron ore has not been regarded as an important article of export until comparatively recent time. In 1870, 9,485 tons were exported from Sweden, whilst in 1887, 41,765 tons was the amount. On the latter quantity only 657 tons came to the United Kingdom. In the same year Norway supplied 2,485 tons. The total quantity of ore received in 1887 from Scandinavia by the British Iron Trade therefore was only about 3,000 tons. In 1888 the figure 657 rose to 62,672 tons, this increase being due to new railway and steamship companies, formed chiefly with English capital. Most of the ore smelted in England gave disappointing results, the pig-iron produced containing from 0.3 to 0.6 per cent. of phosphorus, which of course rendered it unfit for acid steel-making. As there was very little demand for such pig-iron the allied companies had to discontinue operations. As a result the imports of Swedish ore into the United Kingdom fell to 15,427 tons in 1889, and to 3,108 tons in 1891.

The English companies had made the mistake of mixing, as a rule, the products of the various quarries, thus producing an average quality which was neither pure enough for acid nor phosphoric enough for basic purposes. A Swedish company was formed which, profiting by the experience of their predecessors, took care to keep separate the products of the various workings, and to grade them according to the contained phosphorus.

They established four grades, A, B, C, and D, the first containing about 69 per cent. of iron and 0.01 per cent. of phosphorus intended for the acid process, and the last containing about 65 per cent. of iron and 1 to 2 per cent. of phosphorus intended for the basic process, with intermediate classifications for the other grades. The yield of the 131 quarries thus carefully assorted was found to be 20 per cent. of grades A and B, and 80 per cent. of the lower grades. The effect of these improved arrangements was soon apparent, and the figures rose from the above quantity in 1891, to 13,722 in 1892, and 35,901 tons in 1893. The material being almost entirely of the A quality. The Continent apparently has taken more advantage of this new source of iron ore supply, as the figures have risen from 117,530 tons in 1888 to 447,931 tons in 1893, this quantity

being received mostly by Germany and Austria. The basic process being largely employed on the Continent, the material sent there was mainly of the lower grades, which would hardly pay to send to England.

The author then enters into the question of the prices of transit, and says there is little prospect of Swedish ore of any kind being delivered in Middlesbrough, even through the contemplated new railway to Væsterås on the Norwegian coast, at less than 12s. per ton, a price which excludes the lower-grade ores. It is obvious that a reduction of this figure would be effected by the discovery of iron ore deposits nearer to this port, if such could be made.

It would appear that Titanic acid in variable proportions is a common constituent of Scandinavian ores. When used alone they have been found extremely difficult to smelt, owing to the Titanic acid.

If smelted, however, with poorer ores, especially such as contain alumina, the refractory nature seems to disappear. Titanic acid does not convey any impurity to the pig, but it seems to communicate to the iron unusual toughness, and causes sulphur and phosphorus to go into the slag, requiring also an increased coal consumption in the furnaces. The advisability of encouraging Scandinavia as another ore supply for Great Britain is advocated by the author.

—A. W.

#### THE WORLD'S PRODUCTION OF NOBLE METALS.

*Chem. Ind.* 16, 485.

The production of platinum is increasing in the United States. The nickel ore from Copper Cliff, near Sudbury, contains 0.20 per cent. of platinum, and similar ores have been observed at Denison (Ontario), containing from traces to 0.53 per cent. of platinum. A recent discovery of platinum ores on the river Tulamee, in British Columbia, appears to be of even greater importance. The production of nickel in America amounted in 1882 to 281,600 lb., which is about equal to the production of Sweden and Norway. Some of the occurrences of nickel ore in Arkansas, Colorado, Connecticut, Iowa, Massachusetts, Missouri, Nevada, North Carolina, and Oregon, are said to be very promising. The prospects of the nickel mines of New Caledonia have been considerably improved since the construction of roads to the mines. The exports of nickel ore, which in 1885 amounted to 19,700 tons only, subsequently rose to 35,000 tons in 1891. The Sudbury mines exported about 72,300 tons in 1892. The total production of the world amounts now to 10,307,000 pounds of nickel, the price of which has fallen as the production increased. Of late years nickel has attained considerable importance in the manufacture of very hard alloys of nickel and iron. This nickel steel is largely employed for the manufacture of armour plates and for ships' engines. An alloy of 20 parts of nickel with 80 parts of copper is used as a casing for the bullets of the new small calibre. The world's production of silver amounted to 2,592,000 kilos. in 1881, and remained stationary until about 1887. Since then the production of silver has increased at an enormous rate on account of the discovery of the rich deposits in the Sierra Mojada, Colorado, and Montana, and rose to 4,479,000 kilos. in 1891, and 5,935,000 kilos. in 1892. On the other hand, the production of gold, which in 1881 amounted to 158,000 kilos., remained stationary until 1888. Then it increased through the opening of the South African goldfields to 192,000 kilos. in 1892. Part of this increase is, however, due to the extraction of gold from American silver ores. The silver output of America alone amounted to 692,000 kilos. in 1872, 1,240,000 kilos. in 1885, and 2,018,000 kilos. in 1892. The relation between the price of gold and silver was as 1:15.56 in 1870, 1:18 in 1886, and 1:22 in 1890, and in spite of the Sherman bill came to 1:23.73 in 1893.—C. O. W.

#### ALCOHOL IN GERMANY.

Official figures relating to the spirit industry of the German Empire have just been published. They show that 60,025 alcohol-distilleries were at work in the Empire last year, against 59,789 in 1892. More than one-third (22,929) of these are found in the grape-growing provinces of Alsace-Lorraine; 19,577 in Baden; 6,508 in Württemberg; 4,667

in Bavaria; and only 6,108 in Prussia. The great majority of the distilleries (47,385) are small ones, using fruit, brewery-refuse, &c., as raw material. Only 27 works distil molasses—a branch of industry which appears to be dying out, as in 1891—92 there were only 39 molasses-using distilleries at work. Farinaceous substances (potatoes and grain) are used by 12,613 works. Prussia, although possessing the smallest number of works, has by far the largest, her share of the total alcohol-output of 3,028,920 hectolitres (of about 22 gallons) absolute alcohol being 2,449,626 litres. The largest Prussian factory produced over 10,000 hectolitres. The consumption of absolute alcohol in Germany for drinking purposes in 1892—93 was 2,254,919 hectolitres, or 4.5 litres per head of the population (= about 1 gallon). In 1891—92 it was 4.4 litres. The quantity used for industrial purposes was 60,670 hectolitres, against 551,300 the year before, of which 115,026 were used in vinegar-manufacture, and 315,254 were denaturalised. The total exports of German alcohol were only 111,447 hectolitres absolute. The gross revenue from the spirit tax was 111,435,216 m. in 1892—93 against 134,185,046 m. the year before. The following quantities of raw material were used for distilling:—Potatoes, 2,105,890 tons (1891—92, 1,334,602 tons); corn, 338,370 tons (1891—92, 490,420 tons); molasses, 37,453 tons (1891—92, 94,924 tons); stone-fruit, 252,900 hectolitres (1891—92, 306,031 hectolitres); wine-berries, 231,467 hectolitres (1891—92, 168,229 hectolitres); malt residue, 195,338 hectolitres (1891—92, 208,875 hectolitres); fruit (various), 64,006 hectolitres (1891—92, 89,434 hectolitres); liquid wine-berries, 30,252 hectolitres (1891—92, 18,211 hectolitres); pressing-residue from grapes, 29,517 hectolitres; (1891—92, 44,096 hectolitres); grape-wine, 15,743 hectolitres (1891—92, 11,301 hectolitres).—*Chemist and Druggist*.

#### BENGAL SALTPETRE.

In the Presidency of Bengal, saltpetre is manufactured in the Patna and Bhagalpore divisions and in the city of Calcutta and its suburbs. Crude saltpetre, however, is not manufactured in or near Calcutta, operations being confined there to the refining of the article imported from up-country. The control of the saltpetre refineries in Bengal is vested in the Commissioner of Northern India Salt Revenue and his subordinates. A large quantity of edible salt is obtained in the process of refining saltpetre, and under the existing rules the owners have the option either of destroying it or removing it from the refineries on payment of full duty. A market has, however, been created for this species of salt, and large quantities of it are now exported to Burdwan and other places. The fee for a licence for manufacturing and refining saltpetre, including the extraction of salt therefrom, is Rs. 50, and that for the manufacture of saltpetre, sulphate of soda (*khari*), either by solar heat in evaporating pans or by artificial heat, and of any other substance included under the term of "saltpetre" as defined in section 3 of the Salt Act of 1882, is 4 annas. Besides the saltpetre brought from the Patna and Bhagalpore divisions, a considerable quantity comes down from the North-West Provinces and the Punjab. The bulk of the saltpetre brought to Calcutta is exported to the United Kingdom, the United States of America, Mauritius, and China. The total exports in 1892—93 amounted to 438,408 cwt., valued at Rs. 43,36,588.—*Ibid*.

#### THE MANUFACTURE OF CITRIC AND TARTARIC ACIDS IN SICILY.

Our Sicilian correspondent writes:—"The Italian Government being desirous of encouraging the preparation in Sicily of citric and tartaric acids, especially the former, has issued a decree offering prizes to persons who shall open factories for the preparation of these acids in Sicily. The first prize is to be one of 500*l.*, the second of 350*l.*, and the third of 175*l.* sterling, in addition to which small bonuses will be given to the managers of works. To obtain the first prize both citric and tartaric acid must be made, the former in a quantity of at least  $\frac{1}{2}$  ton a day. Preference will be given in the competition to those firms making their own sulphuric acid, for the production of both citric and tartaric

acids. It is also laid down as a guide to the jury that they are to give the preference to those making the best use of subsidiary products, and those using vacuum apparatus, or apparatus made of enamelled iron or porcelain for evaporation. Applications for the prizes must be made on Italian stamped paper not later than December 31, 1893, and the works must be in operation not later than February, 1896, in which year the prizes will be distributed by the Minister of Agriculture. The competition appears to be open to foreigners. There are already two works in operation in the island.—*Ibid.*

#### THE ITALIAN TRADE IN TARTARIC MATERIALS.

Tartaric materials form one of the chief items of export from the southern provinces of Italy; the season commences in October, and official statistical reports for the year are generally given from that month to September of the following year. In 1891—92 the total export amounted to 9,814 tons, valued at 425,240*l.*; while in 1892—93, 9,881 tons, valued at 337,275*l.*, were shipped. The great difference

in value arises from the depreciation this article has undergone in consequence of the large stocks accumulated everywhere through the excellent grape harvests of the last two years. The tartaric materials exported during the last two years were composed as follows:—

		Half Refined Tartar.	Ammonium Argols.	Crude Argols.
1891—92.....	Tons	1,542	3,124	2,470
" .....	"	98,280	14,100	101,640
1892—93.....	Tons	1,317	2,959	2,801
" .....	"	72,435	133,155	98,025

More than one half of these products, especially "argols," is shipped to the United States; the remainder is taken by Great Britain, France, Germany, and Austria in about equal shares.—*Ibid.*

#### THE MINERAL PRODUCTION OF THE UNITED STATES, 1892 AND 1893.

No.	Product.	Customary Measures.	1892.		Value at Place of Production.	1893.		Value at Place of Production.
			Customary Measures.	Metric Tons.		Customary Measures.	Metric Tons.	
1	Asbestos.....	Short Tons	100	91	Dols. 5,000	120	109	Dols. 6,000
2	Antimony ore.....	"	850	771	51,000	850	771	11,000
3	Asphaltum and asphalt rock.....	"	47,040	42,675	254,016	34,944	31,701	174,720
4	Barytes (crude).....	"	28,476	25,833	142,380	26,632	24,161	133,160
5	Bauxite.....	"	9,800	8,891	49,600	11,044	10,106	55,205
6	Borax.....	Pounds	12,538,196	5,687	140,365	8,669,000	3,946	652,125
7	Bromine.....	"	379,180	172	64,512	348,339	158	87,100
8	Building stone.....	"	"	"	11,589,500	"	"	10,000,000
9	Cement, hydraulic.....	Barrels of 300— 400 lbs.	8,211,181	"	5,999,150	7,569,385	"	5,180,797
10	Cement, Portland.....	"	547,440	"	1,153,690	566,531	"	1,152,830
11	Coal, anthracite.....	Long Tons	46,850,405	47,352,636	89,727,982	48,041,834	48,818,376	103,661,670
12	Coal, bituminous.....	"	114,220,101	116,659,045	124,236,532	113,696,871	115,263,204	118,795,854
13	Coke.....	Short Tons	12,046,820	12,204,203	23,421,117	9,792,336	9,949,986	14,698,495
14	Cobalt oxide.....	Pounds	8,600	73,900	6,150	3,893	4,766	4,700
15	Copperas.....	Short Tons	13,250	12,021	110,272	16,000	14,515	95,440
16	Copper sulphate.....	Pounds	"	"	"	54,000,000	24,482	1,222,500
17	Corundum.....	Short Tons	1,504	1,364	132,094	1,747	1,585	140,580
18	Chrome ore.....	Long Tons	1,650	1,677	16,500	1,920	1,646	16,000
19	Feldspar.....	"	16,000	16,258	80,000	17,000	17,274	85,000
20	Flint.....	"	37,000	37,596	185,000	38,000	38,612	190,000
21	Fluorspar.....	Short Tons	9,000	8,165	54,000	9,700	8,800	65,070
22	Grindstones.....	"	"	"	304,800	45,780	11,370	345,320
23	Gypsum.....	"	256,250	232,158	695,492	250,000	226,739	562,500
24	Infusorial earth and tripoli.....	"	1,323	1,200	41,950	1,760	1,570	47,800
25	Lime.....	Barrels, 200 lbs.	70,000,000	6,350,200	38,500,000	70,000,000	5,143,164	30,000,000
26	Limestone for iron flux.....	Long Tons	4,500,000	4,633,116	2,027,500	3,750,000	3,810,375	2,250,000
27	Magnesite.....	Short Tons	1,462	1,272	9,814	1,143	1,037	8,000
28	Manganese ore.....	Long Tons	19,117	19,425	129,586	9,150	9,237	69,000
29	Marls.....	Short Tons	125,000	113,400	65,000	110,000	99,792	55,000
30	Mica.....	Pounds	75,000	34	10,000	75,000	34	10,000

(f) Estimated.

• Kilograms.

THE MINERAL PRODUCTION OF THE UNITED STATES, 1892 AND 1893—*continued*.

		1892.			1893.			
No.	Product.	Customary Measures.	Quantity.		Value at Place of Production.	Quantity.		Value at Place of Production.
			Customary Measures.	Metric Tons.		Customary Measures.	Metric Tons.	
					Dols.			Dols.
31	Millstones .....	..	..	..	20,000	..	..	18,000
32	Mineral paints .....	Long Tons	50,000	50,895	650,000	..	..	546,000
33	Natural gas .....	..	..	..	14,800,000	..	..	14,000,000
34	Onyx .....	Cubic Feet	3,500	..	40,000	2,175	..	28,750
35	Ozokerite (refined) .....	Pounds	130,000	59	7,500	None.	..	..
36	Petroleum .....	Bbls., 42 galls.	50,512,156	7,000,982	30,229,128	50,349,228	6,978,493	30,223,505
37	Phosphate rock .....	Long Tons	902,723	917,257	3,322,021	981,340	997,140	3,434,630
38	Plumbago (crude) .....	Short Tons	900	816	3,500	1,590	1,365	7,500
39	Plumbago (refined) .....	Pounds	1,398,363	634	87,992	596,603	406	39,503
40	Potters' clay .....	Long Tons	150,000	157,340	1,000,000	303,000	393,327	830,000
41	Precious stones .....	..	..	..	188,000	..	..	200,000
42	Pyrites .....	Long Tons	104,250	10,9957	357,000	95,000	96,526	285,000
43	Salt .....	Barrels, 280 lbs.	11,784,954	1,542,133	5,900,900	11,435,487	1,452,388	5,717,743
44	Slate (for pigment) .....	Short Tons	3,400	3,085	21,000	3,000	2,721	18,000
45	Slate (for roofing) .....	In squares	953,000	..	3,306,625	871,500	..	2,780,000
46	Slate (other kinds) .....	..	..	..	750,500	..	..	737,400
47	Soapstone .....	Short Tons	23,208	21,054	423,449	20,100	18,235	366,825
48	Soda, natural .....	..	3,300	2,904	16,500	2,500	2,268	12,500
49	Soda, natural sulphate .....	..	1,689	1,521	8,400	90	82	450
50	Sulphur .....	..	1,825	1,656	51,750	1,314	1,219	26,880
51	Talc (fibrous) .....	..	41,925	38,034	472,485	56,500	33,113	337,625
52	Venetian red .....	..	4,205	3,815	89,335	3,830	3,475	81,475
53	Whetstones ( <i>gr.</i> ) .....	Gross Pounds	1,009,000	..	107,580	900,000	..	105,925
54	Zinc, white .....	Short Tons	27,500	24,943	2,200,000	25,000	22,678	1,875,000
	Total non-metallic .....	..	..	..	396,610,582	..	..	371,376,365
METALLIC.								
55	Aluminium, value at New York .....	Pounds	205,000	134	191,750	312,000	142	202,800
56	Antimony, value at San Francisco .....	Short Tons	200	181	56,900	250	318	63,000
57	Copper, value at New York .....	Pounds	325,500,000	147,647	58,716,400	322,585,500	146,324	31,677,940
58	Gold, coinage value .....	Troy Ozs.	1,590,365	* 90,652	32,097,951	1,739,081	* 54,091	35,950,000
59	Piez iron, value at New York .....	Long Tons	8,957,869	9,122,413	131,668,935	7,043,384	7,156,782	93,888,369
60	Lead, value at New York .....	Short Tons	205,630	186,518	16,450,400	193,328	175,931	14,467,029
61	Nickel (metal) .....	Pounds	36,152	* 13,614	57,391	25,803	* 11,745	12,429
62	Platinum (crude) .....	Troy Ozs.	350	* 11	1,750	300	* 9.3	9,500
63	Quicksilver, value at San Francisco .....	Flasks, 76½ lbs.	27,293	971	1,119,720	39,164	1,046	1,108,527
64	Silver, coinage value .....	Troy Ozs.	65,000,000	* 2,622,195	84,038,500	60,500,000	* 1,881,732	78,220,450
65	Spiegeleisen and ferromanganese .....	Long Tons	179,161	182,015	46,947,200	81,118	82,424	2,803,229
66	Tin .....	Pounds	143,400	65	29,827	None.	..	..
67	Zinc, value at New York .....	Short Tons	84,082	76,279	7,785,903	76,255	69,178	6,214,782
	Total metallic .....	..	..	..	320,740,427	..	..	267,707,795
	Estimated products, unspecified .....	..	..	..	77,500,000	..	..	76,000,000
	Grand total .....	..	..	..	724,821,009	..	..	645,084,750

(*f*) Estimated.(*g*) Includes scythestones and novaculite.

\* Kilograms.

(*h*) Value taken as average of spiegeleisen and ferromanganese, assuming production to have been one-third ferromanganese.—*Engineering and Mining Journal.*

## BOARD OF TRADE RETURNS.

## SUMMARY OF IMPORTS.

Articles.	Month ending 31st May.	
	1893.	1894.
	£	£
Metals.....	1,903,602	1,503,982
Chemicals and dyestuffs.....	497,259	499,394
Oils.....	577,916	544,970
Raw materials for non-textile industries.	3,825,123	3,642,386
Total value of all imports ....	36,838,213	31,134,069

## SUMMARY OF EXPORTS.

Articles.	Month ending 31st May.	
	1893.	1894.
	£	£
Metals (other than machinery) ....	2,846,097	2,417,432
Chemicals and medicines .....	773,125	711,093
Miscellaneous articles.....	2,719,582	2,567,465
Total value of all exports.....	17,822,490	17,484,212

## IMPORTS OF METALS FOR MONTH ENDING 31ST MAY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Copper:—			£	£
Ore..... Tons	4,129	3,815	25,073	31,174
Regulus .....	10,128	6,939	249,266	187,943
Unwrought ....	3,007	4,648	139,916	191,897
Iron:—				
Ore.....	348,564	351,285	232,105	239,134
Bolt, bar, &c. ....	5,594	6,957	44,594	69,292
Steel, unwrought..	852	457	8,396	4,414
Lead, pig and sheet ..	17,948	14,227	175,863	102,745
Pyrites .....	52,022	49,575	92,475	87,328
Quicksilver..... Lb.	89,920	303,206	75,543	212,38
Silver ore..... Value £	..	..	36,977	160,656
Tin..... Cwt.	67,210	64,975	398,879	224,113
Zinc..... Tons	5,510	3,766	100,162	59,890
Other articles ... Value £	..	..	144,382	123,218
Total value of metals	..	..	1,943,602	1,503,982

## IMPORTS OF OILS FOR MONTH ENDING 31ST MAY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Cocoa-nut..... Cwt.	12,948	19,453	18,461	22,644
Olive .....	2,439	1,805	84,305	65,922
Palm .....	89,411	114,093	199,238	146,917
Petroleum .....	12,037,764	12,036,671	190,253	157,099
Seed .....	2,127	2,400	55,728	55,282
Train, &c..... Tons	1,119	1,927	20,892	39,549
Turpentine .....	4,045	791	5,157	799
Other articles .. Value £	..	..	92,866	95,896
Total value of oils...	..	..	574,910	544,970

## IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDING 31ST MAY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Bark, Peruvian .. Cwt.	7,592	7,896	15,931	14,949
Bristles..... Lb.	351,522	250,986	57,345	34,823
Caoutchouc..... Cwt.	39,814	32,335	355,949	353,909
Gum:—				
Arabic.....	8,023	3,644	17,095	12,792
Lac, &c.....	14,944	14,778	73,798	89,842
Gutta-percha ....	4,929	4,185	32,399	43,376
Hides, raw:—				
Dry.....	44,485	36,619	114,493	85,617
Wet.....	41,967	44,138	99,985	94,741
Ivory.....	624	496	36,356	21,884
Manure:—				
Guano..... Tons	2,343	1,343	12,925	8,782
Bones.....	5,969	7,495	25,494	35,383
Nitrate of soda... ..	22,253	14,264	294,842	137,397
Phosphate of lime ..	27,976	38,905	48,526	71,864
Paraffin..... Cwt.	89,833	59,769	92,069	53,440
Linen rags..... Tons	1,123	1,677	9,897	13,830
Esparto.....	17,166	15,256	88,188	96,590
Pulp of wood ....	21,973	22,615	115,582	112,881
Rosin..... Cwt.	156,554	151,556	58,826	34,495
Tallow and stearin ..	169,908	117,912	228,952	152,534
Tar..... Barrels	5,810	6,599	3,124	3,697
Wood:—				
Hewn .....	225,687	207,591	452,582	355,645
Sawn .....	399,939	395,022	694,401	889,058
Staves .....	8,827	9,855	52,829	45,472
Mahogany .....	4,104	3,901	55,796	32,573
Other articles.... Value £	..	..	97,999	895,008
Total value .....	..	..	3,825,123	3,642,386

Besides the above, drugs to the value of 72,200<sup>l</sup>. were imported, as against 72,726<sup>l</sup>. in May 1893.



IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH  
ENDING 31ST MAY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Alkali..... Cwt.	8,358	8,558	£ 7,987	£ 8,185
Bark (tanners', &c.) ..	34,300	26,917	13,111	9,449
Brimstone..... "	33,591	49,052	8,549	11,728
Chemicals..... Value £	..	..	116,292	132,673
Cochineal ..... Cwt.	77	197	723	1,918
Cutch and gambier Tons	2,441	2,517	52,274	61,656
Dyes:—				
Aniline..... Value £	..	..	13,496	17,354
Alizarin ..... "	..	..	25,300	25,838
Other ..... "	..	..	2,073	2,909
Indigo ..... Cwt.	2,042	1,914	34,743	33,954
Nitrate of potash . "	26,589	15,882	22,318	14,785
Valonia ..... Tons	1,668	1,311	22,131	16,717
Other articles... Value £	..	..	177,983	155,205
Total value of chemicals	..	..	497,239	490,991

EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH  
ENDING 31ST MAY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Gunpowder..... Lb.	727,800	721,100	£ 18,066	£ 18,864
Military stores.. Value £	..	..	61,082	129,874
Candles..... Lb.	1,599,300	1,395,500	30,098	24,237
Caoutchouc..... Value £	..	..	96,717	88,883
Cement..... Tons	43,988	48,120	74,481	81,256
Products of coal Value £	..	..	125,304	112,866
Earthenware ... "	..	..	186,563	132,987
Stoneware..... "	..	..	16,029	14,066
Glass:—				
Plate..... Sq. Ft.	181,700	149,954	9,609	7,030
Flint..... Cwt.	5,747	7,415	15,857	17,369
Bottles..... "	54,672	60,041	25,029	28,473
Other kinds.... "	17,777	14,187	15,280	11,063
Leather:—				
Unwrought .... "	13,611	12,255	124,800	106,972
Wrought ..... Value £	..	..	24,087	18,737
Seed oil..... Tons	7,147	5,645	154,917	119,139
Floorcloth ..... Sq. Yds.	1,563,500	1,609,400	63,133	58,085
Painters' materials Val. £	..	..	135,134	137,658
Paper ..... Cwt.	88,535	83,647	137,379	128,924
Bags..... Tons	4,620	5,157	35,000	28,402
Soap..... Cwt.	54,165	51,913	77,659	57,115
Total value .....	..	..	2,719,582	2,567,465

EXPORTS OF METALS (OTHER THAN MACHINERY) FOR  
MONTH ENDING 31ST MAY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Brass..... Cwt.	10,143	11,732	£ 40,063	£ 41,136
Copper:—				
Unwrought ..... "	68,166	37,189	165,350	82,672
Wrought..... "	26,746	23,082	77,370	64,803
Mixed metal .... "	28,684	17,209	67,231	37,996
Hardware..... Value £	..	..	186,541	155,157
Implements..... "	..	..	195,785	103,718
Iron and steel..... Tons	285,429	238,914	1,927,819	1,646,516
Lead ..... "	8,909	6,265	94,139	63,571
Plated wares... Value £	..	..	24,886	22,088
Telegraph wires .. "	..	..	15,487	90,077
Tin ..... Cwt.	12,499	7,760	54,740	29,504
Zinc ..... "	15,111	12,810	12,215	8,819
Other articles .. Value £	..	..	69,990	71,275
Total value .....	..	..	2,846,607	2,417,432

EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING  
31ST MAY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Alkali..... Cwt.	551,453	491,062	£ 169,425	£ 129,657
Bleaching materials ..	145,889	119,662	60,390	47,366
Chemical manures. Tons	21,754	29,137	155,445	156,767
Medicines..... Value £	..	..	75,272	85,555
Other articles ... "	..	..	312,593	291,808
Total value .....	..	..	773,125	711,093

## Monthly Patent List.

\*The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### APPLICATIONS.

9815. P. M. F. Laurent. Improved apparatus for sterilizing liquids, applicable also for heating, cooling, condensing, distilling, and other similar purposes. Filed May 19. Date applied for November 20, 1893, being date of application in France.

9958. T. Craney. Improvements in evaporating apparatus. May 22.

9959. T. Craney. Improvements in evaporating apparatus. May 22.

10,523. J. F. Beins. An improved method of and apparatus for saturating liquids with gases. May 30.

10,906. A. Chapman. Improvements in apparatus for evaporating brine or other liquors or solutions, and extracting the salts or other solid matters therefrom. June 5.

11,226. D. B. Morison. Improvements in apparatus for heating, evaporating, or condensing. June 9.

11,354. J. Wilkinson. Improvements in furnaces applicable to metallurgical, chemical, and other purposes. June 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.\*

1893.

1017. G. Pettigrew. Improved still and apparatus for the distillation of ammoniacal liquor and other fluids, and the manufacture of sulphate of ammonia. May 30.

10,427. T. E. Mitton. An improvement in pressure-gauges and vacuum-gauges. May 30.

12,528. A. Lavy. An improved distilling apparatus. May 23.

13,567. V. Edwards. Copper distillation flask. May 30.

13,784. J. Horne. Improvements in means for mixing or blending liquids. May 23.

15,622. A. E. H. Field. An improvement in compound asbestos and other fibrous packings. June 20.

16,198. W. C. Currie and W. E. Rowlands. An improvement in apparatus for drying animal and other matters. June 6.

1894.

7027. L. Wagner and J. Marr. New or improved process and apparatus for purifying liquids. May 30.

### II.—FUEL, GAS, AND LIGHT.

#### APPLICATIONS.

9709. E. Mason. Fuel economiser. May 18.

9913. W. P. Thompson. Improvements in separating gases and in apparatus therefor. May 22.

10,029. R. Fegan. Improvements in compressed fuel. May 23.

10,438. G. S. Cory and C. Cory. Improvements in the manufacture of artificial fuel. May 29.

10,184. T. A. Garnett, G. Forster, D. Lister, H. Taylor, and F. Birchall. Improvements in carbonising coal or other gas. May 30.

10,772. A. J. Tempire. Process of deodorizing petroleum and other hydrocarbons. Filed June 2. Date applied for December 4, 1893, being date of application in France.

11,256. J. Ormerod. Improvements in and connected with gas carbonising apparatus. June 11.

11,432. G. N. Stockman. Cylinder to increase the illuminating power of gas in lighting railway carriages. June 12.

11,522. N. K. H. Ekeland. Apparatus for the manufacture of coal powder of peat, saw-dust, and such like. Complete Specification. Filed June 13. Date applied for November 29, 1893, being date of application in Sweden.

11,536. J. T. Kay, J. W. C. Holmes, and P. F. Holmes. Double or single incline retort for the destructive distillation of coal or other material, for the production of gas for illumination and other purposes. June 14.

11,645. A. Mortimer. An improved fire-lighter. Complete Specification filed June 15. Post-dated April 20, 1894, and re-numbered 11,645.

11,657. J. T. Westcott. Improvements in apparatus for the manufacture of water-gas. June 15.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

5647. J. H. R. Dinmore. Improvements in or connected with the manufacture of gas. June 20.

12,440. L. Mond. Improvements in obtaining combustible gases, ammonia, and tar from fuel, and in apparatus therefor. June 13.

12,903. J. Bowing. See Class III.

14,918. Brin's Oxygen Co. Lim., and K. S. Murray. Improvements in or connected with the production of oxygen from atmospheric air, and apparatus to be employed therein. June 13.

21,089. C. D. Abel. — From the Gas Motoren Fabrik Deutz. Improvements in gas producers. June 13.

1894.

7372. M. Bernstein and A. Silbermann. Improvements in and relating to incandescent gas lighting. June 13.

7527. C. Barker. An improved fire-lighter for lighting coal or coke fires. June 6.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

4097. J. A. Wanklyn and W. J. Cooper. Improvements in the distillation of petroleum. May 30.

12,440. L. Mond. See Class II.

12,903. J. Bowing. Improvements in coking processes and apparatus, and in the recovery of the products of distillation. June 13.

## IV.—COLOURING MATTERS AND DYES.

## APPLICATIONS

9529. S. Pitt.—From L. Cassella and Co., Germany. The production of polyazo-dye-stuffs from gamma-amidonaphtholsulpho acid. May 15.

9610. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of colouring matters and materials therefor. May 16.

9611. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of colouring-matters. May 16.

9681. H. E. Newton. From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of chemical compounds derived from amidoazobenzene or analogues thereof and suitable for use in dyeing and printing. May 17.

9806. J. C. L. Durand, D. E. Huguenin, and A. J. J. d'Andiran. Manufacture of new violet, blue, grey, and black substantive colouring-matters for dyeing cotton. Complete Specification. May 19.

9856. C. D. Abel.—From The actien Gesellschaft für Anilin Fabrikation, Germany. Improvements in the manufacture of colouring-matters dyeing blue with mordants. May 21.

10,194. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. The manufacture and production of new dyes of the rhodamine series. May 25.

10,218. A. Zimmermann.—From The Chemische Fabrik auf Actien vormals E. Schering, Germany. The treatment of phenols and phenol derivatives and the production therefrom of hydroquinone, hydroxylated phenol derivatives and allied bodies. May 28.

10,333. J. C. L. Durand, D. E. Huguenin, and A. J. J. d'Andiran. Manufacture of new colouring matters varying from blue-violet to blue-green. Filed May 28. Date applied for, October 31, 1893, being date of application in France.

10,854. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in the manufacture of colouring-matters related to the indulines and eurhodines June 4.

11,157. S. Pitt.—From L. Cassella and Co., Germany. The production of phenyl-amido-oxynaphthaline sulpho acid, and of colouring-matters deriving therefrom. June 8.

11,216. A. Wohl. Manufacture of hydroxylamine, hydrazine, and derivatives thereof. June 9.

11,569. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Production of red dye from paranitro-aniline. June 11.

11,641. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Process for production of black azo dyes on the fibre. June 15.

11,708. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Improved manufacture of paramethoxy and para-ethoxyphenyl-urea. June 16.

11,717. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of colouring matters. June 16.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

12,323. H. H. Lake.—From Wirth and Co., agents for A. Leonhardt and Co. Improvements in the manufacture of colouring matters. May 30.

13,833. C. D. Abel.—From The Actien Gessellschaft für Anilin Fabrikation. Manufacture of mordant-dyeing colouring matters. May 23.

14,014. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik. The manufacture and production of new basic colouring matters. May 23.

14,345. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. Improvements in the manufacture or production of colouring matters derived from anthraquinone. May 30.

14,671. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Improvements in the production of green and bluish-green colouring matters. June 13.

14,678. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik. The manufacture and production of new amido-sulpho acid, and of new azo dyes therefrom. May 23.

14,895. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation. Improvements in the manufacture of colouring matters. June 6.

15,045. H. E. Newton. From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of dye-stuffs. June 13.

15,080. H. H. Lake.—From Messrs. Wirth and Co., agents for A. Leonhardt and Co. Improvements in the manufacture of colouring matters. June 6.

15,223. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. Improvements in the manufacture or production of naphthylamine sulpho acids. June 13.

15,327. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Production of para-ethoxy and of para-methoxy-phenyl-succinimides. June 20.

15,328. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Improvements in the production of iodo-derivatives of the succinimide and of substituted succinimides. June 20.

15,444. S. Pitt.—From L. Cassella and Co. The production of new acetonaphthylenediamine sulpho acids and of colouring matters derived therefrom. June 20.

15,509. R. G. Williams. Improvements in colouring matters. May 30.

16,370. R. Willcox.—From The Badische Anilin und Soda Fabrik. The manufacture and production of new acid colouring matters. June 6.

23,578. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis. Manufacture of colouring matters applicable to unmordanted fibres or fabrics. June 6.

1894.

8536. F. Petersen-Møller. Manufacture of colouring matters, dyeing unmordanted cotton, and their salts and sulpho-derivatives. June 6.

## V.—TEXTILES, COTTON, WOOL, SILK, Etc.

## APPLICATIONS.

10,337. H. Cohrs and J. Oesterreich. Process and composition for waterproofing textile materials or fabrics and finished garments. May 28.

10,450. H. F. Moreau and L. Canivet. The manufacture and production of a new kind of lace. May 29.

10,542. J. Campbell. Improvements in the method or process of washing "waste" silk. May 31.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

11,110. T. Birnbaum. Improvements in the manufacture of coloured waterproof fabrics. May 23.

13,921. F. Walton. Improvements in apparatus for the manufacture of mosaic floorcloth. May 23.

14,551. G. MacFaur. Improvements in the manufacture of flax or vegetable fibre roofing. May 30.

15,357. J. F. Coates. Improvements connected with the printing of fabrics such as oil cloths, linoleum, and other like flexible substances. June 20.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

### APPLICATIONS.

9524. L. Schaefer. Improved method of and apparatus for marking patterns on fabrics. May 15.

10,391. F. A. Blair. Improvements in apparatus for dyeing or otherwise treating with liquids textile materials in a more or less manufactured condition. May 29.

10,392. J. Pritchard. Improvements in apparatus for dyeing yarns and slubbings or rovings or otherwise treating the same with liquids. May 29.

10,466. E. B. Manby. An improved medium or vehicle for thickening and fixing calico printer's pigments, colours, and mordants, and for use as an improved albumen substitute in calico printing and dyeing. May 30.

10,564. J. Tait. Improvements in bleaching jute. May 31.

11,077. E. Hoffmann. Improvements relating to dyeing wool with chromium lakes. Complete Specification. June 7.

11,641. O. Imray.—From The Farbwerke vormals, Meister, Lucius, and Brüning, Germany. See Class IV.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

14,154. J. O. Obermaier. Improvements in or relating to dyeing apparatus. May 23.

14,656. W. Hadfield, J. J. Sumner, and H. Hadfield. Improvements in and apparatus for printing fabrics. June 6.

15,028. J. Clapham, J. Picard, C. Villedieu, and W. W. L. Lishman. Improvements in the means and method of preparing vegetable and animal fibres, fabrics, or piece goods for the reception of aniline black. June 6.

15,326. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Improvements in the process for producing fast colours from direct dyes on cotton. June 20.

## VII.—ACIDS, ALKALIS, AND SALTS.

### APPLICATIONS.

9782. A. Campbell and A. Walker. Improvements in utilising acid sulphates and acids, and in obtaining nitric acid and hydrochloric acid. May 19.

9979. T. Craney. Improvements relating to the manufacture of sodium bicarbonate and to apparatus therefor. May 22.

10,127. T. Hawkins, S. H. Hawkins, and G. Newnham. A dichromate oxyhydrogen compound. May 25.

10,555. A. McDougall. Improvements in the manufacture of salts of ammonia. May 31.

10,589. O. Imray.—From P. Gredt, Belgium. Process for treating blast-furnace gases for the extraction of alkaline and ammonium compounds, such as iodides and iodine, therefrom. Complete Specification. May 31.

10,604. J. Y. Johnson.—From The Verein Chemischer Fabriken, Germany. A process of manufacture of chlorine from hydrochloric acid gas and nitric acid by the employment of sulphuric acid. May 31.

10,781. A. R. Davis. Improved apparatus for the manufacture of compounds of ammonia and carbonic acid. June 2.

10,782. A. R. Davis. Improvements in the manufacture of compounds of ammonia and carbonic acid. June 2.

10,983. E. Pew and W. Blenheim. The production of vinegar or acetic acid by means of ozone. June 6.

11,233. A. Crossley. An improved process and apparatus for the manufacture of zinc oxides, sulphates and sulphides, from zinc ashes, zinc blende, carbonate of zinc, and other ores of a complex nature, such as ores containing lead, zinc, silver, copper, manganese, iron, or other metals in combination. June 9.

11,655. J. Greenwood. Improvements in and connected with the manufacture or production of ammoniacal. June 15.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

10,171. G. Pettigrew. See Class I.

12,995. H. Lane and J. Pullman. An improved method of and apparatus for producing carbonic acid gas. June 13.

13,654. G. Lunge and F. Maxwell Lyte. Improvements in the production of nitric acid, caustic alkali, and ferric oxide. June 6.

13,708. Sir Lowthian Bell, Bart. Improvements in the treatment of bicarbonate of soda. May 23.

13,709. Sir Lowthian Bell, Bart. Improvements in evaporating saline solutions. May 23.

14,407. A. K. Huntington. An improved manufacture of compounds of iron with fatty acids. May 30.

19,539. R. E. Chatfield. A process for utilising acid sulphates of soda. May 23.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

### APPLICATIONS.

9546. H. J. Hadden.—From the Columbian Pottery and Brick Kiln Co., United States. Improvements in kilns. Complete Specification. May 15.

9554. W. W. Pilkington. Improvements in apparatus for the manufacture of sheet and plate glass with embedded metallic wire. May 16.

9860. F. W. Golby.—From M. Hoffmann, Germany. A process for producing pressed glass plates for lining walls, ceilings, and for similar purposes. May 21.

10,085. A. Cay. A new or improved manufacture of ornamental sheet glass. May 24.

10,117. E. M. C. Gondouin. Improvements in ovens or kilns for use in burning or baking articles of porcelain, earthenware, and the like, and in apparatus for use in connection therewith. May 24.

10,734. C. J. Hall. Improvements in and in the application of a certain composition or material specially suited for use in the plastic arts, such as the production of artistic objects in bas-relief, relief, or in the round. June 2.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

10,503. A. G. Brookes. From A. Kattenloft. Improvements in the manufacture of lamp glasses for safety-lamps and such like glasses for similar uses. May 30.

1894.

7412. W. Garraway. Improvements in the manufacture of caustic soda and potash, and of nitric and sulphuric acids. May 23.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### APPLICATIONS.

9511. H. C. Stephens. A process and apparatus for treating timber so as to modify its condition and preserve it from decay. May 15.

9545. A. Baubère. Improvements in continuous or running kilns for burning lime, cement, and other similar materials. May 15.

9559. F. W. Golby.—From C. Bayer, Austria. Improvements relating to the manufacture of inscriptions from or in artificial stones. May 21.

9948. H. Maurer. An improved process of manufacturing porous bricks. Complete Specification. May 22.

10,243. R. D. Bailey. Improvements in and appertaining to street pavements and house flooring. May 28.

10,416. J. Briggs. Improvements in kilns for burning limestone, cement, and other similar materials. May 29.

10,585. C. Geige. A new or improved method or process for the manufacture of artificial wood. May 31.

10,607. J. P. Brasseur and N. Lambert. A process for the production of cement from furnace slag. May 31.

11,704. L. Jones. Improvements in the manufacture of fireclay. June 16.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

13,386. H. Salmon and J. E. Whiting. New or improved substitute for pumice stone, and process of manufacturing the same. June 2.

15,496. The Alamant Company, Limited, and J. Wilkinson. A new or improved fire-proof flooring. June 6.

22,978. T. Potter. Improvements in the construction of fire-resisting floors. June 20.

1894.

8540. R. Stone. Improvements in the manufacture and treatment of fire-proof plastic material for cement. June 20.

## X.—METALLURGY, MINING, ETC.

### APPLICATIONS.

9536. A. G. Fell. Improvements in methods of treating lead ores. Complete Specification. May 15.

9578. C. T. J. Vautin. An improved process and means used therein for the production of an alloy of tin with the alkaline metals or metals of the alkaline earths. May 21.

10,197. C. T. J. Vautin. An improved process and means used therein for the production of metallic sodium and potassium from salts of the same. May 25.

10,249. G. Thomson. Improvements in obtaining gold and silver from ores, mattes, or other compounds. May 28.

10,322. J. P. van der Ploeg. Improvements in or relating to the extraction of gold and other precious metals from their ores, or other compounds, and the recovery of the solvents used in the process. May 28.

10,344. C. M. Pielsticker. Improvements in the extraction of gold and silver from ores. May 28.

10,439. A. H. Moore and G. Whitlock. Improvements in and relating to the manufacture of combined iron and steel ingots. Complete Specification. May 29.

10,478. T. W. Walker. An improved method of preparing iron or steel blooms, or billets to be used in guide and other small mills. May 30.

10,820. E. Böcking. An improved method or process of preparing moist purple ore (fine iron ore) for smelting. June 4.

10,827. M. Boly. An improved process for the chemical disaggregation of pyrites of complex composition with a view to rapid extraction of the metals contained therein, such as gold, silver, arsenic, antimony, and tellurium. June 4.

10,923. W. P. Thompson.—From E. Bertrand and O. Thiel, Austria. Improvements in the manufacture of steel. Complete Specification. June 5.

11,138. F. Siemens. A method of protecting iron, steel, or other metal from oxidation during heating. June 8.

11,148. W. P. Thompson.—From O. Nicolai, Germany. An improved flux for use in soldering aluminium and other metals. Complete Specification. June 8.

11,168. H. H. Lake.—From The Metallgesellschaft, Germany. Improvements in and relating to apparatus for use in the manufacture of sodium. June 8.

11,274. J. D. Wilson. Improvements in tuyères for blast furnaces and cupolas. June 11.

11,307. C. Hoepfner. An improved method or process for the treatment of the ores of nickel and cobalt. June 11.

11,447. J. J. W. Carr. Improvements in furnaces for melting, heating, or annealing metals, metallic alloys, and the like. June 13.

11,570. S. Hufty and J. K. Caldwell. Improvements in methods of partially decarbonising and converting iron and increasing carbon in wrought iron and steel. June 14.

11,574. H. R. Müller and F. O. Müller. A means or apparatus for producing pure castings. Complete Specification. June 14.

11,724. C. Hoepfner. Improvements in the treatment of calamine and similar minerals for the extraction of zinc. June 16.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

8964. J. A. Mays. Separating and purifying metals. June 13.

12,647. W. Mills. Improvements in metallurgy. May 13.

12,979. J. W. Hall. An improved process for treating complex and refractory ores and materials, and obtaining the metals and pigments therefrom. June 6.

13,817. R. A. Hadfield. Improvements in the manufacture of what is known as manganese steel. May 30.

15,117. R. Howarth. Improvements in the manufacture of metallic alloys or compounds. June 20.

15,949. O. Imray.—From B. T. Laey and J. Storer. Improvements in dissolving, leaching, and filtering ores or minerals, and apparatus for that purpose. May 30.

1894.

8085. P. C. Choate. Improvements in the art of producing metallic zinc. June 6.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### APPLICATIONS.

9761. T. Craney. Improvements in electrolytic apparatus. Complete Specification. May 18.

9862. W. Morison. Improvements in batteries. Complete Specification. May 21.

9949. T. Craney. Improvements in electrolytic apparatus. Complete Specification. May 22.

9975. W. P. Thompson.—From C. L. Collin, United States. Improvements in the method of, and apparatus for heating, welding, or working metals by electricity. Complete Specification. May 22.

10,032. E. Gautier. Improvements in, and connected with electrolysis, and in apparatus connected thereto. May 23.

10,169. G. R. Blot. Improvements in plates for secondary voltaic batteries. Complete Specification. May 25.

10,463. J. M. Moffat. Improvements in cells or boxes of electric batteries. May 30.

10,823. A. F. B. Gomes. A new or improved process for the extraction of aluminium from certain clays by electrolysis. June 4.

10,853. J. Y. Johnson. From La Société L'Accumulateur Fulmen, France. Improvements in electrical accumulators or storage batteries. Complete Specification. June 1.

10,952. C. Hoepfner. Improvements in electrodes. June 5.

11,312. H. H. Lake. From La Société Electrogenique, France. Improvements in electric accumulators or secondary batteries. June 11.

11,587. L. P. Hulin. Improvements in the process of and apparatus for the electrolytic decomposition of solutions and for the immediate separation therefrom of the liquid or dissolved electrolytic products. Filed June 14. Date applied for, November 25, 1893, being date of application in France.

11,606. F. R. Davenport. Improvements in separators for electric storage cells. June 15.

11,664. T. Drake. An improved method, means, or process of producing caustic soda and chlorine gas, or caustic potash and chlorine gas, by the electrolysis of common salt in solution, or chloride of potassium in solution. Complete Specification. June 16.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

4106. L. Epstein. A process for maintaining positive plates of secondary voltaic batteries in good operative condition. May 30.

13,317. The Lithanode and General Electric Co., Limited, and J. T. Niblett. Improvements in secondary electric batteries. June 13.

13,336. C. Hoepfner. Improvements in the electrolytical production of nickel and other metals. May 30.

15,396. F. Hurter. Improvements in and connected with apparatus for the manufacture of chlorate of potash by electrolysis. June 20.

1894.

6046. L. A. P. Lienard and H. E. A. Lienard. Improvements in the methods of effecting the electrolysis of saline solutions. June 20.

7985. T. Drake. An improved method, means, or process of producing caustic soda and chlorine gas by the electrolysis of common salt in solution. May 30.

8917. H. Alexander. Improvements in or in connection with apparatus for the electro-deposition of metals. June 13.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

### APPLICATIONS.

9526. A. W. Winter. See Class XVIII.—A.

9888. N. Womersley and H. Mutell. Supo Gallienne. May 22.

10,222. H. J. Phillips. An instrument for ascertaining the degree of fluidity of several oils at various temperatures in one operation. May 28.

11,324. F. B. Aspinall. Improvements in the treatment of cotton seed oil. June 11.

### COMPLETE SPECIFICATION ACCEPTED.

1894.

8676. W. H. Horton. An improvement in what is commonly called dry soap or soap powder. June 6.

## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

### APPLICATIONS.

9655. J. S. MacArthur. Improvements in making white lead. May 17.

9823. D. Coventry. Whitish, being an improved flat size-colour for painting and shading purposes. Complete Specification. May 21.

9,883. L. L. B. Meyer. New or improved manufacture of varnish or of a substance for use in making varnish. May 21.

10,230. E. J. T. Digby. A new or improved manufacture or material for "colouring," polishing, or burnishing. May 28.

10,424. P. F. Johnson. Improvements in the manufacture of plumbago or graphite, commonly called black lead. Complete Specification. May 29.

10,531. C. P. Shrewsbury and H. R. Gregory. Improvements in the manufacture of lead pigments. May 31.

10,676. E. F. Wailes. Non-conducting bituminous composition for protecting the surfaces of structures from waste or decay. June 1.

11,398. W. G. Murray. A new and improved varnish for violins and other articles. June 12.

11,454. J. Hargreaves and T. R. Hewlett. Improvements in the manufacture of pigments. June 13.

### COMPLETE SPECIFICATION ACCEPTED.

1893.

12,979. J. W. Hall. See Class X.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

### APPLICATIONS.

10,151. E. T. Day and T. W. Matthews. Improvements in the treatment of raw hide, and the subsequent use and application of the same. May 25.

10,154. W. A. Lamplugh. A new or improved composition for cleaning, softening, and waterproofing leather, and for other purposes. May 25.

10,361. F. Searis-brick. Improvements in apparatus for boiling size. May 28.

11,381. W. H. E. Boulton. Improvements in or relating to the manufacture of cement, applicable for leather belting and other purposes. June 12.

11,426. P. C. Hewitt. Improvements in processes and apparatus for cooling glue and forming it into sheets. June 12.

11,678. W. J. P. Peacock. An improved method of drying albumen of eggs. June 15.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

19,481. N. Kumagaya. A new or improved material capable of being used in substitution for leather and for ordinary paper. June 6.

24,463. W. Zahn. Improvements in the art or process of tanning hides or skins. June 13.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

11,153. W. P. Thompson. — From 'The Actien Maschinenbau Anstalt vormals Vennet and Ellenberger, Germany. Improvements in the process of and apparatus for drying distillers' washes and the like, and for removing the acids therefrom. June 13.

12,609. P. W. May and B. E. R. Newlands. Improvements in and apparatus for sterilising or preventing fermentation or acetification of beers, beverages, or the like. May 30.

12,837. L. Lederer. Improvements in the manufacture of yeast and the distillation of spirits. June 13.

14,280. W. Watson. Improvements in and relating to the treatment of hops. June 6.

1894.

1717. R. R. P. Schmiedeecke and E. O. Gade. Improvements in apparatus for the preparation of malt. June 20.

### XV.—AGRICULTURE AND MANURES.

#### APPLICATIONS.

11,295. G. H. Richards. A new or improved composition or compound. June 11.

11,296. G. H. Richards. A new or improved composition or compound. June 11.

11,297. G. H. Richards. A new or improved apparatus for vaporising compounds or compositions for use in horticulture. Complete specifications. June 11.

### XVI.—SUGARS, STARCHES, GUMS, Etc.

#### APPLICATIONS.

9674. C. D. Abel. — From L. König, Germany. A process for deodorising and bleaching dissolved or fused amylaceous materials. Complete Specification. May 17.

9880. F. E. V. Baines. Improvements in the treatment of rice or other raw grain or other starchy substances for the production therefrom of a material suitable for use in brewing or for other purposes. May 21.

10,956. R. Wilson. Improvements in the manufacture of starch. June 6.

### XVII.—BREWING, WINES, SPIRITS, Etc.

#### APPLICATIONS.

9880. F. E. V. Baines. See Class XVI.

10,375. E. Manbré, J. L. Barrett, and C. N. Poehin. Improvements in the manufacture of malt and maltose extracts. May 29.

10,664. J. H. West. Improved means for cooling worts. June 1.

10,667. R. W. Preston and J. M. Hogarth. Improvements in or relating to the treatment of brewers' and distillers' mash, and the saccharine liquid or wort obtained therefrom. June 1.

11,019. A. Egestorff. Improvements in utilising spent hops. June 6.

11,310. H. H. Lake. — From P. W. Sothmann, Germany. Improvements relating to the ageing or maturing of wine, cognac, and other alcoholic beverages, and to apparatus therefor. June 11.

### XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

#### APPLICATIONS.

#### A.—Chemistry of Foods.

9526. A. W. Winter. Improvements in compound edible fats. Complete Specification. May 15.

9626. H. E. Hansen. Method of producing condensed milk. Complete Specification. Filed May 16. Date applied for October 27, 1893, being date of application in Sweden.

9765. T. P. Hawksley. A simple and effectual apparatus whereby milk may be sterilised or rendered absolutely free from disease germs and adapted for family use. May 19.

10,105. A. Bernstein. Improvements in the production of a peptonised alcoholic beverage from milk. May 24.

10,106. A. Bernstein. Improvements in the production of a peptonised beverage from milk. May 24.

11,393. W. F. E. Casse. Process and container for preserving milk, cream, and other liquids. June 12.

#### B.—Sanitary Chemistry.

9787. G. H. Wolstencroft. Improved means for precipitating the impurities in the waste or effluent water from bleach-works, dyeworks, and other places. May 19.

10,616. W. D. Scott-Moncrieff. Improvements in and apparatus for the treatment of sewage or other organically contaminated liquids. May 23.

10,228. W. H. Hill-Hartland. Improvements in deodorising and purifying sewage or other waste liquors containing organic matters, in combination with generating and storing electricity, and in means or apparatus therefor. May 28.

11,188. J. E. Keirby. Improvements in apparatus for supplying, precipitating, and purifying materials employed in the treatment of sewage and other analogous purposes. June 9.

#### C.—Disinfectants.

10,188. A. J. Boulton. — From F. Haase, Germany. Improvements in insecticides. Complete Specification. May 25.

10,445. C. T. Kingzett. Improvements in sulphur candles or fumigators. May 29.

10,816. S. A. Vasey and C. F. Townsend. Improved compound forming a disinfectant, deodorant, and antiseptic. June 4.

11,129. E. Clement. An improved wash or preparation to be applied to hop-vines and plants generally. June 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.

##### A.—Chemistry of Foods.

1893.

13,600. L. C. Tipper. Improvements in the preparation and treatment of animal and vegetable substances and fruit, and compounding the same with other ingredients for use as human food. May 23.

13,704. H. H. Lake.—From Baron Helfried Kaiserstein. Improvements in and relating to the manufacture of food for horses. May 23.

14,894. C. Ekin and W. H. Thew. Improvements in or relating to the manufacture or preservation of condensed milk. June 13.

15,595. W. Kuebel and F. E. Sahlfeld. A fish extract or essence, and in utilising the residual or waste products resulting from the production of the same. June 20.

15,757. J. Effront. Improved method and means for preserving food and other substances. June 15.

16,458. W. Brothers. Improved baking powder. May 30.

1894.

7543. E. O. Tatlin. An improved composition for use as food, and method of preparing the same. May 30.

##### B.—Sanitary Chemistry.

1893.

13,154. W. E. Adeney and W. K. Parry. Improvements in the purification of sewage and other like waste liquids, and in apparatus employed therein. May 30.

14,642. R. C. Tanner. Improvements in the treatment of sewage and other foul waters. June 13.

14,801. C. A. Burghardt. Improvements in and in the production of compositions adapted to be used in the treatment of sewage and other foul waters. June 6.

##### C.—Disinfectants.

1893.

14,411. I. S. McDougall and J. T. McDougall. Improvements in fumigants. May 30.

14,412. I. S. McDougall and J. T. McDougall. Improvements in the production of insecticides. May 30.

14,903. C. T. Kingzett. Improvements in sulphur candles or fumigators. June 6.

#### XIX.—PAPER, PASTEBOARD, Etc.

##### APPLICATION.

11,264. J. Hargreaves and T. R. Hewlett. Improvements in or applicable to paper making. June 11.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

12,420. W. Borland. Improvements in apparatus for mixing and regulating paper stuff. May 30.

19,481. N. Kumagaya. See Class XIV.

1894.

6221. G. Fehrenbach and L. Prud'hon. An improved process for producing artificial whalebone and method of forming same into strips. June 6.

#### XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

##### APPLICATIONS.

9675. C. D. Abel.—From C. Pieper, Germany. Improvements in the production of dextrins and leucocenes. Complete Specification. May 17.

9676. C. F. Cross and E. J. Bevan. Manufacture of cellulose acetate. May 17.

10,112. S. Pitt.—From The Chemische Fabrik auf Actien vormals E. Schering, Germany. The production of homologues of vanillin. May 21.

10,375. E. Manbré, J. L. Bairett, and C. N. Pochin. See Class XVII.

10,793. H. Baum. A process for obtaining the higher homologues of pyro-catechine. June 2.

11,128. E. H. Wild. Glucine. June 8.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

13,401. J. Bertram. Manufacture of geraniol,  $C_{15}H_{12}O$ , from citronella-oil. May 23.

13,478. A. W. Macellwaine and G. G. M. Hardingham. Improvements in or in connection with the extraction of oil by volatile solvents, and apparatus therefor. May 23.

21,417. A. R. Ling. A new improved process for the manufacture of anhydro-ortho-sulphaminobenzoic acid (orthobenzoic sulphinide) or of its salts, from commercial saccharin. June 20.

#### XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.

##### APPLICATIONS.

9479. R. Berl. An improved method of tinting or colouring photographic prints. May 15.

10,202. G. T. Teasdale-Buckell. Improvements in the production, by the aid of photography, of pictures or representations in colours, and in means or apparatus employed therein. May 28.

10,766. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Production of coloured pictures by means of diazo compounds. June 2.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

14,024. F. C. D. Beacham. Improvements in or relating to colours for and colouring photographs and other articles. May 30.

21,487. A. Burchett. Improvements in photography. May 23.

1894.

6590. E. J. Tobin and F. G. Bement. Improved actinic prints and improved method of producing same. May 30.



## XXII.—EXPLOSIVES, MATCHES, Etc.

## APPLICATIONS.

- 11,508. J. Hauff. Employment of trinitroresorcin as a less driving powder. Complete Specification. May 19.
- 11,533. W. Schael and C. Schael. Improvements in matches. June 5.
- 11,553. R. E. Brown and C. O. Lundholm. Improvements in the manufacture of explosive compounds. June 13.
- 11,490. W. J. Mimpriss. Improvement in wooden matches. June 13.
- 11,582. A. V. Newton. — From A. Nobel, France. Improvements in the manufacture of explosive compounds. June 14.
- 11,644. A. V. Newton. — From A. Nobel, France. Improvements in explosive compounds. June 15.

## COMPLETE SPECIFICATION ACCEPTED.

1893.

- 11,645. A. V. Newton. — From A. Nobel. Improvements in explosive compounds. June 6.

## XXIII.—ANALYTICAL CHEMISTRY.

## APPLICATION.

- 11,222. H. J. Phillips. See Class XII.

# THE JOURNAL

OF THE

# Society of Chemical Industry:

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## NOTICES.

Notice is hereby given that, in accordance with the alterations in Rule 27 sanctioned at the Annual General Meeting in Edinburgh, all new members elected after January 1st next, and also those elected after the date of this notice who do not pay subscription for 1894, will be required to pay an entrance fee of one guinea. In accordance with the same rule the Life Composition Fee will be 20*l.*, instead of 15*l.* as heretofore, after the 1st January next.

## BANKERS' ORDERS.

For the convenience of Members, the Treasurer has arranged with the Bankers of the Society that they shall collect subscriptions from Bankers in town and country; and Members, who have not already done so, are invited to fill up and sign the Banker's Order enclosed with the December number of the Journal, which should then be sent to the Honorary Treasurer, Mr. E. Rider Cook, East London Soap Works, Bow, E.

Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 41 of the bye-laws, the Society has the right of priority of publication for three months of all such papers. Infringement of this bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. EYRE and SPOTTISWOODE, the Society's printers and publishers, to whom all communications respecting them should be addressed.

Notice is hereby given that the next List of Members will contain the professions or business occupations of the Members of the Society. Any Member who desires to confirm or alter his description as given before election should communicate with the General Secretary.

Attention is directed to a table showing the course of average prices of general commodities in England, compiled by Mr. A. Sauerbeck, F.S.S., which is contained in the Trade Report of this number of the Journal.

## LIST OF MEMBERS ELECTED 18th JULY 1894.

Bishop, Jos. T. F., 123, Cottenham Street, Chorlton-on-Medlock, Manchester, agent.

Breyer, Theodor, c/o The American Glucose Co., Peoria, Ill., U.S.A., chemist.

Charlton, H. C., The Dales, Whitefield, near Manchester, cloth finisher.

Charlton, Robt., The Dales, Whitefield, near Manchester, cloth finisher.

Douglas, London M., 112, Tooley Street, London, S.E., chemical manufacturer.

Ingalls, Walter R., No. 12, Old Slip, New York, U.S.A., metallurgist.

Jenkins, Jno. H. B., Laboratory, G.E.R. Works, Stratford, E., analytical chemist.

Lenders, A. W. H., Portsmouth, Bay Co., Mich., U.S.A., technical chemist.

Marshall, Jas., 7, Buchanan Terrace, Paisley, N.B., chemical student.

Miller, Dr. Jno. A., Niagara University, 203, Ellicott Street, Buffalo, N.Y., U.S.A., professor of chemistry.

Nesbit, Jas., 19, Wellington Street, Portobello, Edinburgh, chemical student.

Parkes, Harry, 25, Halkyn Avenue, Sefton Park, Liverpool, copper smelter.

Rowlands, W. Ellison, 34, Tynwald Hill, Stoneycroft, Liverpool, paint manufacturer.

Scott, J. Gillespie, Annislea, Northfield, Liberton, near Edinburgh, analytical chemist.

Tucker, Alex. E., 35, Paradise Street, Birmingham, metallurgical chemist.

White, J. Freebairn, c/o Cassel Gold Ext. Co. Lim., 13, West Scotland Street, Kinning Park, Glasgow, chemist.

Wright, Sidney B., Lincarfe, Albemarle Road, Beckenham, Kent, analytical chemist.

Yocum, Dr. John H., 78, Gold Street, New York, U.S.A., chemist.

## CHANGES OF ADDRESS.

Allen, R. L., 1/o Heaton Chapel; Brookfields, Middlewich, Cheshire.

Babington, P., 1/o Dudley Road; 19, Culverdon Park Road, Tambridge Wells.

Barratt, Alex., 1/o North Wales; c/o Barratt Bros., 5, 26th Street, Birmingham, Ala., U.S.A.

Biggs, B., 1/o 3; Vestry House, Laurence Pountney Hill, E.C.

Bird, H., 1/o Mutley; South Down House, Millbrook, Plymouth.

Bloxam, A. G.; Journals to 16, Bolingbroke Road, West Kensington, W.

Boake, E. J., 1/o Highgate; 11, Christchurch Road, Crouch End, N.

Bradburn, J. A., 1/o Solvay Process Co.; 1816, West Genesee Street, Syracuse, N.Y., U.S.A.

Cooper, H. P., 1/o Upper Holloway; 2, New North Road, Hoxton, E.

Cowburn, W. H., 1/o Cheetham Hill; Sutton House, Ashley Road, Bowdon, Cheshire.

Crawley, A. H., 1/o Leeds; 205, Westminster Road, Liverpool.

Donald, Wm., 1/o Eglinton Street; Salteoats, Ayrshire, N.B.

Dreaper, W. P., 1/o London; 15, Prince's Road, Great Yarmouth.

Duncan, Jas., 1/o Biarritz; c/o Mrs. Mowbray, Gartmore Villa, Rocheliff, Dalbeattie, N.B.

Fogg, Jas., 1/o Glasgow; 720n, Old Kent Road, London, S.E.

Frew, Dr. W., 1/o München; 2, King James' Place, Perth, N.B.

Fulton, Robt., jun.; Journals to Baldorran, Salteoats, N.B.

Gilchrist, P. C., 1/o Westminster; Frogual Bank, Finchley Road, N.W.

Greiff, R. W., 1/o Mincing Lane; 3, Eastecheap, E.C.

Heal, Carlton, 1/o Leeds; Hertford Lodge, Church End, Finchley, N.

Haywood, J. H., 1/o Holland Street; Sparth Cottage, Manchester Road, Rochdale.

Holloman, F. R., 1/o Custom House; 396, Barking Road, Plaistow, E.

Horie, G. H., 1/o Nobel's Villas; Glendye Cottage, Stevenston, Ayrshire.

Ingle, Dr. H., 1/o München; Poel, near Leeds.

Joseland, W. H., 1/o Chesterton; Taike, near Stoke-on-Trent.

Joynson, F., 1/o Barnsley; Killamars, near Rotherham.

Kay, Dr. P., 1/o Leiden; Lilymount, Heaton Road, Manningham, Bradford.

Lowson, J. G. F., 1/o Beltonford; Polton Paper Works, Midlothian, N.B.

Marshall, Wm., 1/o West Hill; 119, Drake Street, Rochdale.

Napier, Jno. W., 1/o Loanhead; Chemical Works, Addiewell, N.B.

Pitts, J. C., 1/o Sheffield; Rycroft Glen, Dore Station, near Sheffield.

Procter, H. R.; Journals to Thornleigh, Wheatley Road, Ilkley, Yorks.

Saltmarsh, J. G., 1/o Manchester; School Lane, Lostock Gralam, Northwich.

Smith, Watson, 1/o University College, London; 34, Upper Park Road, Haverstock Hill, London, N.W.

Spiller, J.; Journals to Cleveland Villa, Southwold, Suffolk.

Strangman, J. Pim; retain Journals until further notice.

Taylor, W. G. H., 1/o North Bow; 68, Woodlands Road, Ilford, Essex.

Turner, H. B. H., 1/o Cambridge; 4, Calverley Terrace, Tunbridge Wells.

Watson, C. E.; Journals c/o Peter Spence and Sons, Alum Works, Manchester.

Watson, D., 1/o Great Clowes Street; Willow Bank House, Kersal Vale, Manchester.

Welsh, Jas., 1/o Hayfield; Horrocks Lane Dyeworks, Red Bank, Manchester.

White, Henry, 1/o Carlisle; 8, Brown Street, Masboro', Rotherham.

Whitehead, W. T.; Journals to Magog, Province Quebec, Canada.

#### CHANGES OF ADDRESS REQUIRED.

Gibson, A. M., 1/o 11, Queen Victoria Street, E.C.

Harris, T., 1/o Union Acid Co., Runcorn.

Young, B., 1/o Home Lyn, Woodberry Down, N.

#### Deaths.

Clark, D. G., 44, St. John's Wood Park, N.W.

Wright, Dr. C. R. Alder, F.R.S., 3, Castellain Road, Maida Vale, W.

## PROCEEDINGS

OF THE

## THIRTEENTH ANNUAL MEETING.

THE Annual General Meeting of the Society in Edinburgh proved a successful gathering. The arrangements made by Professor W. Ivison Macadam, F.R.S.E., F.I.C., the honorary treasurer of the Scottish Section, and Mr. J. Stanley Muir, B.Sc., honorary secretary, though in some instances modified by the exigencies of the moment, showed extreme care and ability.

#### FIRST DAY.

THE Annual General Meeting was held on Wednesday, 18th July, in Queen Street Hall. The President took the chair at 10 o'clock.

#### WELCOME BY THE CITY OF EDINBURGH.

BALIE DUNLOP, on behalf of the Lord Provost, in welcoming the Society to Edinburgh, said he was not exactly sure if there was much in the city that called for their attention as experts. The city was, however, noted for the eminence and excellence of its educational institutions, and it occupied a prominent position in respect of the history of the country with which it was identified. The city itself was incomparable in regard to its beauty, and he hoped that the members of the Society would carry away with them pleasant and profitable recollections of all that might come under their observation. He further trusted that the arrangements which the Corporation had made for the comfort of the Society would meet with their approbation.

THE PRESIDENT thanked Balie Dunlop, in the name of the Society, for his very warm expression of kindness. He congratulated the city on the fact that the Lord Provost had gone that day to Windsor to receive from Her Majesty the very richly merited honour of knighthood. Those who had the pleasure of knowing him, knew that there never was an honour more richly and handsomely deserved. He hoped that when the Lord Provost came back from Windsor as Sir James Russell it would be to the still greater respect and esteem, if it were possible, of his fellow-citizens, who would honour, not the knight, but the man.

BALIE DUNLOP said he would have pleasure in conveying to the Lord Provost the handsome terms in which they had spoken of him.

THE Minutes of the Annual General Meeting in Liverpool having been read and confirmed, the Secretary read the

#### REPORT OF COUNCIL.

WE have the honour to report that the number of members on the register is 2,828, as compared with 2,784 at the last Annual Meeting. During the year, 244 new members have been elected and seven members have rescinded their resignations, making a total of 251 gains, as against 208 last year; while 207 have been removed by death, resignation, and other causes, as against 204 last year.

Among those who have died are Mr. Alexander Crum, of Thornliebank; Professor C. W. Heaton, of the Charing Cross Hospital; Dr. J. M. Milne, of the Glasgow Royal Infirmary Medical School; Sir Alex. Orr Ewing; Mr. John Head, long associated with the late Sir Wm. Siemens; Mr. John May, founder of the firm of May and Baker; Mr. R. H. Davies, of the Apothecaries' Hall, London; Dr. P. Norman Evans; and Mr. Alfred Solvay, of Brussels.

During the past session, 77 original papers have appeared in the Journal, as against 74 last year.

The excess of revenue over expenditure, as will be seen from the Treasurer's report already in your hands, amounted last year to 37*l.* 17*s.* 9*d.*, as against 59*l.* 12*s.* 4*d.* in 1892.

The Committee on Patent Law has held several meetings, and embodied certain suggestions in a circular which was issued to each member of the Society. Having discussed these suggestions, the various Sections were unanimously in favour of petitioning Parliament to grant a Royal Commission to consider the whole question. Since the Council received this expression of opinion, the course of public business has been such that it has been held inadvisable to approach Her Majesty's Government lest, by inopportune action, the object in view should be defeated.

Experience having shown that some modifications of the bye-laws are required, those which the Council, after careful consideration, has decided to propose, and which the members will be asked to accept at this meeting, were published in the April number of the Journal. If accepted, these modifications will raise the Life Composition Fee to 2*l.*, so as to bring it into a fairer ratio with the subscription; will require new members to pay an entrance fee of one guinea; will somewhat modify the method of retirement of members of Council; will regulate communications between the Sections and other public bodies; but will otherwise make changes only of wording and of arrangement.

It is with great pleasure that the Council announces that it has acceded to the request of some of our fellow-workers in the United States to form a Section in New York. This is the first time that such a request has been preferred from abroad, and marks a new stage in the history of the Society, the importance of which cannot be too highly estimated.

To all those who have contributed to the work of the Society, our hearty thanks are due.

Mr. A. E. FLETCHER, Her Majesty's Chief Inspector of Alkali Works, moved the adoption of the report.

Mr. R. PULLAR, Perth, seconded, and the motion was agreed to unanimously.

#### THE TREASURER'S REPORT.

The GENERAL SECRETARY submitted the report of the Hon. Treasurer, already published in the Journal (this Journal, June 1894, 574).

Mr. D. HOWARD said he had great pleasure in moving that the report of the Treasurer be received and adopted, and that a vote of thanks be accorded to him for his services. It was no light matter to undertake the treasurership of a great society like theirs, and it would be impossible to find a treasurer who took more thorough, business-like pains with it than theirs. Every detail, he knew, had his most careful consideration. The management of their funds was carried on at almost nominal expense, due to personal care and attention of their Treasurer. It was therefore in no formal manner that he put the motion.

Mr. J. M. GIBSON, Chester, said he had great pleasure in seconding the resolution proposed by Mr. Howard. The whole thing was exceedingly satisfactory, and as a non-official member he was exceedingly grateful.

The resolution was adopted.

#### ALTERATION OF BYE-LAWS.

Mr. T. TYRER, London, proposed the adoption of the alterations of the Bye-laws as recommended by the Council, and referred to in the report. The proposed alterations were as follows:—

1. That *Rule 7* be amended by the deletion of "and" in the first line thereof, and by the addition of the words "and 47" in the same line.

2. That *Rule 15* shall read as follows:—

"Four Ordinary Members of Council shall retire each year, and those who thus retire shall not be eligible for re-election to the Council, except under Bye-laws 14, 23,

and 24, until the Annual General Meeting next following that at which they retire. When an Ordinary Member of Council is nominated for election to the office of President, he shall not be counted among those who are to retire."

3. That the first sentence of *Rule 16* read as follows:—

"Except that any Ordinary Member of Council may be nominated for election to the office of President, and that such member, if any, shall not be included in the operation of the present Bye-law, the Ordinary Members of Council shall retire as far as may be possible in the order in which they were elected."

4. That *Rule 17* shall read as follows:—

"When an Ordinary Member of Council is nominated for election to the office of President, the Council shall nominate a Member for election as an Ordinary Member of Council in his place."

5. That under *Rule 18* the eighth line be amended to read as follows:—"and also the name of the person, if any, who has been nominated, &c., &c."; that the form of nomination of candidates for the Council be amended by transferring the candidate's declaration to the end thereof; and that the words "and by the nominee" be added in the third line after the above-mentioned form after the word "subscriptions."

6. That the proviso at the end of *Rule 22* be deleted.

7. That in *Rule 26* the Nomination Form for candidates for Membership be amended to read as follows:—

"We, the undersigned members of the Society of Chemical Industry, hereby propose  
(candidate's name in full) as a fit and proper person to become a member of the Society.

For names only of members }  
recommending the candidate. }

"And I, the said candidate for membership, hereby declare that if elected I will do my best to support the Society and will observe its Bye-Laws.

(Signature) \_\_\_\_\_ (Profession) \_\_\_\_\_  
(Address) \_\_\_\_\_ (Date) \_\_\_\_\_

Extract from Bye-Laws (Rules 27, 29, and 30).

8. That *Rule 27* should read as follows:—

"Each member shall pay an annual subscription of twenty-five shillings, due on the 1st January in each year; except that any member may pay a life composition fee of twenty pounds in lieu of an annual subscription. He shall also pay on receipt of notice of election an entrance fee of one guinea in addition to his first year's subscription; except that should a member who has paid an entrance fee resign and subsequently seek re-election the Council may dispense with a further entrance fee."

9. That in the second line of *Rule 28* the words after "and" read as follows:—"issued to the members once every year."

10. That *Rule 29* read as follows:—

"No person shall be entitled to the privileges of membership who has not paid his subscription for the current year. He shall not attend meetings nor vote, nor shall he receive gratuitously the publications of the Society. Should the subscription remain unpaid eight months after it is due, the Secretary shall give the member notice that unless payment be made within four months after the date of such notice, his name will be struck off the register. At the expiry of the said period, if the member should still remain in default, the Council may order his name to be removed from the register, whereupon he shall cease to have any right, privilege, or interest in the Society, and the Council may notify the same in the Journal, and recover the arrears of subscription at law."

11. That *Rule 30* read as follows:—

"Any member may withdraw from the Society at the end of any year by giving previous notice to the General Secretary of his intention to withdraw; always provided that such notice, to be valid, must be accompanied by the payment of all subscriptions, if any, which may be due from

him to the Society at the date of the notice. By such notice and payment he shall be released from all further liability as a member."

12. That the following rule be added after *Rule 53* of the present Rules

"All communications between Local Sections and Public Bodies or Societies shall be conducted through or be at the discretion of the Council; and no publication shall be issued by any Section to the general public without the previous consent of the Council."

13. That *Rules 54 and 55* be placed after *Rules 24 and 38* respectively, and that the succeeding Rules be renumbered accordingly.

Mr. CHAS. DREYFUS, Manchester, seconded.

Mr. J. M. GIBSON, Chester, said he did not rise for the purpose of pressing an amendment. Personally he was not interested, but a large number of young men whom they wanted to bring into the Society would possibly feel the entrance fee a hardship. It might not be so, but he had heard from some of them that it was so, and he had promised that he would mention the subject at that meeting. Looking at the state of the finances there seemed to be no necessity for it. They put more money on one side last year than they ever did before. Of course, if they were to go into a scheme for palatial buildings, the money might be required, but in the meantime they had sufficient funds, and this proposal might have the effect of shutting out some young men. He would respectfully suggest that a ballot should be taken of the Sections before these proposals were absolutely made into law.

Mr. TYLER explained that the Council considered that one way of preventing the constant leakage of membership which took place was to impose an entrance fee, because they thought that a man would consider before he lost an entrance fee of 21s. This would be lower than in other societies.

Mr. WM. THORP, B.Sc., London, called attention to the fact that both their subscription and the proposed entrance fee were extremely small, especially when they took into account the Journal they gave to their members. Some members seemed to want the Journal one year and not the next, which was very much to be regretted.

#### THE POWERS OF THE SECTIONS.

Mr. G. T. BEILBY, Edinburgh, called attention to the 12th clause of the alterations, relating to the powers of Sections. The subject, he thought, was of sufficient importance for the General Meeting to take some note of it.

Mr. HOWARD pointed out the importance of a Society like theirs being thoroughly unanimous, and the analogy there was in other similar bodies, such as Chambers of Commerce. In the majority of cases it was desirable that the whole weight of the Society should be thrown in any particular representation. It came very much stronger if, by waiting a very few weeks at the outside, the recommendation of a Section could go forward to any public body, or to the public, as the recommendation of the whole Society. This would be far better than that the Council should have to explain that the action of a Section was the action of a Section only, and not of the whole Society. There was just one other risk to be guarded against, and which every Section would desire to guard against for themselves, but in which they might possibly fail from not knowing what was going on in other Sections. They might find one Section opposing another. In such a case it was extremely desirable that the Council should know what was going on, and should have an opportunity of pointing out how a very different feeling might exist in another Section. The object of the rule was in every movement, if possible, to give the entire weight of the body, and to make it clear that it was not the opinion of a section only, but of the whole. They would thereby prevent chance and unintentional misunderstandings between Sections, a danger which

was not inconsiderable in public utterances. Their experience was that by extra care and extra consideration there was a gain and not a loss.

Mr. TYLER said that the object of the adoption of this rule would cause all influence to gravitate to the central institution was the furthest possible from the fact. There must be some centre, but still they should be a great federation, all bound together and no part separable from the other. Any man who looked at the foregoing could not fail to see that the same spirit animated them all. He had never seen the slightest indication whatever in the Council of a desire to criticise mildly, or to question motives, when any particular subject was brought forward from any particular Section. On the contrary, the wish of the Council was that if there were a strong movement on the part of any local Section in any matter, it should be confirmed in a broad spirit.

Replying to a question by a member, Mr. TYLER said he should say that the expression "local section," meant "local committee," because a local committee was assumed to represent a Section.

Dr. STEVENSON MACADAM, Edinburgh, was very glad that the subject had been brought forward. It appeared to him that there was no case whatever for interfering with the mode in which local Sections conducted their affairs. It appeared to him, besides, that they would be introducing a most dangerous element, because local men would be always thinking of what the Council in London might say about a matter. The Council in London might not have heard the evidence, nor seen the witnesses, and would, therefore, have to depend upon representations to be forwarded, which must be imperfect, and which might be partisan. At present if a Section forwarded a memorial to a public body, it was only a memorial from that particular Section, and did not bring the Society into any invidious position, even although the Section were going wrong. It was said that the Section would be strengthened if the Council in London supported them, but it would be the duty of the Council to support a Section if it approached a public body in a proper manner. Then and then only should come into force the element which had been referred to, viz., the Council giving the weight of their support to the Section. It appeared to him that the proposal would affect the freedom of action of the Sections. The wording was quite clear: the Sections were not to take action except after consulting the Council, and it appeared to him that there were questions affecting the industry which local Sections might have better knowledge, and better means of considering, than the Council.

Mr. A. ERNEST POWELL, Manchester, thought clause 12 was a most desirable one in the matter of advertising. It would prevent some who read papers from using them as a pure advertisement, which had been done. He thought it was most desirable that no Section should approach the public without the intervention of the Council.

Mr. DANIEL HARRIS, Edinburgh, did not quite follow a reference made to Chambers of Commerce, for this reason: that there were no National Chambers of Commerce, although there was an affiliation. They were really one Society; that he admitted at once; but it did seem as if the Council had gone too far in the direction they had gone. He admitted the argument of the Council, but thought their proposal would need to be modified. They in Scotland had a subject they were very much interested in, and they wished the Council to take it up, and he could not say that their action would be hindered thereby.

Mr. HOWARD explained that the London Chamber of Commerce had a great many different interests, and it was quite possible that, say, the West African Section might have very strong views upon a certain subject, which might be sent up to the Government of the day as the information of the Section, which the whole body would not be prepared to accept.

Mr. C. DREYFUS pointed out that they had already, by their third rule, resolved that the Council should have the management of the Society, and it was in order to emphasise this rule that clause 12 had been devised. Some

Sections had not at all conformed to Rule 3, but had acted independently without any reference to the Council. It was to stop such independent action, which sometimes operated very curiously on the Society, that the suggested rule was made. Local authorities did not know, sometimes, that the resolution of a local Section was not backed up by the whole Society, and clause 12 would prevent such a misunderstanding.

Mr. TYRER said they must bear in mind that the Sections were represented on the Council, and had the power to very largely influence the Council. The constitution of the Society was such that no Section could possibly complain that it had not opportunities of discussing any question before it.

Mr. REILLY said he had cast no reflection upon what the Society had done in the past. He wished to maintain the *status quo*; he thought it had worked very well in the past. He therefore proposed as an amendment that clause 12 be deleted.

Dr. STEVENSON MACADAM seconded.

Upon a show of hands the amendment was lost, and the original motion was then adopted by a large majority, and the proposed alterations were adopted *en bloc*.

#### ELECTION OF OFFICE BEARERS.

The GENERAL SECRETARY read the report of the scrutators, which stated that 46 ballot papers had been received, and that all the candidates proposed for election had been elected.

The composition of the new Council is as follows:—

##### President:

Prof. T. E. Thorpe, F.R.S.

##### Vice-Presidents:

Sir F. A. Abel, Bart., F.R.S.	Sir John Evans, K.C.B., F.R.S.
Hornee T. Brown, F.R.S.	E. K. Muspratt.
H. Brunner.	Dr. W. H. Perkin, F.R.S.
John Calderwood.	John Spiller.
Chas. Dreyfus.	E. C. C. Stanford.
Dr. A. Dupre, F.R.S.	Prof. W. A. Tilden, F.R.S.

##### Ordinary Members of Council:

R. Forbes Carpenter.	Prof. A. K. Huntington.
Frank Clowes, D.Sc.	Manning Prentice.
Thos. Fairley.	G. X. Stoker.
C. A. Fawcitt.	Wm. Thorp, B.Sc.
Prof. G. G. Henderson, D.Sc.	T. Tyrer.
David Howard.	Frank Wilson.

With the Chairmen and Secretaries of Sections.

##### Honorary Treasurer:

E. Rider Cook.

##### Hon. Foreign Secretary:

Ludwig Mond, F.R.S.

#### THE PRESIDENT'S ADDRESS.

When the Council of this great and influential Society invited me to become President, I fully realised for the first time the feelings of the old Highland retainer, when he was told to "come awa' and pe trooned the please the laird," seeing that whatever use my small efforts might be to the Society, I should certainly succeed in obliterating myself. But then it occurred to me that self-sacrifice is a Christian virtue, not too common, and that probably such an excellent opportunity of practising it might not again be presented to me; and after all it must be only a small sacrifice that I could offer on the altar of the Society we all revere so much and love so well.

With these feelings, therefore, of a stern sense of duty, I reluctantly accepted this honourable and difficult position, and assumed a chair which I have promised to occupy, but not to fill. Honourable, because any man may be proud of

having a band or even a finger in the building up of this Society, which is evidently destined to revolutionise the chemical industry of this country, by giving it that scientific character which must ever be the only sure foundation. Difficult, because it is no easy task to follow the eminent scientific men who have preceded me, and find something to say in the annual address which one or other has not already said infinitely better. We all abhor a vacuum, and perhaps there is nothing much nearer it than an empty address, and it is so easy to say something and so difficult to find something to say. Fortunately, however, our annual meeting offers many compensations for the penalty of listening to an indifferent address, and these will be amply secured in this beautiful city which the Society now visits for the first time. We have met once before in Scotland, in its great commercial centre, the city of Glasgow, celebrated, amongst other things, for the great extent and variety of its chemical manufactures. We shall not see the larger manufactures here; the tall chimney of the alkali works does not yet adorn the sky-line of this city, but there are many important specialities as well worth seeing; and we all owe her a deep debt of gratitude as the birthplace of chloroform. Modern Athens, with her lovely neighbourhood, combining mountain and flood, and full of historic traditions, is always attractive, and the hospitality of her citizens is proverbial.

We are also visiting our most northerly and one of our most active Sections, completing its decade this year, and hitherto the only one which represents a nation.

The progress of this Society is shown in the annual report presented at this meeting, and I have only to congratulate the members that it has so largely exceeded the most sanguine anticipations of the first promoters; that, although still in its teens, it presents all the characteristics of early and vigorous manhood. The Council, fortunately from the first, wisely recognised the paramount importance of a thoroughly progressive and educational journal; there was nothing of the kind then, and now nothing so good exists elsewhere. The Journal is so fully illustrated, and so cosmopolitan, and the field of view so comprehensive, that it appears to touch every trade and manufacture in the country, and that makes it deeply interesting to a very wide circle of readers. 3,200 copies are at present circulated all over the world, and we have reason to believe that it is as much studied and valued abroad as at home.

Whilst duly recognising the great value of the Journal, the members of this Society generally little know how deeply they are indebted to the watchful, unwearying labours of the Publication Committee, meeting monthly in London, where all communications are discussed and submitted to the most minute inspection. So long as they continue to devote so much time and attention to it, so long will it remain the first journal of applied chemistry in the world. Some of the best work of the world is done for nothing, but we ought all to be grateful to them for this devoted labour of love. So many valuable papers, and so much information, are contained in the twelve volumes already published, that I hope we may soon be able to afford a full and well-classified general index.

Is not our Journal now rather ahead of the national scientific and technical teaching? No doubt much has been lately done in this direction, but we are still far behind, especially in organic chemistry. According to Lunge, they have at the Polytechnic School at Zürich four full professors of pure and applied chemistry, with four assistant professors, and a fifth chair is just founded. Zürich has a population of 100,000; how many cities and towns of equal size have we with such advantages? Indeed, how many have we in this country, with 13 times the population of Switzerland and 25 times the revenue. Contrast this with the position of the Glasgow Technical College, founded as a people's university in 1796, in the second city of the kingdom, with nearly 3,000 students, doing a great educational work, but sorely hampered for want of funds. Or with our great metropolis, the largest and wealthiest city in the world, without an educational university. We ought for shame to hide our national head in our national pocket, which is quite big enough, if we will not put our hands there to supply such educational needs. Fortunately, we have at least one private citizen who has come from

Germany to reside amongst us, and who not only can point out our needs, but is ready to supply them. The munificent gift of Dr. Mond of the Davy-Faraday research laboratory to the Royal Institution is a noble contribution to our national scientific research. The modest and quiet way in which the large sum involved has been handed over astonishes all who do not personally know him. We who do know our former President, and our present perpetual honorary foreign secretary, as I hope he will allow me to call him, are not so much surprised at his great liberality; but we are proud indeed that he has enabled another former President, who presided when we last met in Scotland, and who occupies the chair of Faraday, to carry out what he proposed 50 years ago. I think not the least part of the value of this great gift will be the advantage of Dr. Mond's great experience in fitting up the research laboratory. We all wish the greatest success to this grand work, the national importance and results of which it would be impossible to over-estimate.

The main work of this Society is done in the various Sections, of which we have seven spread over the country, and all in such active work that the London Section has been obliged to call extra meetings to cope with the large number of important papers submitted to them. These sectional papers are remarkable for their great wealth of illustrations, and for the valuable discussions so carefully reported in the Journal, and thus appealing to a world-wide circle of chemists and others. We enter now upon a new phase: another national section has just been founded in New York. We have 310 members in the United States and Canada, and we heartily welcome our new Transatlantic Section, which, under the able chairmanship of Mr. A. H. Mason, is already sure of all the success we wish for it. We are pleased to add the stars and stripes to our highly respectable old colours, and to shake hands across the ocean with the great English-speaking race who "shall brothers be for a' that."

We have also 123 continental members, 103 in the colonies, and 21 in Japan; altogether we have 578 members residing abroad, or more than one-fifth of our whole number; but this is the first time any have been represented by a separate section, and I think the movement should be warmly encouraged and extended. Our Society furnishes an excellent combination of decentralisation with a central head directing. We have thorough local home-rule with imperial government; we have unity with strength, and peace with honour, and long may it continue so!

It has already done much to bring manufacturing chemists, who generally walked on opposite sides of the street, together for discussion of the many interests and difficulties they have in common.

We may look on the Journal and the Sections as now so well established that other fields may attract the attention of our future councils. This Society occupies a central and important position, which is quite unique, holding out the hand to assist science on the one side and commerce on the other.

It forms a connecting link between scientific societies and chambers of commerce, and will be essential and invaluable to both. You have only to look over the Journal to see that there is scarcely any science that is not introduced into its columns, or any trade which it does not affect.

We are the industrial and industrious descendants of the old alchemists, who spent fruitless days and weary nights in trying to discover the philosopher's stone, which was to turn everything to gold; we are still engaged in trying to extract the root of all evil from everything around us, and sometimes we also imitate our ancestors in putting more gold in than we take out.

The chemical manufacturer of to-day must practically know a little of everything. He must not only have a thorough knowledge of chemistry and physics, but he must be a good mechanical engineer and understand machine construction and mechanical drawing. Hence has arisen the important science of chemical engineering, the essential characteristic of which is that it deals with the devising of apparatus on the large scale with almost every known material. He has, moreover, to deal with all sorts of solids,

liquids, and gases, of all degrees of violence, of all kinds of gravities and pressures, in every variety of vessel, in shape, size, and material. The common laws affecting only water and air are not enough for him. He must also know electrical engineering, and may perhaps require to know this best. In fact, many of the greatest successes of the present day are due to increased knowledge of chemical engineering applied to old and well-known processes. The vacuum pan, the multiple evaporator, the filter press, the gas and petroleum engines, the mechanical and other refrigerators, the electric furnace, compressed gases, osmose apparatus, superheaters, and a host of other appliances have placed great powers in his hands, and he must know how to use them to the best purpose.

It was not always so. I have heard the question actually disputed as to whether it was at all necessary for a manufacturing chemist to know anything about chemistry, and have even known some who boasted that their success was due to their ignorance of the science. I remember, many years ago, a manufacturer showing me over his works, and, bestowing a hearty kick on a passing worker wheeling a barrow too slowly, remarked to me, "that's what I call chemistry," making me admit how little I knew about it, or about his rule-of-thumb or rule-of-toe method.

This was before the introduction of the Alkali Act, which has done much to improve our manufactures, and this year has placed 14 new groups of works under supervision, many of which required it badly. It is satisfactory to know from the Alkali Inspector's last report that the escapes of gases are now only about one-third of the permitted legal limit.

35,000 tons of sulphur are now produced annually from the obnoxious waste heaps, and this improvement may be traced back to the operations of this Act. But it is surprising to find that 15 per cent. of the sulphur passes through all the condensers. We did not expect that sulphuretted hydrogen and sulphurous acid would go off harmoniously together, but then neither could we anticipate that one work alone would have to deal with 5,000,000 cubic feet in 24 hours of a mixture of these gases and air.

No doubt perfect success requires a trinity of science, practice, and commerce; a perfect knowledge of pure chemistry, capacity for erecting plant and handling men, and commercial ability are qualities not often found under one hat; under three will do, if they work together. But there is still plenty of room for induction, observation, and experiment—at least our German rivals think so, for they go on different lines: they employ a large staff of really accomplished chemists, and keep many engaged on pure research. But then they believe in research and not in examinations; they keep their honours for originators. But we level down our future masters to a common pass examination, and the trade unions do the same for our men. Both repress excellence and exceptional ability—the very qualities which we ought to cultivate. And then what do we really mean by a works chemist? Is he not too often only a "tester"? And yet the manager in his too exclusive wisdom may be devoting his whole time to the saving of fuel and labour, little dreaming that some patient researcher in a far-off laboratory may be quietly undermining his whole work.

I think it could be easily proved that nothing ultimately pays better than original research, but it requires encouragement, for often the absolute necessity of living is against it, and a man may spend a lifetime on an important subject, doing great work, of which only those who survive him know the value. It is so true that "the human race is divided into two classes—those who go ahead and do something, and those who sit still and inquire why it wasn't done the other way."

The researcher will find that the latter class forms the bulk of his acquaintance. But is a man perfectly successful who fills his own pocket and adds nothing to the knowledge or welfare of the nation, and is he entirely unsuccessful who forgets his own pocket in his research after knowledge, but leaves the world better than he found it. I say no to both these propositions: I believe there is a higher life even in industrial chemistry.

Remember, for instance, our dear departed friend, Walter Wehler, whose name will never be forgotten by this Society,



which owes him so much: no one can say he made money on "his great work, but who does not revere the memory of the man who, as Dumas said, "cheapened every quire of paper and every yard of calico throughout the world." Or refer to another former President, W. H. Perkin, who still occupies a high position in organic research, and we hope he may long be spared to hew out new paths of inquiry: was he adequately remunerated for teaching the world how to make colours from a waste material, and for revolutionising the whole art of printing and dyeing?

The great success of these eminent chemists is not measured by their paltry money return, but by national results: they represent the higher life. This higher life can and ought to be promoted by this Society. Money is plentiful and should be profitably invested, but talent is scarce and ought to be encouraged. I think we might devote some of the former to bring out some of the latter: in other words, that prizes should be given to promote original research, as is done by some of the smaller societies abroad. The Industrial Society of Mulhouse, for instance, offers this year for various researches, 160 prizes in medals and sums ranging from 400 to 5,000 francs. Certain rules might be drawn up, and certain suggested lines of inquiry outlined and published in the Journal. Many manufacturers could suggest subjects worth working on, and some might be induced to assist in providing the money for the prizes under certain conditions.

I think also that our Society might be made more attractive to these flocks of young chemists who crowd all the gangways leading to the Chemical Society, under the impression that the title F.C.S. will give them a proper hall-mark qualification, which the President has assured us it does not. Now most of these young men will come into manufacturing laboratories.

May we not some day model our Society more on the lines of the Institute of Civil Engineers, which it resembles, and admit students, associates, and members, who must give evidence of careful training before election, and adopt for these the title of C.I. or industrial chemist, and make it a qualification worth having. These young men may pass the examinations of the Institute of Chemistry, they may join the Society of Analysts, they may swell the ranks of the Chemical Society, but they must join the Society of Chemical Industry: they may read the other journals, but they must read ours if they are to keep abreast of the progress of applied chemistry.

I am one of those who think that mechanical engineering can only be learnt in the machine shop, and that applied chemistry can only be learnt in the works. A young man going into it should begin by connection with this Society as a student; he should undergo a certain curriculum or pupillage with a member, as in the Engineers, who will testify, for instance, that he is well up in technical and proximate analysis, or that he has conducted a department in the works with ability, and that should be his hall-mark of qualification; that if he misbehaves—betraying laboratory or trade secrets, for example—he will lose it.

May we not hope in some such way to fix the foundations of this Society, so that it may ultimately rest on merit and merit alone.

A royal charter would be required, and a larger house and a better home in London would be useful to the Society in many ways.

Of course it must be very distinctly understood that I hold no mandate for the Council. These are only independent and crude suggestions which may lead to something better.

But we have a large field before us. We may assist the Society of Analysts in verifying analytical standards and promoting general methods in commercial analysis, and do away with the terrible names of "high and low chemists." We ought to help Professor Armstrong in his praiseworthy desire to reduce and classify the great bulk of chemical literature, and to secure monographs on special subjects, bringing this special knowledge up to date.

Every movement in technical education must be carefully watched and promoted by this Society. The Council have

therefore appointed Messrs. Prentice and Spiller, who will worthily represent us at the Technical Education Conference.

We may even some day get a curriculum, or some other substitute for pass examinations, doing away with crammers, and rearing men who will do us credit in the future and not in the past, who will be living and not stuffed.

Then we may assist commerce in such matters as the patent laws, for which we hope to secure a Royal Commission: then we may ascertain why it is possible under our present system to patent so many absurdities and impossibilities, and why foreign patents may be taken out in this country, not to introduce a new and valuable manufacture, but, by not working it, to keep it out.

We have got a select committee to consider the flash-point of mineral oils, and we shall watch its proceedings with interest. The Adulteration Act is not all that is required; food and drugs ought certainly to be separated in a new Act.

We may perhaps induce carriers to understand that dangerous goods are not rendered safer by being packed all together, but, on the contrary, often become more dangerous.

We may object to the serious way in which we are handicapped by not being allowed the use of pure spirit duty-free. Our President-elect may be able to help us here.

We might even some day venture to put a spoke in the wheel of those professional humbugs who induce credulous people to invest their money in schemes for making real silver out of quicksilver, or gold out of Dutch metal.

But perhaps this is too much to expect, for

"The world of fools has such a store  
That he who would not see an ass,  
Must bide at home and bolt his door,  
And break his looking-glass."

Now my first difficulty arises—the choice of a subject for the next part of this address.

My accomplished predecessor gave us an eloquent review of the progress of chemical industry during the last 50 years. I need not bring that up to date, for it is done in the Journal, but I was tempted to a prophetic view of the progress for the next 50 years. Chemical industry, however, consists of hard facts and stern realities, and is just one of those uncertain prospects where you should never prophesy unless you know, for it does not leave much to the imagination, except sometimes the profits. Then I could not estimate the possible future development of the microbes, for if they only kept up their present rate of progress, it seemed possible they might become the directors of the company, or the heads of the firm, and we the humble workers. For these minute bodies, whose deficiency in size is amply made up by their prodigious number and immense powers of reproduction, were bakers, brewers, and wine-makers long before we knew anything about fermentation or about them, but now we know they are constantly working with us or waging war against us. We thought that chemical industry could have nothing to do with living organisms, and now we find that many of our great manufactures are entirely dependent on them. But it is not to be always so; the accomplished French chemist Berthelot gives us a different view of the sweet bye-and-bye. In addressing the chemical manufacturers of Paris, he predicted our position a century ahead.

He said that in A.D. 2000 all locomotion will be aerial; chemical engineering will dig with diamond spades wells 4,000 metres deep, from which hot, pure water, free from microbes, will drive all the machinery on earth. No coal will be raised; there will be no agriculture, no animals reared, nor vegetables grown for food. All this will be made in the laboratory. Carbon from carbonic acid, hydrogen and oxygen from water, and nitrogen from the air.

Man will go about in bliss with "his small tablet of hydrogen, his crumb of synthetic starch or sugar, his small vial of aromatic spices, his mite of fatty matter, all manufactured economically and plentifully, independently

of season or climate, and all free from the microbes that now originate epidemics and war on human life."

The menu does not sound attractive to our grosser natures, though it may be suitable to the toothless, hairless, æsthetic spirits of that day, with the minute stomach and colossal brain; that halcyon time when the President's address will be actually more attractive to the then over-learned members of this Society than the annual dinner, and it will be a very poor one if it isn't. How the gigantic athletic female senior wranglers of that day will look down on these poor male scraps of humanity. But what will the "harp of thousand strings" be like in those days? Will there be any smoking concerts, and will they really care at all for the "warm precincts of the cheerful clay"?—with my best apology to the author of the Elegy.

But is this the way in which we are to continue our manufactures, when, having refused to listen to the excellent advice of Dr. Mond, and make the best use of our coal while we have it, we have gone on with our reckless extravagance, until we find our industries banished to the great coal-field of China? Has not boring into the earth, even at ordinary depths, been often found a trifle risky? How about the petroleum well at Baku, which blew out the heavy boring-tools and ran for four months before it could be capped, and ejected 100,000,000 galls. of oil, value 1,000,000*l.* sterling? or another in which the height of the column was 250 ft. and the pressure 200 lb. on the inch? Subterranean fires are not easily dealt with, and our volcanoes have not yet been controlled. However, Vesuvius and Etna are convenient for experiment; why not begin by putting bridles on these and drive them in double harness?

Coal will no doubt be further economised, and more power is yearly obtained from it, but there is still much to be done, especially in gasifying it for fuel; when it is exhausted no more can be got, and peat, if utilised, will not last us long; but petroleum may be continually produced if some theories of its formation be correct.

Electricity can never be properly called a prime mover as long as it depends on coal, but perhaps it is not hopeless to find some chemical reaction that may enable it to stand by itself.

Then there are other forces; there is the heat of the sun, which would take us to the desert of Sahara.

The waterfalls, which would remove us to those countries where "desperate rivers go about a-begging folks to dam 'em."

The wind. Scotland might have a look in here, for though we never make any use of this force, we have plenty to spare. Then we have the force of the rivers, the tides and the waves; the power of these may be seen at Wick, where they have tried their strength against our engineering Canutes and beaten them.

This reminds me that none of my predecessors have touched the boundless ocean; all that is left to me. I cannot grumble at any want of material, for even the land it covers is almost the only and by far the largest *terra incognita* to us; in fact, they have left me all the water, and three-fourths of the land as well. Unfortunately it is difficult of access, and that is a great difficulty. Our professional brethren may easily find iron in the sun's atmosphere, but if we are called upon to work it, we have to get there. No doubt it would be an advantage to have it in a state of vapour, but the difficulty of keeping a cool head would be insurmountable to the average chemist; of course I except Professor Dewar, who deals specially in frigidity, and is equal to anything in the way of force. A German chemist has proved to his own satisfaction that the moon is composed of iodine; it is difficult to disprove it, but if he had to work it, we should soon know. As O. W. Holmes says, "If a man asserted that the moon is a green cheese coagulated from the milky way, I would be puzzled, but if he offered to sell me a ton of this same cheese, I could sample it before I purchased."

That is where the theorists have the pull over us.

For the industrial chemist is paid only by results, often in inverse proportion to his ability; e.g., it may pay him better to boil down salt or old horses than to make alkaloids, though it may not demand so much chemical skill.

I do not intend to take the Society to sea, though there is no fear of the internal tosy turydyon which prevents the enjoyment of some, as its capacity for absorbing information is so great that it would not be likely to reject mental food of any kind, or leave it undigested under any circumstances.

I wish I could take up this great subject; it is such a grand solution of everything, though rather dilute; but the Chemical Society has made such a specialty of solutions, and spent so many evenings upon them, that it would be unfair to deprive them of almost the only one they have not yet investigated, especially as it is a pretty large order, and may keep them going for another century.

The subject would be particularly appropriate here—the scene of the great labours of Dr. Murray and Mr. Irvine, who are still working at the Scottish marine station for scientific research at Granton, and through whose kindness you will have an opportunity of visiting the only sea laboratory of the North, where the results of the "Challenger" expedition are being worked out, and many new results of their own added to the investigation.

I can only refer in passing to some.

They find that sea-water is alkaline from dissolved calcium carbonate, of which it contains 16 tons in a pure mile 100 fathoms deep; that the sea-bottom over 51,859 square miles contains 76.44 per cent. of this salt; that whereas river-water has 10 times the proportion of calcium carbonate to sulphate, in sea-water the proportions are exactly reversed. They have shown, by placing them in artificial sea-water, that the crustacea can extract the calcium carbonate to form their carapace from calcium in the form of sulphate, silicate, and phosphate; that the carbonate is derived from the sulphate by reduction into sulphide. They find that manganese nodules are constantly forming in the Firth of Clyde and other places. These gentlemen have not yet started a marine chemical industry, but it is not for want of material; and many other of their researches are most interesting to marine chemistry. No analysis can give us any idea of what the ocean really contains. If we admit that all the elements are partially soluble in a large proportion of water, what metal may not the ocean or its deposits contain, either dissolved from its vast bed or washed down by the great rivers. Silver has been found in the proportion of 1 centigramme to a cubic metre, and it has been estimated that 200,000 tons of it are there ready for some enterprising metallurgist who is fond of the small rupee to extract it. The presence of gold has not been satisfactorily proved; it was expected it might accumulate in the copper-sheathing of ships, and Messrs. Muntz obliged me with specimens of old sheathing, both copper and Muntz metal. Mr. Inglis, who kindly examined these for me, found both gold and silver, but not in larger proportion than usual.

The results were, per ton—

	Copper Sheathing.	Muntz Metal.
Gold.....	2 dwts. 21 grs.	1 dwt. 15 grs.
Silver.....	4 oz. 12 dwt. 9 grs.	5 oz. 3 dwt. 12 grs.

Besides the rich food-harvest of the sea, which costs us nothing but the taking, it is our only source of whalebone, spermaceti, and fish and blubber oils, and even the whales have been utilised in making extract of meat of peculiar flavour. It was at one time almost our only source of potassium, sodium, and magnesium salts, and of chlorine, bromine, and iodine. Dartnall has shown that the relation of bromine and chlorine is constant, 0.34 to 100. The chlorine amounts to 1.9 per cent., the bromine to 0.646 per cent. Bromine is easily detected, but iodine is very difficult of detection, as the proportion is only 1 in 291,000,000 according to my results. By the evaporation of 3,399 lb., or over a ton and a half, of Atlantic water, and crystallising out the salt, I obtained 123.5 oz. of a dark viscid liquid, of 1,352 sp. gr. From this liquid, with much difficulty, the specimen shown was extracted; probably no audience has seen it from this source before. The salt beds of Cheshire

containing neither iodine nor bromine, but the water of Wexford Spa, in Wexfordshire, is rich in both, containing 4.07 parts per million of bromine, and 5.21 of iodine.

But we can only "play on the sea-shore while the vast ocean of truth lies undiscovered before us." I can only speak of the fringe of the subject, and the fringe of the ocean is seaweed, and a lovely fringe it is in some places. Most of the marine products, fish, shellfish, and sponges, contain traces of iodine, the latter are rich also in bromine.

Our principal seaweeds are the fuci and the laminaria. In the Falkland Islands some seaweeds attain a gigantic size and length—one, the *Macrocystis Pyrifera*, extends to 1,500 feet. In the Gulf Stream, or rather surrounded by it in mid-Atlantic, a remarkable fungus grows—*Sargassum bacciferum*, forming what is known as the Sargasso Sea.

Some of the algae are largely used for food in China and Japan, but little in this country. We neglect what, no doubt, is a wholesome diet; and, unlike the fungi, there are no poisonous species.

The only well-known products are from the gelatinous species, some of which yield a gelatin of great setting power; these are the *Gelidium cornutum*, from which the Japanese isinglass is made; the *Eucheimia spinosa*, an Australian species; and the *Chondrus crispus* or carrageen, a valuable food, not so much used as it should be. Japanese isinglass was introduced 30 years ago, but the jelly is of too high a melting-point to melt in the mouth.

These gelatinous species contain no iodine nor algio, neither does the sea-grass, *Enteromorpha compressa*, though when dry it has a very strong odour of the sea.

Potash salts are the only commercial salts extracted from seaweed, but it always contains also those of magnesium. Professor Henderson has recently discovered traces of rubidium in these salts.

The fuci, which are not utilised now, at the beginning of the century were burned to supply the sodium carbonate which was used in the soap and glass works of Glasgow and Dumbarton up to 1845. I cannot ascertain what it cost from this source; it could not have been less than 100*l.* per ton, but they did not use so much soap and glass then. There was a large chemical work in the island of Barra, where it was said 80,000*l.* were expended and lost in making soap from kelp.

However, no more valuable book of reference could be compiled for applied chemistry than a dictionary of failures, if we could only get someone sufficiently qualified and courageous to undertake it. We are all grateful to the pioneer who shows us the way to the gold mine, and we ought to be equally grateful to the watchman who warns us away from the bog, the precipice, and the thief, which are much more common. Such a book would be a very large one, and many of our most successful men could write good chapters in it. Indeed, it has been said with truth "that the failures of some are eternities beyond the successes of others," and we all know that a retreat is always a difficult strategic movement.

There are always difficulties in the way: you may have difficulty of access to, or scarcity of, raw material; you may produce more than you can sell; or you may produce something new, for which there is no market.

You need not go outside Scotland if you wish to be inaccessible; it would take you longer to go to St. Kilda and back in the winter than to the centre of Africa, and there is no post and no telegraph, though it would make an excellent and much-wanted meteorological station. My own personal experiences in another inaccessible island in the Hebrides over 30 years ago would be interesting and amusing now, had I time to relate them; there was no chemical engineering in those days, and the modes of travelling very primitive, and the erection of works extremely difficult. Many a chemist, after perfecting all his arrangements for production, must have wished that his product was like Hodge's razors, "made to sell."

The commercial applications of iodine outside of medicine are of little importance, but I will refer to some. In medicine I only refer to iodoform, so much used for application to wounds, because this remarkable substance, with its peculiar and persistent odour, contains 96.8 per cent. of iodine, which cannot be applied to the skin without inflamma-

tion, and which is so entirely different in appearance and properties. Many attempts have been made to find an efficient substitute for this body without the odour, but none with similar properties and with anything like the same proportion of iodine, and the goal is still open. The iodine colours exhibited are mostly sodium salts of iodo- or iodo-bromo-fluoresceins.

It is used in the precipitation of gold and silver from weak copper ores treated in the wet way, and I am indebted to Mr. Inglis for some pretty specimens illustrating it.

Iodide of antimony and other iodides have been patented by Mr. Fawsitt, for the vulcanisation of india-rubber, especially for very elastic goods, such as wading boots. The reaction has not been yet explained, but the advantages are, that the process is much shortened, and the temperature required is so low that delicate fabrics are uninjured. This is shown in one of these fabrics, and in a pair of fishing boots which have been exposed to the heat and light of the Chicago Exhibition, and are as good as ever.

One of the little-known compounds of iodine is the iodo-mercurate of copper,  $\text{HgCuI}_4$ . It is not mentioned in ordinary text-books, though first described by MM. Willm and Caventon in 1872. It was patented by H. Crookes some years ago as a means of testing rise of temperature, but has not been used. It is simply prepared by adding solution of cupric sulphate to a boiling solution of Meyer's reagent; part of the iodine is liberated, and a black precipitate thrown down, which becomes bright scarlet on cooling.

At temperatures of about 150° F. this becomes black, and regains its colour on cooling. If painted on a bearing, it at once shows any heating long before it can do mischief.

In marine engines, where there are so many bearings, and some difficult of access, this should be useful.

Many mills also have been burnt down from this cause, and many patents taken out to indicate heating, but none so simple. The temperature of change can be altered by varying the ingredients. The colour is very like that of post-office red, which can be painted on in alternate slips. I have a tin case so painted, and when filled with boiling water the effect is at once seen.

I will now describe a new product, for which no commercial application has been found.

Hitherto the ash of seaweed only has been used, as if we employed oak and mahogany for fuel instead of making it into furniture. Nothing has yet been done in utilising the seaweed itself, although large quantities of the fuci might be obtained. Some products have been prepared, such as mannite and acetic acid, of which specimens are shown. Mannite is often seen on the frond of the laminaria as an efflorescence, and is probably the result of fermentation. Acetic acid was obtained by Stenhouse, by allowing the fucus to ferment in the presence of lime.

The larger algae are composed of a cellular fabric which amounts in the air-dry weed to about 10 per cent., and to which the name of algulose has been given. It hardens into a tough mass, easily turned, and quite permanent. The other important constituent, besides the salts, is about 20 per cent. of an insoluble albuminous substance to which the name of algin has been given; the remainder consists of mucilage, dextrin, and water.

I shall only allude to some of the prominent properties of this substance. Algin or alginic acid is a nitrogenous organic acid, soluble in all alkalis, replacing carbonic acid from the alkaline carbonates, even in the cold, and forming definite gelatinous salts, all of which have been described. It is very insoluble, especially in acidulated water. It resembles albumin, but is not coagulated by heat.

It differs from gelatin in giving no precipitate with gallo-tannic acid; from gelose, by not gelatinising on cooling; from starch, by giving no colour with iodine; from dextrin, from gum arabic, and pectin, by insolubility in dilute alcohol and dilute mineral acids, and by containing nitrogen.

The alginates of sodium, potassium, lithium, and magnesium are all soluble, forming very viscous solutions, having about 14 times the viscosity of starch. A 2 per cent. solution is at once coagulated and rendered semi-solid by a dilute mineral acid, and a 5 per cent. solution will scarcely pour.

The solution is coagulated by alcohol and acetone, but not by ether or glycerin, and by all the mineral acids except boric and arsenious acids.

Citric, lactic, oxalic, and picric acids all coagulate it, but acetic, butyric, tannic, carbolic, gallic, benzoic, pyrogallie, and succinic do not affect it.

It is precipitated at once by lime and baryta-water, insoluble alginates being formed. Magnesia, however, when added to alginic acid, combines with it, and these two very insoluble substances dissolve together in the water, forming a soluble salt.

Nearly all the metals are precipitated but not mercuric chloride—another singular distinction from albumin; mercurous salts give a white precipitate.

It is not precipitated by chlorine, bromine, nor iodine, nor by potassium silicate, chromate, and ferrocyanide.

The metals are very completely precipitated from their salts, forming gelatinous masses, some of which are soluble in ammonia; these are aluminium, iron, copper, nickel, cobalt, zinc, cadmium, chromium, manganese, tin (stannous), uranium, and platinum.

These all, on evaporation, form films insoluble in water.

Copper-ammonia alginate contains 17 per cent. of copper, as compared with cupr-ammonium, which only contains about 2 per cent. in solution.

The aluminium alginate is also soluble in caustic soda, and the solution, which is quite neutral, on evaporation dries to a film quite soluble in water.

Stannic, arsenic, and antimony alginates are also soluble in ammonia, and on evaporation yield films soluble in water.

Strontium alginate is also soluble in ammonia, which on evaporation becomes insoluble in water.

The alginates of barium, calcium, lead, and bismuth are all insoluble in ammonia.

Silver is only precipitated in strong solution, and then incompletely; it is soluble in ammonia, but on evaporation it dries to a red film with a brilliant silver mirror surface.

Ordinary sodium alginate or soluble algin dissolves perfectly in cold water, but the solution requires time; it has the advantage over gelatin of keeping indefinitely. It behaves exactly as gelatin does with potassium bichromate, the film becoming insoluble on exposure to light.

Alginic acid in an insoluble film form is easily coloured, and it dialyses well. It combines with the vegetable alkaloids or quinine, and also with aniline and chinoline and toluidine, forming soluble films.

Ammonium or sodium alginate, if mixed with one-third of shellac dissolved in ammonia, forms a tough film, which, by passing it through a weak acid bath, becomes a tough insoluble sheet. Many resins and oils can be combined with algin in this way. From the insolubility in acids, phosphoric acid can be easily separated from insoluble phosphates of calcium, iron, and aluminium by dissolving them in hydrochloric acid and precipitating with sodium alginate, the phosphoric acid combining with the sodium.

A variety of insoluble compounds may also be obtained by precipitating an insoluble alginate with an insoluble phosphate, borate, or silicate. Some of these compounds resemble bone, calcium alginate particularly.

Many colours are at once precipitated by sodium alginate, the alginic acid forming a mordant.

In fact one of the disadvantages of this peculiar substance is the ease with which it is precipitated, and another is its great viscosity, which prevents workable concentrated solutions.

In bringing this remarkable substance before you, I have only lightly drawn the main threads together of a subject in which a large amount of work has been done, in a small and unremunerative corner of chemical industry; but it is better to have worked and lost than never to have worked at all; if only as a humble pioneer "axing the way" to make it easier for the army of progress to follow.

One of the great advantages of these meetings is that they bring together for a few days members who work in so many different fields that their suggestions to each other are most valuable, and more particularly because they are not working in the same groove—the groove which we all drop into, more or less and sooner or later, in spite of

ourselves. Then we can always claim their sympathy and encouragement to all who try to add something to the general stock of knowledge, however small it may be. That encouragement which goes so far towards preventing a faint worker from coming to think that his mis-directed energies have been spent in vain.

"Letting down buckets into empty wells,  
And growing old with drawing nothing up."

and rather encourages him to be

"Still achieving, still pursuing,  
to "learn to labour and to wait."

I leave this chair to my able successor, in the full assurance that "Men may come, and men may go," but the Society of Chemical Industry will flourish in perpetual youth, and scientific research will more and more cover the ground of the chemical manufactures of this country.

Professor F. E. THORPE, F.R.S., President-elect, said that the pleasant duty had been placed upon him of expressing to their President their appreciation of the instructive and interesting address which he had just given them. Those who knew the President were thoroughly prepared that the address should be instructive and interesting, but he was quite sure that, whatever their expectations might have been, they had been realised to the full. He could only say, as a measure of his appreciation, that the address was characteristic of Mr. Stanford, and that it was worthy of him. He had ranged over a great variety of topics, some of which were very tempting, and upon which he dared say, some of them might like to dilate at some length. Time, however, pressed, and therefore, beyond making one or two very slight observations, he would not inflict himself further upon them. Some allusion had been made as to the necessity for the preparation of an index of the publications, in their various forms, of the Society. It was perhaps a very small matter in one sense, but not in another. It was a most important one, but he must confess it had struck him rather forcibly that a Society such as theirs—which was very largely in the hands of business men, of practical men, to whom time was perhaps a more primary object than it was to the student—should have delayed so long in a systematic ordering, so to say, of the subject-matter of their publications. He wished to give, on the basis of his own experience, a slight word of warning. They in the Chemical Society had within recent times set themselves to the accomplishment of a similar task, and he assured them that it was a task which did not become easier by being the longer delayed. He would therefore earnestly press upon them, before it was too late, to take that matter in hand, and by the rapid and systematic publication, at periodically short intervals of time, bring, in a digested form, the valuable information of the Journal within the ken of all their members. Mr. Stanford had practically ranged over every department which chemical industry touched. He had made some allusion to the cosmopolitan character of their work, and he had dealt, with all proper emphasis, on the fact that they were now bringing within their fold not only English-speaking chemists, but chemists from all parts of Europe; in fact, wherever chemistry was taught, or wherever it had a practical application, from China to Peru, from Japan to America. He had also dwelt upon the extraordinary example which was afforded them by a very small Swiss canton, in the extraordinary thing they saw at Zurich, and he had made use of a phrase which he ventured to say, would become classical, that they "ought to thrust their national heads into their national pockets, for they were big enough, if they were not prepared to put their hands there." Some of them had ventured to think that that national pocket might undergo a process of shrinkage; but if they thought that the national genius of this country was the better for the publications of that Society, which was rapidly coming to the forefront as a kind of focus of information, they might take credit that they were doing for chemistry, and particularly for applied chemistry, much the same work. They ought, he considered, to stand in the same relation to the instruction and the practice of

chemistry, as they did in the diffusion and inculcation of knowledge through the instrumentality of their Journal. There was just one aspect of the extremely interesting exhibit which had been shown there, and which was so entirely bound up with Mr. Stanford that he should like to make one reference to it. Mr. Stanford had identified himself with the investigation and practical application of that most interesting substance alginic acid, and he had said something about the useful purpose which a Society like that served, in bringing together ideas from every department of chemical work. Now, it had struck him very forcibly, during the account which Mr. Stanford gave of the purposes of that remarkable body, that it would have a very considerable future before it in a certain department of scientific chemistry. He had alluded to the part which the Chemical Society had played in a theoretical journal on the subject of solutions. He himself had furnished an admirable illustration of how those substances might subserve their interests in the further development of that particular subject.

Mr. HORACE T. BROWN, F.R.S., seconded the motion, which was agreed to unanimously, and the President returned thanks.

#### APPOINTMENT OF AUDITORS.

Dr. J. CLARK proposed, Dr. J. B. Readman seconded, that Theobald Bros. and Miall be appointed auditors at a fee of ten guineas. This was agreed to.

Professor CRUM BROWN, Edinburgh, proposed the election of Mr. E. Rider Cook as the Society's representative on the Board of the Imperial Institute. He might, perhaps, be allowed to say a word upon another matter, and that was, the University authorities would be very happy to see any fellows or members of the Society at the chemical laboratories in the New University Buildings. Those of them who were interested in laboratories would, he dared say, see something of interest to them there.

Mr. R. IRVINE, Edinburgh, seconded, and the motion was agreed to.

#### NEXT YEAR'S PLACE OF MEETING.

Professor SMITHELLS, of the Yorkshire College, invited the Society to visit York-shire in 1895. It was his very pleasing duty, he said, and his last act as Chairman of the Yorkshire Section, to ask the Society to honour them by visiting Yorkshire next year, and by holding their meeting, in all probability, in Leeds. In endeavouring to get them to accept that invitation, fortunately he had not to try to persuade them to leave Edinburgh, because they were bound to leave Edinburgh. He need not enlarge upon the claims they had in Yorkshire. They were the youngest Section of the Society, and on that account they were pleased to receive the solicitude of their parent; and they were extremely central so far as railway communication was concerned. In Leeds they had a great variety of chemical industries, and they had, in Leeds, on the whole, from an industrial point of view, a greater variety of industrial manufactures than they would find in almost any other town in England, and perhaps in Scotland. Apart from that, they had educational institutions of all kinds, ministering very specially to chemical industry, and he was sure that on behalf of all, and particularly of the York-shire College, with which he was connected, they would offer them a hearty welcome. Another claim they might put forward was that Leeds, although not in itself of great attraction, was situated in a very beautiful neighbourhood. If they decided to accept that invitation, it would be a source of great pleasure to them in that district to have the Society under the presidency of one who had been so closely associated with Leeds in the past, and whose name was very highly honoured in that neighbourhood.

Mr. T. FAIRLEY, Leeds, said he had very great pleasure in seconding the invitation. They were a comparatively young and inexperienced Section, but what they wanted in knowledge and experience they would try to make up in zeal and endeavour to make the meeting a successful one. They had no very great succession of chemical industries

in Yorkshire, such as they had in some places upon the Tyne, the Clyde, the Mersey, or the Thames, but their river had a yield in colour and smell as large as any river in the United Kingdom.

The invitation was accepted.

On the motion of Mr. Irvine a vote of thanks was accorded to Mr. Stanford for his services in the chair and as President, and the proceedings terminated.

#### VISITS TO WORKS AND PLACES OF LOCAL INTEREST.

After the morning's business was concluded, the first excursion was made by coach to Roslin and Rosslyn Chapel.

##### 1. ROSLIN AND ROSSLYN GLEN AND CHAPEL.

###### *Excursion A.—Leader: Robt. Irvine, Esq.*

A large party set out by coach to inspect these attractive points of interest. Roslin is delightfully situated about seven miles from Edinburgh, and forms a centre of classic interest immortalised by Sir Walter Scott, and perpetuated by the poet Drummond of Hawthornden. Rosslyn Glen is the most romantic and picturesque spot anywhere in the Edinburgh neighbourhood. The Castle was the seat of the St. Clairs, Earls of Orkney. It stands on a rock overhanging the Esk. Three victories were achieved in one day in 1303 in its vicinity by the Scottish forces over the English. Rosslyn Chapel is a brilliant example of Gothic architecture. It was founded in 1446, and consists of a chancel and transept, and is divided into centre- and side-aisles by rows of exquisitely sculptured pillars supporting Saxon Gothic arches.

##### 2. THE PAPER MILLS OF MESSRS. ALEX. COWAN AND SONS, LTD., AND JAMES BROWN AND CO.

###### *Excursion B.—Leader: Dr. Stevenson Macadam.*

At the same time that the party for Roslin started, a somewhat smaller one also set off by coach, under the leadership of Dr. Stevenson Macadam, for Penienik. Since the important matter of the prevention of river pollution by the two paper mills in question is under the immediate supervision and inspectorate of Dr. Macadam, it may be anticipated that the interest of the visitors would be fully satisfied by the able demonstration given. In Messrs. Cowan's Valleyfield Mills four paper machines are employed for the manufacture of about 100 tons per week of the higher qualities of writing- and printing-papers, the materials used being chiefly rags and esparto grass, with also a small quantity of wood-pulp. The treatment of the waste esparto liquors is carried out by processes of filtration upon ash filter-beds, settling the sludge, running off cleared liquors, &c., the final effluent being, for such an effluent, surprisingly clear, non-alkaline, and only containing 100 grains of solids per gallon at the outside. Dr. Macadam is justly proud of the success of his remedial process, and especially as the result has proved to have repaid the labour and expense, and to be yielding a small profit. The settled sludge already referred to is filtered, pressed into blocks, or finally utilised for the production of an inferior paper.

The visitors who enjoyed the privilege of hearing Dr. Stevenson Macadam's demonstration of the working of the remedial measures against river pollution, and of the entire process of paper manufacture, both at Messrs. Cowan's and the Esk Mills, will not soon forget the lucid manner in which the descriptions were given.

After the inspection of Messrs. Cowan's mills, the party was most kindly entertained to tea and refreshments at the romantically situated residence close to the mills, by Mrs. Cowan, assisted by her daughters. It was a typical example of homely and hearty Scottish hospitality.

The Esk Mills of Messrs. Jas. Brown and Co., Lim., are situated in the beautiful valley of the North Esk, and the raw material here employed is also esparto grass, from every two tons of which about one ton of paper is manufactured. Processes are carried out in connection with the remedial

measures for recovering the alkali used, and whilst 80 per cent. of the latter is recovered at Messrs. Cowan's, no less than 83 per cent. is returned in the Esk Mills. This Dr. Macadam puts as equivalent to getting the alkali at half-price. In the Esk Mills a multiple effect evaporator (Yarvan system) has lately been introduced for promoting efficiency and economy in the soda recovery. On the return journey Dr. Macadam, determined if possible to beat any other record, promised the party a participation of the visit to Rosslyn Glen and Chapel if by any means possible; this promise was carried out most successfully, the party arriving in Edinburgh on the return journey at about 8 o'clock, and comfortably in time for preparing for the event of the evening, *à wit*, the

#### RECEPTION AND CONVERSAZIONE.

The reception and conversazione took place in the Museum of Science and Art adjoining the University, in Chambers Street. Here the members and friends, with their ladies, were received and welcomed in the magnificent hall by Bailie Dunlop, as representative of the Lord Provost of Edinburgh, the Magistrates and Council. The galleries and entire suite of exhibition rooms were thrown open, and whilst some of the company inspected the objects of interest, zoological, geological, and antiquarian, in this fine museum, others listened to music finely rendered by two military bands, notably that of the celebrated "Black Watch." Refreshments were served during the evening in the most bountiful fashion by an arrangement in which the usual difficulty of overcrowding and congestion, was well overcome. The function altogether was perhaps the most brilliant one the Society has yet witnessed at its Annual General Meetings.

#### SECOND DAY.—THURSDAY, JULY 19TH.

A new feature was introduced at the Edinburgh meeting, in the arrangements for visiting the numerous works, &c., which had generously consented to open their doors to the visitors, viz., the furnishing of daily programmes giving every necessary item of information, issued by the chief officers, to whose untiring energy and sound judgment so much of the great success of the meeting is to be ascribed; we refer to Mr. Robt. Irvine, the Chairman of the Scottish Section, Mr. J. Stanley Muir, the Hon. Secretary, and Professor W. Ivison Macadam, the Hon. Treasurer.

##### 1. MESSRS. THOMAS NELSON AND SONS, PARKSIDE WORKS, EDINBURGH.

*Leader: David Harris, Esq.*

A party started by coach to visit the above factory, in which the following industries were represented and illustrated:—Bookbinding, Illustration and Colour Printing, Map Engraving, and General Book Work.

##### 2. MESSRS. BERTRAMS, LIMITED, ST. KATHRINE'S WORKS, SCIENNES.

The visitors here had opportunity to inspect the processes of manufacture of general machinery. Especially there was shown in operation a large wall-planing machine, working either vertically or horizontally. Along with general paper-making machinery, the members had opportunity to inspect a paper-making machine of novel construction intended for despatch to Lucknow, India.

##### 3. MESSRS. JAMES AND THOMAS USHER, PARK BREWERY, EDINBURGH.

All the operations connected with brewing were here shown in working order. Messrs. James and Thomas Usher having been so good as to make special arrangements whereby a brewing was started and conducted during the visit of the members.

This notable and extensive business dates its history from the year 1828, when it was founded by the firm

whose title it still bears, at premises situated in close proximity to George IV. Bridge. Here the brewing industry of Messrs. Usher was successfully carried on until the year 1862, when the firm erected their present large and convenient premises, the Park Brewery. The firm has made the most of every advantage attaching to their new establishment, both in position and in working resources, and the Park Brewery at the present time ranks among the most conveniently arranged and most perfectly equipped in Edinburgh. The premises comprise several large blocks of substantial buildings, with spacious yards, the whole covering some acres of ground. The buildings are several storeys in height, and are laid out upon a well-considered plan, the various structures being admirably adapted to their allotted purposes as brew-house, malt-floors, malt-stores, barley-lofts, and very capacious cellars, under all. There are also hop-stores of large capacity, and the kiln equipment is eminently efficient. All the arrangements for malting and for the subsequent conducting of the liquor through the various stages of brewing are based upon gravitation principles, and in this way an appreciable saving is effected in steam power. The processes carried out at the Park Brewery are of the kind usually associated with the brewing of high-class ales and porter in accordance with the most approved modern methods; and the mash-tuns and coppers, as well as the coolers and fermenting tuns, are all of large capacity and of the best class and construction. The brewery water is of singularly fine quality, an unlimited supply being obtained from wells on the premises; and, as the Park Brewery stands at the outskirts of the city, and upon high ground, when its site is compared with many of the surrounding districts, the water here used is of assured purity, there being no chance of pollution.

*Leader: Dr. J. B. Rouburn.*

##### 4. MESSRS. WM. McEwan and Co., Ltd., FOUNTAIN-BRIDGE, EDINBURGH.

The brewing of Edinburgh and Export Ales was shown at this Brewery.

##### 5. THE NORTH BRITISH DISTILLERY CO., LTD., GEORGIE ROAD, EDINBURGH.

Here were exhibited maltings with grain conveying machinery; the milling and mashing departments, the distilling apparatus, and the draft and pot-ale drying machine.

Lunch was kindly prepared for the visitors here.

##### 6. THE BROXBURN OIL COMPANY, LTD., BROXBURN.

The visitors started by train from Waverley Station at 9.20 in the morning for Drumshoreland Station, and thence travelled by coach to the Oil Works, where they were most cordially received by Messrs. Kennedy, Henderson, and Stewart, who subsequently acted as guides and demonstrators, the party being broken up into three groups, each of which was led by one of the gentlemen named. After the inspection a bountiful luncheon was provided for the party, and the President, Mr. Stanford, then expressed in the warmest terms, on a cordial vote of thanks, the sense of indebtedness of the large party which had availed itself of this opportunity for the great pleasure thus afforded. The following report will give an idea what that indebtedness was, and what the pleasure must have been:—

##### BROXBURN MINERAL OIL WORKS, AND THE PRODUCTS FROM SHALE.

*The Shale.*

The shale is mined in much the same manner as coal, and conveyed to the breaking-machine in railway waggons. The waggons are tipped by hydraulic machinery into the large hoppers of the breaking-machine, where the shale is broken into pieces about three inches square. It is then conveyed to the retorts in hatches containing about 12 hundredweights.

### The Retorts.

The retorts (known as "Henderson's patent improved retort") are vertical, and 28 ft. long. In the upper portion, about 11 ft. long, and made of iron, the oil is first distilled off the shale at a temperature of 900° F. The spent shale then passes downward into the brick portion of the retort, and is kept in constant motion by a roller at the bottom, which delivers it into a hopper below at any required rate. This portion is kept at a temperature of about 1,300° F. This higher temperature largely increases the yield of ammonia and permanent gases from the carbon of the shale. The hot products of distillation are drawn from the top of the retorts in the form of oil and water vapours, and permanent gas, passed through a long range of cooling pipes, and the condensed liquids, on being separated, are called crude oil and ammonia water.

The incombustible or permanent gas is used as fuel.

The spent shale, being of no further use, is stored in a large rubbish bin.

The ammonia water is distilled with steam in a special column-still, and the ammonia is passed into sulphuric acid, and sulphate of ammonia produced.

The crude oil is refined and fractionated into various qualities of oils, such as lamp oils, gas oils, lubricating oils, solid paraffin, &c., &c.

### The Crude Oil.

The crude oil is refined by repeated distillations, during which it is fractionated into products having the different gravities required, and by treatment with oil of vitriol and with caustic soda. The distillations are according to Henderson's patent continuous system.

The first distillation gives—

Green naphtha.

Green oil.

Still coke (a valuable smokeless fuel).

The "green naphtha" is treated with oil of vitriol and with caustic soda, and after another steam distillation, is ready for the market as shale spirit or naphtha.

The green oil receives treatments with oil of vitriol and caustic soda, and is then ready for the second distillation.

In the treatments the oil is stirred first with oil of vitriol and allowed to settle, when a black tar falls to the bottom, and is run off. The oil is run by gravitation into another vessel, and is mixed with solution of caustic soda. On settling, another black tar separates out. The chemicals are recovered from the tarry matters, and the residual tars are used as fuel by being injected with steam into the still furnaces (in the form of a fine spray) and burned. The recovered sulphuric acid is used for making sulphate of ammonia. The oil of vitriol used in the oil refining is manufactured by the Company.

In the second distillation the green oil is fractionated into light oils and heavy oils, the latter (heavy) containing solid paraffin. The light oils are treated and distilled several times, and are ready for the market as burning oils.

The heavy oils are cooled below the freezing point of water, and the paraffin strained out by passing through filter-presses, the two products from the filter-presses being now termed blue oil and solid crude paraffin.

The blue oil is treated and distilled, fractionated into intermediate oils—used for gas making and other purposes—and heavy oils for lubricating machinery.

The crude solid paraffin is passed direct from the filter-presses to the sweating chambers (Henderson's patent, for purifying paraffin), where it is fractionated and refined to from 100° to 120° F. melting point, and it is then known as semi-refined paraffin.

If this paraffin needs to be further refined or made pure white, the sweating and fractionating process is repeated, and the paraffin is then known as refined paraffin wax of from 125° to 130° F. melting-point.

After the sweating process, the liquid paraffin is mixed with char, settled, and then filtered through paper, when it is cooled into blocks of paraffin wax, and is ready for making into candles, night lights, wax matches, or for other purposes.

The total refined paraffin produced is about 4,000 tons per annum, the greater portion of which is made into candles at the Broxburn Works, and sent over all parts of the world.

The candle works are extensive, and capable of making from 14 to 16 tons of candles per day.

The soft paraffin, melting-point 100° F., is called match paraffin, being now used for dipping the ends of the wood matches instead of brimstone, and also for burning in miners' lamps, and various other purposes.

The average throughput of shale is about 1,300 tons per day, the Broxburn seam yielding 32 gallons of crude oil and about 46 lb. sulphate of ammonia per ton, while the lower seams of the Roman camp shales yield about 20 gallons of crude oil and over 72 lb. sulphate of ammonia per ton.

Over 11 million gallons of crude oil are refined annually.

The works and mines cover an area of 250 acres, and there are about 1,700 men and boys employed.

The following samples are to be seen in the show-case:—

BROXBURN SHALE, from which the following products are made.

	Specific Gravity.	Flash.
		° F.
Crude oil .....	..	..
Sulphate of ammonia .....	..	..
Coke from crude-oil stills .....	..	..
Naphthas .....	0·725, 0·730, 0·740	..
Burning or lamp oils:		
Petroleum .....	0·800	110 to 120
No. 1 paraffin oil .....	0·806	120
Lighthouse oil .....	0·810	160
Marine sperm .....	0·830	230
Mineral colza .....	0·840	250
Gas oil .....	0·840 to 0·850	200
Lubricating oil .....	0·865	300
" .....	0·875	320
" .....	0·885	340
" .....	0·895	350
" .....	0·890	340
Crude paraffin .....	..	..
Semi-refined paraffin .....	..	..
" match paraffin .....	..	..
Refined paraffin wax .....	..	..
Candles, various, white and coloured ..	..	..
Night-lights .....	..	..

7. MESSRS. JOHN FORD AND CO., HOLYROOD GLASS WORKS, EDINBURGH (Glass Manufacturers to the Queen).

Leader: John Ford, Esq.

These works are the oldest and most complete in Scotland, having been originally built at the beginning of the century, in succession to those of a similar description, carried on simultaneously for a time, at Portobello; owing to locality and the enterprise of the firm, a great variety of work has been and is done, comprising every description of flint glass for scientific, lighting, domestic, and decorative purposes.

The richest quality of lead glass is generally made, and has a world-wide reputation for quality, colour, and brilliancy; all the chemicals for this and other special glasses are obtained from England, France, and Germany.



The furnaces and workshops cover a considerable area, and have been added to and improved from time to time. The principal furnace is stoked by a Frisbie feeder, and consumes from 3 to 4 tons of coal per week; splint coal is in favour. This furnace has been burning for ten years, and can melt 8 tons of glass at a time. The premises for the operations of cutting, sand-blasting, engraving, etching, &c., have been specially constructed for efficient production. The machines and showrooms are stated to contain the largest and most varied stock in Great Britain.

The most magnificent engraved service of table-glass has just been completed for H.R.H. the Duchess of York, the representative pieces of which were commanded by Her Majesty the Queen to be submitted at Balmoral. The work occupied 12 months, and has been carried out by the highest class of artists, either trained by the firm, or having a long term of service under the direction of Mr. John Humphrey Ford, upon whom have rested the whole details of design and manufacture. The firm is presently engaged on some work for H.R.H. the Princess Beatrice, and H.E.M. the Empress Frederick of Saxony, who has graciously indicated by her own drawing the line of design which is to be followed.

Many distinguished visitors, including H.R.H. the Prince of Wales, have seen the works, which have always been carried on by the Ford family; four generations have taken part at Holyrood.

Mr. John Humphrey Ford, with his brother-in-law and partner, Mr. William Ranken, assisted by the staff, were prepared to show visitors over the works during any of the days of the conference, without restriction.

#### 8. MESSRS. WILLIAM YOUNGER AND CO., LIMITED, ABBEY BREWERY, EDINBURGH.

The visitors had the opportunity here presented of seeing over one of the oldest established breweries in the country. Mr. J. Younger, Esq., one of the directors, acted as conductor and demonstrator.

#### 9. THE EDINBURGH GAS WORKS, CANONGATE, EDINBURGH.

For those interested in the gas manufacture an opportunity was here afforded of inspecting an economic mode of firing gas retorts, the Siemens system being employed, and a set of 16 retorts being worked by one producer.

#### 10. THE GLASS WORKS OF A. D. JENKINSON, OF ABBEYHILL.

An opportunity was here afforded of witnessing the manufacture of ornamental glass, Venetian and Art glass.

#### 11. VISIT TO THE FORTH BRIDGE, AND GARDEN PARTY, on the Invitation of MESSRS. A. B. FLEMING AND CO.

At 1.40 p.m. a large party started from the Waverley Station by train for Granton, where a steamer was in attendance to convey the members and friends to view the Forth Bridge. The weather being exceptionally bright and clear, fine views were obtained, and the great bridge itself was seen under the most favourable conditions. Luncheon was partaken of on board the steamer. Later on in the afternoon the steamer returned to Granton, and then to Royston, where the party disembarked and ascended the hill to the residence of Mr. A. B. Fleming, which is most beautifully situated, and where all the preliminaries for an enjoyable Garden Party had been arranged by the hospitable host and hostess. A Reception was given by Mr. and Mrs. Robt. Irvine. Subsequently an inspection was made of the following branches of manufacture in the extensive factory of A. B. Fleming and Co.:—Lamp Black, Printing Inks, and Solidified Oils. It may be interesting to record here that the patent solidified oil is the invention of Mr. Robt. Irvine, the Chairman of the Scottish Section, who is also the chemical adviser of the firm. This oil is not sensitive to either heat or cold, but under all circumstances maintains its uniform consistency. It is consequently unaffected by climatic influences. It is specially recommended for use in the shafting and journals of mills, there being no danger

of leakage or saturation of walls or woodwork. Among other specialities should be mentioned Messrs. Fleming's bloomless vegetable and mineral mixing oils, for which it is stated there is a great demand. An opportunity was also afforded of inspecting the Marine Station at Cathline Park, after which the party returned to Edinburgh by special train at 6 p.m.

#### 12. THE LOTHIAN COAL COMPANY, LIMITED.

A supplementary opportunity was afforded the visitors of inspecting the colliery of the above Company, and Messrs. Jas. A. Hood, Jas. Hamilton, Jas. Simpson, and Thos. Webster, Esqs., undertook to meet and conduct the party over the works.

Coal has been worked at this colliery for a great number of years, and the daily output averages about 600 tons. The coal produced is used for both house and steam purposes. The plant inspected comprises air-compressing machinery, compressed air being used for the purpose of driving coal-cutting machines and pumps underground. Dynamos for driving pumps underground and for lighting. Guided ventilating fan and engine.

At the brickworks, which are extensive, the plant includes two brick-making machines, semi-dry, which make something like 17 to 18 thousand bricks a day each. Brick-making by hand was also seen. Machinery for the purpose of manufacturing fire-clay pipes, &c. For the burning of bricks a large Hoffman kiln has been in operation for many years, and there are also kilns for burning the fire-clay pipes, &c. Machinery for the purpose of grinding clay for the manufacture of bricks was also exhibited.

#### THE ANNUAL DINNER.

The annual dinner was held in the Waterloo Hotel on the evening of Thursday, 19th July. Mr. Stanford occupied the chair, and had on his right Lord Provost Sir Jas. A. Russell, of Edinburgh; Mr. R. Pullar, Perth; Professor Crum Brown, F.R.S., Edinburgh; Mr. Chas. Dreyfus, Manchester; and Mr. Horace T. Brown, F.R.S.; and on his left, Mr. D. Howard, London; Dr. John Murray, Edinburgh, &c. Messrs. R. Irvine, Edinburgh; C. A. Fawcitt, Glasgow; and I. Tyrer, London, were croupiers. There was a large company.

The toast of "The Queen and the Royal Family" having been duly honoured,

Mr. D. Howard proposed "The Society of Chemical Industry." He said that the industry of chemistry might claim that in Scotland it had received a marvellously hospitable welcome. If they might believe certain utterances which they had heard at Burlington House, and elsewhere, they were in such a parlous state that to propose their health was a desperate task. Though he knew full well the difficulties that those comments brought in daily life, he knew also that in many ways Britons had greater disadvantages than their neighbours. They were told that their only hope of further existence was to model their works upon a certain pattern which was described, which pattern, he believed, was drawn from a particular works in Germany—a works which had had enormous prosperity, owing to the British system of patent law, which protected the foreigner to the destruction of home industry; a works which also owed its great profits to the manufacture of synthetic remedies which were inaccessible to us, on account of that strange law which some mase-line women and effeminate men had passed for the prevention of the progress of knowledge in physiological chemistry. Those were disadvantages, and there were many more which it was of no use repeating. Yet he did not despair, pessimist though he was, of the future of chemical industry in this country, and very largely because of the existence of that Society, and of the existence of the spirit which had brought that Society into existence, and which was not confined to the operations of that Society. If the industrial chemists of the United Kingdom thought it worth while to spend 3,000*l.* a year in order to inform themselves of the progress of knowledge in the matters of their business, at any rate they were not blind to what was going on; they did not shut their eyes to



what was to be learnt. So long as this spirit existed he thought they had nothing to fear; at any rate they would make it as good a fight as they could. Might he echo once more the motto that was constantly being spoken of, and yet, unhappily, not always acted upon,—“What they wanted was research.” He wanted to carry out that further than it was generally carried out. It was not alone such research as had been nobly illustrated by a past President of that Society; it was not alone the research which found its outcome in papers in the Journal of the Society. It was that same habit of mind which produced great discoveries, which was wanted in the every-day work of the laboratory. It was the same spirit of research which was required in every detail of their work, which was required to make great discoveries. He would urge it upon their juniors not to expect to be taught what they wanted, for the wisest of their teachers only taught them how to teach themselves, and if they found that what they had learnt was not what they wanted, let them not blame their teachers, but blame themselves. It was the duty of the teachers to teach them to learn rather than to teach them to know. There were no two factories alike, not even those who were working the same process and doing the same thing; every one had their own peculiarities, and the chemist was only fit to work in any and all who was not content merely to know what could be taught in the name of that mysterious entity—technical education, but had learnt the great lesson of working by himself. That was what the Society of Chemical Industry had endeavoured, by example and precept, to carry out; and, might he say, they were most happy in their present President, whose name he coupled with the toast, in being a brilliant example of carrying out, in practical life, what he had endeavoured to express in theory.

The CHAIRMAN, in acknowledging the toast, said that what was wanted was original research and not class examination. They did not want to cut down their men to a Procrustean level; they wanted to encourage them each to go higher than the rest. That was the principle which this nation had yet to learn, and the principle which must govern their Society of Chemical Industry.

Professor IVISON MACADAM, Edinburgh, gave the toast of “The Lord Provost and Magistrates of Edinburgh.” He said that the Lord Provost, when first approached, heartily promised to look after the Society; that he was not able to fulfil his promise, was due to the fact that in obedience to the command of Her Majesty the Queen he had to go south at that time. He could assure them that not only was the Lord Provost ready to welcome them, but the magistrates, and Bailie Dunlop, who took his place, thoroughly backed up the Lord Provost, and they had all been most anxious that the visit of the Society to their city should be most happy and successful in every way.

Lord Provost Sir JAS. A. RUSSELL responded. He said he must begin with words of apology for failing to keep his engagement to receive them at his own house. They had already heard the reason, and he had no doubt they would excuse him. At the time he hoped to meet them he was being introduced to Her Majesty the Queen by his old friend (Lord Playfair), whose lectures he attended rather more than 30 years ago; and he thought it rather a remarkable coincidence that at the time he ought have been with the Society of Chemical Industry in Edinburgh, he was being honoured by one who was so much connected with science. They were delighted to see the Society in Edinburgh, partly because, and largely because, they were glad to receive them, and show them their hospitality; but they also expected to derive benefit from the visit of the Society. He thought that a Society which could give such instruction as was given in the speech of Mr. Howard could not visit them without leaving permanent benefit behind. Edinburgh was largely an educational city. The Town Council had from time immemorial been closely connected with education in various forms. It had been connected with the University and with large educational trusts, and they thoroughly appreciated the vital interest of encouraging true education, and not the mere acquisition of knowledge. And he believed that, so far as in them lay,

they were anxious to further that research and the real knowledge of the advancement of science, which would, by the discovery of great ideas, lead to their application in practice, and to the material benefit of the country. Recently the Town Council came into a small sum of money, derived from the Government grant, and he was curious to see from the newspapers what was being done in other towns with that money. He found that many of them applied the grant to the relief of their pockets, by lessening their taxes; others of them applied it partly for educational purposes; the Town Council of Edinburgh passed a resolution that it should be entirely applied to educational purposes. Though they had not the numerous chimneys which were at once the source of wealth and disfigurement to other towns, they had some interest in chemical industries, and some of their neighbours had shown that they had the ability to make original researches, and to apply those researches to practical ends; and he believed that the visit of the Society would be, to those who were engaged in chemical industries, a most valuable stimulus, a means of encouraging them in their work, and a valuable means of educating the public to give their support to work dependent upon science. Mr. Howard confessed himself to be a pessimist, and evidently deplored a tax being made upon the accumulation of wealth. If there was anyone who wished to cheat the Chancellor of the Exchequer, and who wished at the same time to benefit, not only his own generation, but future generations, let such be commended to play the rôle of a pious founder. Their city had benefited by pious founders in the past; but possibly the class was not so plentiful as it was 300 years ago. Educational foundations had done an immense deal for their town, but they had still much room for similar foundations, applied with modern ideas; and if one result of the visit of the Society should be to turn the thoughts of those among them who had been prospering in the lines of chemical industry into doing something for the advancement of science, he was sure the Society would be pleased, and the city would have cause to rejoice at their visit.

Mr. R. PULLAR, Perth, proposed “The University.” He said they heard a good deal of foreign competition. He had seen a good deal of industries abroad, and he could only say that they had many difficulties there, and that if in this country they just set themselves to, and kept themselves up to the mark they should be pretty well able to hold their own. It was not a bad country this to work in; there were a great many advantages in it in many ways, but that one important point in which they could improve the business of the country was to get the very highest and best talent into it, which could only be done by providing thoroughly educated men. There was no doubt that a thoroughly good and suitable education for those who were to be at the head of industries was a good thing, and that two or three years spent at a University, or a high school, was not lost time. He thought the more technical knowledge they had the better; if they had the knowledge they should soon apply it practically. They had no better illustration of that than in the gentleman whose name he was going to couple with the toast—Professor CRUM BROWN. They all knew the enthusiasm he showed in his work, and the interest he took in the progress of chemical science as applied in the various arts.

Professor CRUM BROWN, Edinburgh, in responding, said that a great many people imagined the University to be a place where pure, theoretical, quite unworkable modes of thought existed. Practical men knew that there was not that sharp division between practical and scientific matters which the uneducated imagined. The really wise man was the man who knew what to do in an emergency, and had the strength and presence of mind to do it. Life consisted, whether they were practical or scientific, in a constant succession of emergencies. There was no difference in the way in which they worked; it was sound judgment and common sense applied; and the difference was in the particular subject to which it was applied. They wished everyone to understand that a sensible, well-educated, and intelligent man was what they hoped the University would provide, in whatever kind of teaching it practised. They

wished that truth and reasonableness should prevail, and that scientific and practical men would unite and quench that nonsense which interfered so much with their success in every walk of life. The University had just put its house in order. A good many people felt a good deal of apprehension as to what would come of it. He was not a pessimist, and he never had the least suspicion or fear of what the Commission would do, because there was nothing better in the world than fresh air and light, nor a room or an institution the worse for it. The Commission had been a most painstaking one. He did not suppose that any one of the Commissioners would say that all they had done was right, but they had done a great deal that was right, and they had put the University in a much better position than it was in before.

Mr. R. IRVINE, Edinburgh, proposed "The Guests."

Mr. T. TYRER, in proposing "The Scottish Section," said the Society had been most courteously received by the Section, and that they had received loyal service, honorary service, especially from the local Treasurer and Secretary, who had made the arrangements for the visit, and whose names he begged to couple with the toast.

Professor IYVON MACADAM and Mr. J. STANLEY MUIR, Glasgow, responded, and the proceedings then closed.

### THIRD DAY.—FRIDAY, JULY 20.

#### VISIT TO ST. ANDREWS, RIVER TAY, AND PERTH.

A special train leaving the Waverley Station at 8.55 a.m., conveyed the large party taking advantage of this interesting excursion, and consisting of about 200 ladies and gentlemen, across the Forth Bridge on the Dunfermline route as far as Inverkeithing, then towards Burntisland, passing through the woods of Aberdour. The line, in approaching the coast, has on the left the "Kingdom"—so called because of its importance during the era of the Pictish kings. On the right fine views were obtained of the Firth of Forth and the numerous islands. Aberdour was at one time the port of Dunfermline. The Island of Inchcolm lies off the coast, and contains the ruins of an Augustinian monastery founded by Alexander I. Burntisland, three miles to the east, possesses a sheltered and roomy harbour, overhung by the ancient Castle of Rosend. Further east was Kirkcaldy, the premier manufacturing centre in this part of Scotland. The next place was Dysart, two miles east of Kirkcaldy. The lauds of Ravensraig adjoin the burgh on the west. They include the old Castle of Ravensraig or Ravenshough. This picturesque ruin occupies a conspicuous position on the summit of a lofty rock overhanging the sea. The line now leaves the coast, and turns to the north, *en route* for Thornton Junction, where the line from Dunfermline joins. Proceeding eastwards the coast was again approached at Leven. While on the journey towards Largo, there were observed the three upright red sandstones, popularly known as "The Standing Stones of Lauder." On nearing Largo, a low and sandy shore was approached, which rises towards the north, and culminates in the conical and beautifully green Law. Near Crail, the line turns to the N.W., passing Kingsbarns and Boarhills to St. Andrews. Here the train stopped for one hour and a half.

The party was now shown the various points of interest in this, the most ancient University City of Scotland, under the leadership of Baulie Ritchie Welch. These were:—(1.) The West Port, the last of the old City Gates, probably built about the beginning of the 17th century. (2.) The Madras College and the ruins of the Dominican Friary, rebuilt about A.D. 1520. (3.) The Town Church. Archbishop Sharp's Monument (killed at Magnus Muir A.D. 1679). (4.) St. Mary's College, University Library. (5.) St. Leonard's Church and the remains of College, founded in 1512. (6.) The Cathedral Ruins and St. Regulus Church. (7.) The Castle, Palace of the Bishops. 8. St. Salvator's College and Church, Bishop Kennedy's Monument. He died A.D. 1478.

The train then proceeded, *via* the Tay Bridge, to Dundee, where the saloon steamer "S. Ota" was waiting.

Leaving the pier at Dundee, and proceeding up the river to Newburgh and Perth, the first object which arrested attention was the "Mar-" training ship. This excellent institution supports and educates about 100 boys.

*The Tay Bridge.*—No one can pass through the lofty spans of the new bridge without recalling the terrible disaster which occurred here on the last Sunday of December 1879. The erection of the new bridge was entrusted to Messrs. Arrol & Co., of Glasgow, from plans prepared by Mr. Barlow, engineer, London, and was begun in 1883. The work has been carefully performed under strict Government inspection, and every pier on reaching high-water mark was subjected to a rigorous test before any of the superstructure was placed upon it, these tests having been in the case of every pier most satisfactory. The estimated cost of the bridge was about 700,000*l.*, made up as follows: Piers 5 to 77, are estimated to cost 232,000*l.*; girders and parapet, 265,000*l.*; subaqueous portion of works, 32,000*l.* The diameter of the cylinders for the piers varies from 23 ft. to 17 ft. 6 in. Some of the piers have been sunk to a depth of 35 ft. below the bed of the river. Each pier consists of two cylinders. Those piers under 23 ft. diameter are 26 ft. apart; while the 23 ft. cylinders are 32 ft. apart measuring from centres. The cylinders are carried up independently above high-water mark, and are then joined by a connecting piece consisting of brickwork backed with cement concrete 7 ft. deep, and strengthened with cast-iron bearers and tiers with wrought-iron tie-pieces. Above this is the iron superstructure on which the girders rest. The superstructure is connected to the brickworks by tie-bars. The larger spans are 245 ft. and 227 ft. (that is, in the gap), and 162 ft. and 145 ft. The others are 129 ft., 108 ft., 71 ft., and 68 ft. The cylinders were tested by test-weights varying from 600 to 2,400 tons, and the tests were very satisfactory in all respects. The total length of the bridge is 16,780 ft., while its width at the level of the railway line varies from 25 to 28 ft. The width of the river at the site of the viaduct is 9,580 ft. The height to the boom of the girder at the south side is 65 ft., at centre 77 ft., at north side 16 ft. The number of spans is 85.

On the left was seen Balmerino, where stood the abbey of that name, and where rests the remains of Queen Ermenegarde, wife of William the Lion, the founder of this and the Abbey of Arbroath. Balmerino gives the title to the celebrated lord of that name, who fell a victim to the cause of the Stuarts.

A little to the west a fine view was obtained of Birkhill, the seat of the Wedderburns of that ilk, hereditary Standard Bearers of Scotland. Looking towards the north and west, Castle Huntly and Rossie Priory, together with the villages of Longforgan and Inchtutho, and the Sidlaw Hills were seen. On the left are the Ochil Hills. A little to the west the ruin of Balmabreich Castle was visible, close to the water's edge. Balmabreich was one of the seats of the Leslies of Fife, and is now the property of the Earl of Zetland. Immediately to the south and west of this was fought the battle of Black Inneside or Earnside, where Wallace defeated the English under Sir John Steward. Immediately opposite, on the north bank of the river, on the rising ground, is the village of Errol, to the west of which is the mansion-house of Errol Park, owned by Mr. W. O. Dalgleish, and to the south is Port Allen.

On the Fife side is Parkhill, a modern mansion. About halfway between Parkhill and Newburgh lies Lindores Abbey, the most interesting ruin in the locality. Among the stone coffins found here are two which were said to contain the remains of the children of David, Earl of Huntingdon, brother to William the Lion, and whose death changed the hereditary descent of the Crown of Scotland, and led to the disputes that caused the wars in the days of Wallace and Bruce. This abbey is said to have been built by Earl David on his return from the Holy Land, about the year 1178. On the left is the village of Newburgh, which has long been famous for its fruit. Directly opposite Newburgh, in the middle of the river, is Mugdrum Island,

which was originally a large sandbank formed by the confluence of the Tay and Earn. It was artificially enlarged by the planting of reeds, which retained the mud and sand brought by the tide, and now contains a fertile farm. In the wood, on the south bank of the river, are the mansion houses of Magdram and Carpow. On the north side of the river is the Pifour Brickwork. This company's first contract was for 9,000,000 bricks for the building of the Tay Bridge.

On the left, above Newburgh, Abernethy, with its old Round Tower, was passed. It is beautifully situated at the foot of the Ochils, and is a place of great historical interest, having been at one time a Royal residence, then the see of a bishop and a seat of learning, until these were transferred to St. Andrews. On the right are the villages of Inchyra and Glencarse. On the south bank stands Eleho Castle. A nunnery of the same name also stood a little to the west of the castle. This interesting old castle was the scene of one of Wallace's heroic deeds on his return from France with Thomas de Longueville. On the right, a little further up, is Kinfauns Castle, occupying a fine position on Kinnell Hill, which is the western termination of the Sidlaw range. Its site has been that of a castle for centuries, and is the scene of several incidents described in Scott's works. A short sail round the southernmost portion of Moncrieffe Island brought the visitors to Perth.

Here they were met by Mr. R. D. Pullar, Oehil, and conducted to carriages which were in waiting to convey them to the Commissioners' Hall, where Lord Provost Dewar and the Magistrates took them over the Municipal Buildings, and afterwards entertained them to afternoon tea. The Lord Provost extended a hearty welcome to the visitors, and Mr. E. C. C. Stanford in turn thanked his Lordship, and expressed his delight at visiting the Fair City. Returning to the vehicles, several members of the party drove to the gas works, accompanied by Councillor Wright, who showed the working of Young's oil-gas process, while others had a drive over the length of Bridgend, and afterwards visited the Fair Maid's House, and the East Church with its organ with electro-pneumatic action. Shortly after five o'clock the whole company repaired to the City Hall, and were entertained to a banquet by Messrs. J. Pullar and Sons. The tables were artistically laid out; the front of the gallery was draped with pale blue art muslin, while the platform was covered with a fine collection of plants from Tayside and Rosebank gardens. After the seats running from east to west in the hall had been filled, the following guests, preceded by the Town's officers, took up their position at the top table—Mr. Robert Pullar of Tayside in the chair, and on his right Lord Provost Dewar, Professor Crum Brown, Dr. J. Murray, Lord Dean of Guild Wotherspoon, Dr. Charles Dreyfus, Mr. W. Sharp, Mr. S. K. Muspratt, Mr. A. Coates, of Pitculen, Prof. W. Ivison Macadam, Edinburgh, Mr. J. Stanley Muir, Glasgow; on his left, Mr. E. C. C. Stanford (President), Sheriff Grahame, Mr. R. Irvine, Mr. Wm. MacLachlan, City Clerk, Mr. B. E. R. Newlands, Bailie McNicoll, Mr. J. L. Wade, and Mr. T. Tyrer. Amongst the others present invited to meet the visitors were ex-Lord Provost Whittet, ex-Lord Provost Martin, Bailie Kyd, Councillor Wright, Mr. John Moncrieff, Mr. Robert Keay, city chamberlain, Mr. Thomas Whimster, gas manager. The gentlemen who acted as croupiers were—Mr. R. D. Pullar, Mr. A. E. Pullar, Mr. H. S. Pullar, Rev. A. J. B. Baxter, Mr. John Thomas, sheriff clerk, Mr. W. H. Gregor, and Mr. R. D. Wright, Pullars' Dyeworks. After the dinner the Chairman intimated apologies for absence from Mr. Jas. F. Pullar, of Rosebank Rector Chambers, Mr. John Alexander, Sharp's Institution, Mr. Melville Jameson, procurator-fiscal, Mr. J. D. Lumsden, Huntingtower, Mr. R. W. R. MacKenzie, of Stormontfield and Earlshall, Mr. John Hally, Auchterarder, Mr. R. McKillop, burgh surveyor, Mr. J. Burt-Marshall, Lunarcy, Mr. Jas. Shepherd, Kirkealdy, Bailie Cuthbert, ex-Provost Wilson, Mr. D. M. Watson, of Ballinfield, and Dr. Thomson, Perth Academy. During the progress of the dinner Mr. Edward Nicoll, organist North U.P. Church, played several selections on the grand organ.

The CHAIRMAN gave "The Queen." They could not, he said, say too much in praise of her wonderful reign, and they hoped she would be spared for many years longer to reign over a happy and contented people. They in Scotland were very much favoured by Her Majesty, and he thought they in Perth were particularly favoured, because she passed through the city very frequently on her way to her northern home, and without intruding on her privacy they had an opportunity of seeing her. He would couple the toast with the "Other Members of the Royal Family," and they must not forget the latest addition. If it should come about in the course of things that the little Prince should one day rule over Great Britain, he hoped it would be even greater than it was at present. The National Anthem was then sung.

The CHAIRMAN then proposed "The Ladies." He thought it was an excellent arrangement to have the ladies with them, and he hoped it would be carried out in future. They had ladies with them that day from England, Ireland, Scotland, the United States, and Canada.

Dr. MURRAY, replying on behalf of the ladies, quoted from a French review, with which he agreed, and in which it was stated that in a very short time ladies were to take a large part in scientific research. They could not make research pay so far as money was concerned, but they could make it pay with respect to the higher life, and to a service and duty rendered both to self and country. He thought it was certainly the case that women had a much more acute intellect than men, and knew better how to save the money.

Mr. ROBERT PULLAR in proposing the toast of the "Society of Chemical Industry," said his firm felt very highly honoured indeed that so many gentlemen connected with this industry, both in Great Britain and abroad, had favoured them with their presence there that day. He was sure no more distinguished company had ever been welcomed in Perth—men who were distinguished in many positions as professors, and as lecturers, and as investigators, and as those that were in charge of very large and very important chemical industries. He dated say a good many were rather puzzled as to what chemical industry was. He then quoted from the Journal of the Society, which had now appeared for 13 years, 23 different branches of chemical industry which the members all studied from time to time. It was just by studying such things that the greatness of their country was maintained, and it was by doing these things rightly that they would be able to keep their position. This Society was a very valuable factor in the various chemical manufactures of the country; and he was sure every year it was in existence it would make progress. They had at the head of it at the present moment, a President who knew the subject, and knew it very thoroughly, as was well exemplified by his address on Wednesday at the meeting in Edinburgh. And had there been time one could have enlarged still more upon that; but he could now only say that they, as a firm, welcomed most heartily the members. He had been told that there was one lady member in the Society. He hoped if ever the Society should come again to Scotland, they would pay another visit to Perth, and perhaps have a little more time at their disposal. Referring to the welcome given by the Lord Provost and magistrates, he felt that as a member of the Society they were highly honoured in being so courteously received by the dignitaries of the city. Mr. Pullar concluded by coupling the toast with the name of the President.

Mr. STANFORD said, he ought now to touch on the whole of these 23 subjects mentioned by Mr. Pullar, and probably a good many more. Their President had been already appointed for next year, but he had no doubt that in the following year the lady member spoken of by Mr. Pullar would be elected President. He would be delighted to see that himself, because then they would listen to real eloquence, which some of them were not accustomed to. He thought they were extremely obliged to their Chairman for having discovered the means of bringing their Society so far north. They were, in fact, at the first stopping place in Her Majesty's route to health. He strongly advised them to follow her further north. Mr. Pullar had

discovered that this Society—and this was the attraction—though it might have a very weak head, had a strong appetite. They remembered that Sydney Smith in his libel on the Scottish nation, said it required a surgical instrument to get a joke well into the Scotch understanding. He explained that afterwards by saying that the instrument he meant was a cork's raw. Mr. Pullar had also discovered that that was an instrument too, that was quite able to draw out the English nation. But in the very short time that he had that night he would say nothing more about the Society of Chemical Industry. He wanted to drink the health of their entertainers. They were accepting the hospitality of Messrs. Pullar and Sons, Perth, and with the organ they saw there to almost everything about the city the name of Pullar was associated. Their friend had not only done so much for Perth, but he had done a great deal for the Society of Chemical Industry, and he had never done so much for it as that day. He (Mr. Stanford) only wished they could have seen his works. The Messrs. Pullar employed about 2,000 men and women, and he was told that sometimes no fewer than 15,000 parcels a week were sent out of the establishment. He could not imagine for a moment how that was done. They had not time for Mr. Pullar to explain it, but he did think Mr. Pullar must be, and he knew he was, the greatest authority on parcels in the world. He knew Mr. Pullar was consulted by the Post Office when they began to try—and they failed miserably at first—to deal with the parcels of this kingdom. He believed at one time Perth was a great seat of the glove trade. That had now dropped altogether, he believed. No one had explained it, but it seemed to him perfectly obvious that no one wanted any new gloves now; they all came up to Perth to be cleaned. He knew there were numbers of ladies in London, noble ladies, who had the greatest difficulty with that awful atmosphere—he knew there were cockneys present, and he wanted to give it them—which spoilt their beautiful gowns and made them all black. What was a lady to do if she did not know there was a noble man up in the north who could pull (h)ar out of the difficulty. There was just one other point on which they ought to think of the very beneficent character of this great work, because there was a large class of their fellow-citizens, and especially their countrywomen, that were obliged to keep up a decent appearance with a very small income. He did not think it was possible to estimate the amount of good which was done to this nation in that particular way by the firm of Pullar and Sons, of Perth. Mr. Stanford hoped that, along with his excellent lady, Mr. Pullar would be long spared to enjoy good health.

Mr. PULLAR said he expected when a man had a toast to propose that he would stick to his text. His friend had departed from his text, however, and entered upon a difficult and complicated subject, which certainly they had not time to discuss that evening. If any of the ladies should happen to have a day to spare he would do his best to meet and show them all the interesting features of his works. He felt that it was a very high honour, and he accepted it on behalf of his firm, and they looked not upon themselves alone, as members of it, but also the very able and excellent men who were working along with them. He had now great pleasure in calling upon Professor Crum Brown to propose a toast.

Professor CRUM BROWN said the toast was one which it was impossible to do justice to in the short time he had at his disposal. At all events they must allow him to say three things. First, as to the fair city of Perth. Those who had seen Perth that day for the first time would, he was sure, admit it was a fair city. Although they entered it in a shower of rain, that only increased the beauty of it, for the dull sky set off to admirable advantage the superb environments of hill and wood that surrounded it. The beauty of Perth had been famed from a very early time; for they read when the Romans advanced towards it through a part of the county over which they had yet to march, they broke into exclamations of delight at the first glimpse of it: beauty. So much for the city; he had no time to dilate further upon it. Then

he came to the trade of Perth. They all hoped the trade of Perth would always flourish. He had next to speak of the government of Perth, coupled with the toast the name of Lord Provost Dewar. That such a city might remain fair and beautiful and healthy and prosperous depended to a very considerable extent upon those who governed it. And when they found it prosperous and beautiful, they might be sure it was a well-governed and well-managed city; and he believed that that was the case here. Lord Provost Dewar was one of the most enterprising and most zealous chief magistrates in the country. He was, he believed, the youngest chief magistrate in this country—the youngest man that ever occupied the position of Lord Provost. It was a great advantage that an old city should have a young Lord Provost. The rejuvenescence of the world was one of the great points. They had a good specimen of a gentleman beginning young, and he hoped Lord Provost Dewar had a long and prosperous reign before him. He had extreme pleasure in submitting for their acceptance the toast of "The City and Trade of Perth and the Lord Provost of Perth."

Lord Provost DEWAR returned thank for the very hearty reception they had given the toast. They were all very much pleased and honoured that the Society had paid a visit to Perth, and they welcomed them very heartily on account of the distinguished position that Society occupied, on account of the personal character of all the members, and also on account of their being the guests of their respected citizen, Mr. Pullar. In regard to the city of Perth, he did not think too much could be said of it. It had that peace and quiet and contentment about it, which, as a business man, he did not very much like. He had not the same difficulty in speaking to this part of the toast as he had about the trade of Perth. He was sometimes embarrassed in speaking of the city trade in presence of representatives from other communities that had advanced by leaps and bounds—which they in Perth undoubtedly had not done. He had sometimes to apologise in replying to this toast for the disappearance of the glove-making industry; and he was obliged to their President for suggesting a reason which had not occurred to him before. He was sorry to hear from Dr. Murray that his end of chemical science did not pay; but he could assure them the other end paid very well; and the industry of Perth in that respect was as flourishing as it possibly could be. They were all very much interested in local government; they did what they could to live up to the situation Providence had placed them in—he meant the situation of the town. All did their best to keep it as it was long ago, still the Fair City.

The CHAIRMAN said they had an excellent man at the head of the School Board, and he had very great pleasure in proposing the health of the Rev. Mr. Fleming, chairman of the School Board.

Rev. Mr. FLEMING thanked Mr. Pullar very much for the kind way in which he had proposed the toast of the School Board of Perth, coupled with his name. He might say in the presence of so influential and important a Society as that which they had the honour of seeing there, that they felt the claim which technical science had upon them; and it was their determination to keep up not only all the existing agencies, but, if possible, to extend and render them still more efficient. He had also to state that it was the desire of the present school board to do all that they could in the interests of the science of chemistry. They felt that this science had a very great future before it, and after what he had learned that day, he felt that the researches of the eminent men in connection with this science in future were destined not only to revolutionise the chemical industries of the country, but also the usual associations of the dinner table. He was sure that they all took an interest in everything connected with this science. The school board took an interest in it because they felt that chemistry played a most important part in the industries of this city, more especially of that industry of which their Chairman was the distinguished head. They felt it was their duty to take an interest in it from the circumstance that Perth was a large agricultural centre, and chemistry, he thought, was

very much bound up with the progress of agriculture. He had pleasure in stating the school board had given great importance to the study of chemistry in their educational institutions. They had attached great importance to it in their curriculum in the Academy, which, he might state, was now, as it had been for centuries, the leading educational institution in the city and county. He might also state that great importance was attached to that branch of study in Sharp's Institution, with which his friend, Mr. Pullar, was very intimately associated. They had at the head of the mathematical department of the Academy, Dr. Thomson, a gentleman who was quite an expert in everything connected with chemistry, and he had sent from his school some most distinguished students, and he had no doubt they would soon be in the classes which were so ably presided over by Professor Crum Brown. In conclusion, Mr. Fleming said the visit of the Society to Perth would give an impetus to the study of all the pursuits connected with chemistry.

The CHAIRMAN announced that they had reached the end of the programme, and gave "Sorry to part," &c. "Auld Lang Syne" having been sung, accompanied on the grand organ by Mr. Nicoll, the large party left the hall, and were driven to the station, where a special train was in waiting at 7.30 to take them direct to Edinburgh.

#### THE SMOKING CONCERT.

In the Reception Room of the Waterloo Hotel some 60 or 70 members of the Society attended an enjoyable Smoking Concert, which, commencing at 9.30 p.m., was continued into the small hours of the following morning. An attractive programme of vocal and instrumental music, &c. had been provided, and was duly honoured in the performance; but there were super-added some songs, recitations, and humorous speeches by other members of the company, in which, amongst others, Professor Crum Brown, Mr. B. E. R. Newlands, and the ex-President took part. It will be some time before those who heard it will forget the humorous version of the "Babes in the Wood," so minutely given by Mr. E. C. C. Stanford.

#### THE POWDERHALL REFUSE DESTRUCTOR, EDINBURGH.

Several members took advantage of the opportunity afforded to see the City Destructor, and were shown over the works by Mr. Mackay, the Inspector of Cleaning and Lighting for the City, on Saturday morning, July 21st.

## Nottingham Section.

### UNIVERSITY COLLEGE, NOTTINGHAM.

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Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

Meeting held at University College, Nottingham,  
on Wednesday, May 23rd.

MR. J. M. C. PATON IN THE CHAIR.

### DISCUSSION ON THE PAPER OF MESSRS. SCHWEITZER AND LUNGWITZ ON THE COMMERCIAL ANALYSIS OF LARD.

(This Journal, 1894, 614—616.)

Mr. RIMMINGTON (Borough Analyst, Bradford) said that he found the results of authors' experiments so various and different, that he had come to hear what other people had to say on the subject. Referring to Mr. Stock's experiments, he had taken a sample of what he thoroughly believed to be pure lard as he obtained it from his brother. The melting point was 98° F.; the iodine absorption was 49; and the amount of ether-washed deposit from 3 cc. was 118 mgrms. The same with 5 per cent. of beef-suet showed that the amount of crystallisation was 187. In lard of his own rendering, the melting point was 96° F.; iodine absorption 52; and the amount of crystals 80. In a commercial lard the melting point was 87° F.; the iodine absorption was 60½; and the ether-washed deposit only 27 mgrms. He had not made any attempt at such elaborate analyses as the authors of the paper evidently had done.

Mr. A. H. ALLEN said he congratulated the Section upon having before it a paper by two American members of the Society. It showed that the Society was not simply of a local or even of a national character, but that its ramifications extended all over the world. The authors of the paper appeared to have dealt with the subject very completely, and he did not doubt that their results would be of permanent value. According to the book-writers, animal fats all had a closely analogous chemical composition, varying only in the relative proportions of olein, palmitin, and stearin they contained. But if this were true, it was difficult to account for the very different appearance presented under the microscope by pure lard, beef-fat, and other lard substitutes. He observed that the authors practically adhered to the Manfield method of preparing the lard crystals for microscopic examination; in his own experience that plan still held its own. The use of cotton-seed oil for mounting the crystals was no doubt original on their part, but it had already been used in England. The authors laid great stress on the crystallising point of the lard, as an indication of purity, which, in England, analysts would scarcely be inclined to do. When the sample was proved pure by other means, the crystallising point might afford a useful indication of its origin, but in practice, analysts had to report on lards of which they knew nothing, and the crystallising point varied very greatly according as the article was real leaf-lard (prepared from abdominal fat), back lard, or the hog-fat prepared so largely in Chicago and other American cities, by rendering the fat of the whole animal. This product was of a very different character from the old-fashioned lard of English manufacture, and he thought this should be borne in mind in interpreting the authors' results, which were probably applicable solely to the American product. The method used by the authors in determining the crystallising point differed from Dalican's test for the solidifying point of fatty acids, in the fact that he directed the substance to be stirred while cooling, whereas the authors avoided agitation. The subsequent rise of temperature observed when oleo-stearin was under examination, and the absence of the phenomenon in the case of cotton-seed oil, was of interest and might become of practical value.

With regard to the rise of temperature on treatment with sulphuric acid, there was no doubt that the strength of the acid employed was of the first importance. That is to say, that the temperature developed was largely affected by the exact strength of the acid used. But he had grave doubts of the practical advantage of making up an acid containing 100 per cent. of H<sub>2</sub>SO<sub>4</sub> by mixing fuming sulphuric acid with "acid of 66° Beaumé." He believed that equally good results were obtainable with acid of 98° per cent., which

was an article obtainable in commerce; but in any case the strength employed must be rigidly adhered to throughout a series of comparative experiments, or the figures obtained were not worth the trouble of recording. But he objected to the authors describing ordinary strong sulphuric acid as "acid of 66° Beaumé." It had been shown repeatedly that the density was valueless as an indication of even the approximate strength of concentrated sulphuric acid, but when the density was expressed in degrees Beaumé, and not in actual specific gravity, there was no certainty to 10 per cent. as to the actual strength of the acid used. It was a matter of astonishment that American chemists, who were ahead of those in the old country in so many respects, should continue to use the clumsy hydrometer of Beaumé, instead of expressing their densities in actual specific gravities, or in terms having some less arbitrary relation thereto than was borne by the degrees of Beaumé's scale. It was bad enough if one had to deal with accurately graduated instruments, but it was notorious that the Beaumé instruments made by different manufacturers differed very materially in their indications, and there was not one in ten of the makers who could tell on what principle he graduated them, except that they were tested against some instrument kept as a standard, and usually of unknown history. One was prepared to find the French continuing to record their results in degrees Beaumé, but that American chemists should do so was extraordinary.

As to the iodine absorption, he observed that certain experiments showed a variation from 57 to 85. Assuming that there was no clerical error here, he should regard these limits as extremely doubtful, and should require to know the precise conditions of operating before he accepted them as accurate. He congratulated the authors of the paper on their attempt to shorten the time of treatment in Hübl's process, but looked somewhat shyly at any method the accuracy of which depended on a precise adherence to a particular time and temperature. We had too many of such arbitrary processes already, and he should prefer to allow a longer time if by so doing he could ensure the completion of the reaction. It was only in this manner one could hope to avoid the errors due to personal equation, and could render the results obtainable in different laboratories fairly comparable.

With respect to the tests for cotton-seed oil based on its reducing action on silver nitrate, gold chloride, or phosphomolybdic acid, it was a remarkable fact that this reducing action was not more generally exercised by vegetable oils, supposed to be of a very similar composition to cotton-seed oil. Yet in one of the earlier forms of the silver-test the reagent was actually made up with rape oil. There were so many substances which exerted a reducing action on metallic solutions that he should think a long while before he based a positive opinion as to the presence of cotton-seed oil on the results of such a test. In short, the reduction tests should be regarded rather as useful indications than as absolute proof, and ought in every case to be supplemented by other methods of examination.

Prof. F. CLOWES said that analysts who had used the Mauméné process, considered it not wholly reliable; that although this test was useful when applied to many oils that did not occur as adulterants in lard, it had not been found correct when applied to oils or fats used in the adulteration of lards. The least differences as to detail in the method of working would be sufficient to cause a very great difference which was not in any way due to the fats or oils. Whether the analysts had paid attention to every detail in conducting the test, he could not of course say; but, presumably, they worked with ordinary care, and it was the test that was at fault. If a test could play such pranks as these, they must not place their reliance too firmly upon it. That was the extent of his information. He regretted it was not founded upon personal experience.

Dr. TRUMAN said that the great difficulty was to obtain quantitative determinations of adulteration of lard with cotton-seed oil, &c. The specific gravity, if normal, was no proof of purity, as mixtures might be made giving normal specific gravity. The iodine number was open to the

same objection. But if the normal number was exceeded, then one could estimate amounts in two ways. Taking the iodine number, e.g., 90, then the percentage of cotton-seed oil in the sample would be shown by the formula  $100 \left( \frac{90 - 60}{105 - 60} \right)$  (lard taken at 60 and cotton-seed oil at 105). Taking specific gravity of lard and cotton seed oil at 100° F., as 0.906 and 0.9135 respectively, then the percentage of cotton-seed oil was found by the equation,  $100 \left( \frac{\text{specific gravity found} - 0.906}{0.9135 - 0.906} \right)$ .

The qualitative tests were plain and abundant enough. Personally he preferred the  $\text{AgNO}_3$  reduction test to the phosphomolybdic acid one; it was more certain. The sulphur chloride test was useless. Colour reactions with  $\text{HNO}_3$  and  $\text{H}_2\text{SO}_4$ , rise of temperature with  $\text{H}_2\text{SO}_4$ , solidification point and microscopical examination, all of them he found most useful.

He agreed as to the value of the six tests mentioned on page 615, but with the exception of the first, he had made use of them for years, and other analysts might have used the phosphomolybdic test. The authors state that less than 10 per cent. may escape detection by this test. The heating of the solution for the iodine number determination was an improvement, as it saved so much time.

Allen (Com. Org. Analysis) II., 56) describes the rise of temperature test fully. It was a pity that no figures are given by the authors of the paper; and that Allen gives no figures for lard.

Messrs. H. SCHWEITZER and E. LUNGWITZ (New York) send the following reply:—

A few points in our article evidently need further explanation. We repeat that we do not consider any one of the six tests, neither the crystallising point nor the test with nitrate of silver, as characteristic enough to determine the purity of lard. Our contention is that all of them must be made and that the final conclusion must be drawn from the results of all. After an exhaustive examination of all the tests hitherto known in literature, these six tests were selected, because they give, in our opinion, the quickest and most reliable final result.

It is true, and we find it daily confirmed that the crystallising point is of the greatest importance as an indication of the purity of lard. However, we state with regret that the presence of cotton-seed oil does not in all cases prevent the rise of temperature. According to our experience the temperature does not rise when compounded lards, prepared by mixing about 75 parts of cotton-seed oil with 25 parts of oleostearin, are examined, or when such mixtures are added in large quantities to genuine hog fat. If, however, only very small amounts of cotton-seed oil have been added to pure lard in conjunction with little oleostearin, then the temperature will rise. But the observations made while carrying out the test will furnish valuable material to the analyst. The temperature will sink very slowly, and will probably not go below 29° C., while the subsequent rise will most likely exceed 1° C. All these differences are best found out by comparative examination of lard of known origin with mixtures of known composition. To facilitate such comparative tests we have prepared about 100 mixtures showing the consistency of commercial lard but compounded in varying proportions from lard, lard-oil, cotton-seed oil, and oleostearin. By referring to these standard samples we possess a valuable guide whenever we meet difficulties in our analytical examinations.

With regard to the variation of the iodine number observed by different authors, we quote from Wiley's publication: "For instance, a sample of intestinal lard absorbed 57.34 per cent. of iodine, the leaf-lard from the same animal absorbed 52.55 per cent., the foot-lard 77.28 per cent., the head-lard 85.03 per cent."

We add that Wiley determined the iodine number after the old Hübl method. Quite recently† Samuelson pronounced the iodine number to be the only known reliable

\* Bulletin, 13, 417, 118.

† Z. its. f. anal. Chem., 33, 1-2; and this number, p. 766.



test for the purity of lard. We are obliged to differ very much from him. Since we examine weekly a sample of compounded lard containing 70 per cent. of genuine hog-fat and 30 per cent. of a mixture of cotton-seed oil and oleostearin which shows the correct iodine number of 60–61 after the Hubl method. The same lard also gives the Maumené figure for pure lard, whereas the results of the other four tests distinctly show adulteration. As to the reduction-test with nitrate of silver, we are of the opinion that reliable results can only be obtained if the directions of E. Millian are strictly adhered to. We attribute great importance to this test, and we prefer it decidedly to the reaction with phospho molybdic acid.

Finally, we say, with respect to Maumené's test, that we direct the use of an acid of 100 per cent.  $\text{H}_2\text{SO}_4$  because we find that the rate of action between fat and sulphuric acid increases with the strength of the latter. We do not doubt that acid of 98 per cent.  $\text{H}_2\text{SO}_4$  would also give good comparative results. But what we want to point out most distinctly is that the acid must be always of the same strength, and must be constantly controlled by analysis.

By using the term "66 Bé" we meant any commercial acid which by the addition of fuming acid should be transformed into acid of 100 per cent.

We did not give any analytical results for the Maumené test, as, owing to the personal character of the results, it is obviously impossible to lay down precise figures. Every analyst ought to find his own constants for his apparatus.

The following figures were found:—

---	Initial Tempera- ture of Fat.	Tempera- ture of Acid.	Final Tempera- ture.	Rise.
	° C.	° C.	° C.	° C.
Lard.....	35	35	70.0	35.0
" oil.....	35	35	80.3	45.3
Oleostearin.....	45	45	76.9	31.9
Tallow.....	45	45	76.6	31.6
Cotton-seed oil.....	35	35	90.5	61.5
Armour's compounded lard.	35	35	93.0	58.0

Generally we found 33.5–36° C. as rise of temperature for pure lard, and 61.5–63° C. for pure cotton seed oil; whereas Wiley found as extreme rise 38.8–42.1 for pure lard, and 80.4–90.2 for cotton-seed oil.

We cite the following absurd figures found for the above lard under the above conditions with an acid sold in New York as "chemically pure compound, 66 Bé," which on analysis was found to contain 92 per cent.  $\text{H}_2\text{SO}_4$ .

---	Initial Tempera- ture of Fat.	Tempera- ture of Acid.	Final Tempera- ture.	Rise.
	° C.	° C.	° C.	° C.
Lard.....	35	35	55.8	18.8
" .....	35	35	71.1	36.1

## Communication.

### CENTRIFUGAL METHODS OF MILK ANALYSIS.

BY ANDREW SCOTT.

WITHIN the last few years an entirely new principle has been applied to chemical analysis—that of centrifugal force; and it was with the object of ascertaining how far centrifugal methods could be relied on for milk analysis, that the experiments of this paper were undertaken.

The principal machines in use are the Löffmann Beam and the Lister Babcock Testers. In the former we have a metal arm fitted on a spindle, and to each end are riveted brass cups to hold the test-bottles. At the lower extremity of the spindle we have a toothed pin working in a larger wheel which acts as the driving wheel—each revolution of the latter being equivalent to eleven of the horizontal arm. According to the directions, 15 cc. of the milk to be tested are put into the bottle, 3 cc. of a mixture consisting of equal parts of fusel oil and strong hydrochloric acid are added mixed, and the strong sulphuric acid (9 cc.) poured in slowly, with agitation, until the bottle is filled nearly to the neck.

The liquid becomes hot, and the casein is completely dissolved, a dark reddish-brown solution being formed. The neck is filled to near the zero point with a hot mixture of sulphuric acid and water, and the bottles whirled in the centrifugal machine for from one to two minutes. Milk very poor in fat may require from three to four minutes. The volume of fat which rises in the neck can be read off directly.

The results by this machine are slightly on the high side, and much has been written on the subject of the fatty layer, which is slightly darker than that obtained by the Lister Babcock machine, to which I shall next refer.

I made a blank experiment with the bottles, using the usual proportions of acid and fusel oil, but with water instead of milk. After whirling for the usual time, I found a dark layer had separated and risen to the surface, representing 0.10 to 0.15 in the graduation. Then a sample of milk-fat drawn from one of the bottles and tested with silver nitrate solution gave a decided precipitate of silver chloride, so that the layer probably contains both hydrochloric acid and amyl alcohol, or some compound produced by amyl alcohol.

Various experimenters make the difference in fat between this process and Adams' coil 0.004 to 0.2 per cent. I have done a number of tests, and find that between duplicates the greatest difference is 0.15 per cent., while the greatest discrepancy between it and Adams' coil method is 0.13.

The following representative tests, taken from my notebook, show the results alongside those obtained by Adams' process. (A number of the samples were also tested by Dr. Bell's process, which gave results slightly lower than those obtained by Adams' method.)

Löffmann Beam.	Adams.
3.30	3.32
2.15	2.02
2.42	2.47
3.27	3.23
2.00	2.10
1.30	1.41
2.50	2.47
1.30	1.35

The next machine to which I have referred is what is known as the Lister Babcock. This method was introduced

by Dr. Babcock, of the United States, while the latest modification is produced by Messrs. R. Lister and Co., Limited, of Dursley. In this we have a driving wheel as before, inside which works a toothed pin; by means of a pair of bevel wheels below, the direction of motion is changed and rotates the cast iron flange, the centre of gravity being thus brought lower down than in the other machine. In the iron flange we have slots cut at intervals, in which rest the steel cages holding the bottles. These latter are graduated in a similar fashion to those of the other machine.

17.6 cc. of the milk to be tested is pipetted into the bottle held in an inclined position, then 17.5 cc. of sulphuric acid (sp. gr. 1.831 to 1.834, determined by the hydrometer supplied with the instrument). The milk and acid are well mixed and the bottles whirled in the machine for 10 minutes. After this time they are filled to the centre of the stem with boiling water, and the tank which surrounds the whole, filled to the bottom of the test-bottles. The machine is rotated for two minutes and the fat measured. The speed during the test is 650 revolutions per minute; that is, 65 turns of the driving wheel.

The fat-results are low by this machine, but by a constant figure, and it is necessary to add 0.3 to the measured fat to obtain the correct percentage. I have had occasion to carry out a large number of tests with this machine, and have never got a greater difference than 0.1 between duplicates, while the variation above or below Adams' coil is only 0.11 in the greatest.

The following tests, taken from my note-book, include the greatest and least differences:—

Lister Babcock.	Adams.	Error.	Lister Babcock.	Adams.	Error.
3.40	3.42	- .02	2.40	2.33	+ .07
3.80	3.77	+ .03	2.10	2.10	..
3.20	3.23	- .03	2.10	2.02	+ .08
3.30	3.32	- .02	2.00	2.00	..
3.20	3.34	- .04	1.55	1.59	- .04
2.80	2.91	- .11	1.45	1.52	- .07
2.70	2.78	- .08	1.40	1.38	+ .02
2.50	2.57	- .07	1.35	1.41	- .06
2.50	2.58	- .08	1.39	1.35	- .05
2.50	2.47	+ .03			

(In every case 0.3 has been added to the actual reading of the Babcock machine.)

The low readings in this machine are said by some to be due entirely to the graduation of the bottles; by others, to a proportion of fat remaining in the acid liquid and consequently being lost to measurement. Probably both are responsible, but certainly small globules of fat may often be observed in the acid liquid; and on submitting a sample of milk to centrifugal force for 15 minutes, or five minutes longer than the directions advise, a further quantity of fat rose to the surface representing 0.1 and 0.15 per cent. of the total fat in the sample; again, on working the machine for the usual time, but at double the speed, I succeeded in obtaining 0.15 more fat in the milk. This latter course, however, I would not recommend, as the bottles are apt to break when driven at such a high speed. I find that as a rule 0.3 added to the reading gives the correct percentage of fat; in some cases of milks between 1 and 2 per cent. fat, 0.35 seems a better figure, but on studying the table it is evident that 0.4 is too high.

The advantages of centrifugal analysis for milk are obvious—(1) reliability, I have shown; (2) accuracy of results is also clear; (3) the saving of time is patent. All we have to do is to take the specific gravity of the sample by means of the bottle or a reliable hydrometer, to whirl the measured quantity in the machine, and by Fleischman's or Richmond's formula we get at a glance the total solids.

The ash should also be done. (4) Seeing, however, I would not advocate the use of the centrifugal machine for prosecution or suspected samples, we have this further advantage—that fresh determinations by Adams' or Bell's method can be proceeded with while the milk is sweet.

On the whole the Lister Babcock machine is to be preferred. It is a little cheaper; I have found it give quite as trustworthy results, while it is much simpler. The whole apparatus consists of the machine and bottles, a milk pipette, a measure for the acid, and a hydrometer to test the same. Let me notice here that it is necessary to use the acid of the fixed strength recommended, as stronger acid not only darkens the fat, but causes bad separations, particles of charred curd rising to the surface, and making the reading very difficult.

Nothing but sulphuric acid being used in this machine, the fatty layer consists of nothing but butter-fat; in the Leffmann Beam, on the other hand, we have the use of a volatile acid, hydrochloric, and also amyl alcohol, which is by no means pleasant to work with, while we cannot be sure that pure fat only separates and rises in the stem.

Though it is necessary to test the acid for the Lister Babcock machine, Richmond has shown that it is not only necessary to test the acid for the Leffmann Beam, but variations in samples of amyl alcohol render it necessary to test each lot of this too.

It has sometimes been found difficult to read the fat layer in the Leffmann Beam tester, especially in cold weather, owing to congelation, but that is obviated in the Babcock machine by the hot-water tank, though I have not found the hollow lid filled with hot water, as adopted in a later form of machine, necessary.

The centre of gravity in the Leffmann Beam being so high, causes great oscillation, which is especially objectionable in a chemical laboratory, besides which we have at least a possible danger of one of the cups flying off when driven at such a great speed. One chemist indeed thinks it safer to erect an iron screen round his machine.

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## Journal and Patent\* Literature.

Class.	Page
I.—General Plant, Apparatus, and Machinery .....	713
II.—Fuel, Gas, and Light .....	715
III.—Destructive Distillation, Tar Products, &c. ....	719
IV.—Colouring Matters and Dyes .....	720
V.—Textiles: Cotton, Wool, Silk, &c. ....	725
VI.—Dyeing, Calico Printing, Paper Staining, and Bleaching .....	726
VII.—Acids, Alkalis, and Salts .....	729
VIII.—Glass, Pottery, and Enamels .....	735
IX.—Building Materials, Clays, Mortars and Cements..	736
X.—Metallurgy .....	737
XI.—Electro-Chemistry and Electro-Metallurgy .....	741
XII.—Fats, Oils, and Soap Manufacture .....	744
XIII.—Pigments and Paints; Resins, Varnishes, &c.; India-Rubber, &c. ....	746
XIV.—Tanning, Leather, Glue, and Size .....	746
XV.—Manures, &c. ....	748
XVI.—Sugar, Starch, Gum, &c. ....	748
XVII.—Brewing, Wines, Spirits, &c. ....	750
XVIII.—Chemistry of Foods; Sanitary Chemistry and Water Purification; Disinfectants .....	752
XIX.—Paper, Pasteboard, &c. ....	754
XX.—Fine Chemicals, Alkaloids, Essences, and Extracts	755
XXI.—Photographic Materials and Processes .....	759
XXII.—Explosives, Matches, &c. ....	760
XXIII.—Analytical Chemistry .....	760

\* Any of these specifications may be obtained by post by remitting *sd.* the price now fixed for all specifications, postage included—to Sir Henry Reader Luck, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

## I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

*Action of Aluminium on Cast Iron.* Bull. Soc. Chim.  
11—12, 1894, 374—379.

See under X., page 737.

*Execution of Masonry and Brickwork during Frost.*  
Alfred Greil. Zeits. des österr. Ing. und Arch. Ver. 1894,  
95. Proc. Inst. Civil Eng. 116, 1894, 9—11.

THE information embodied in this paper is the result of the investigations undertaken by a committee of the Austrian Union of Engineers and Architects to determine the most reliable method of proceeding with masonry or brick construction during severe frost.

The first portion of the report summarises the previous records and experiments on the subject, and the experience of experts in Russia, Sweden, Norway, and elsewhere. The consensus of opinion is in favour of Portland cement, with a certain quantity of common salt in the water, and the brick or stone used dry.

The general results indicate that lime-mortar is useless for either brick or stone. Roman cement gives fair results for brickwork, and Portland cement good results; both are improved by mixing with warm water, and distinctly improved by the addition of salt. The "frostproof" Roman and Portland cement give excellent results. For masonry, Roman cement is not very satisfactory, though improved with salt; Portland cement gives good results, though not improved by warm water, but very good with the addition of salt.

It appears in brief that reliable results are to be looked for only with Portland cement used with cold water and the addition of 7 per cent. of the cement and water in common salt; this applies both to brickwork and to masonry.

### PATENTS.

*Improvements in Apparatus for Condensing Vapours and Heating or Vaporising Liquids respectively.* W. Henneberg, Hamburg. Eng. Pat. 5262, March 13, 1893.

THE apparatus consists of a number of pipes or channels, through which the liquid or vapour is passed, and surrounded by a vessel containing a liquid such as water, which latter is caused to circulate through the vessel and so heat or cool by conduction the liquid or vapour contained in the pipes. To promote the efficiency of the apparatus, ribs and pockets are formed in the side walls of the channels to agitate the contents and thereby bring new particles under the action of the heating or cooling medium.—E. G. C.

*Improved Still and Apparatus for the Distillation of Ammoniacal Liquid and other Fluids.* G. Pettigrew, Middlesbrough. Eng. Pat. 10,171, May 23, 1893.

See under VII., page 734.

*Improvements in Apparatus for Evaporating Liquids by means of Steam.* E. Latham and E. G. Guyot, both of London. Eng. Pat. 10,467, May 27, 1893.

A NUMBER of pairs of concentric steam tubes are arranged in the evaporating vessel, the inner tubes being open at both ends and completely surrounded by the outer tubes, which are closed at the top and open to a steam exit chamber below. Steam is allowed to circulate through the inner tubes and in the annular space between the inner and outer tubes, thereby heating the water contained in the evaporator. The evaporated vapour is collected by a

perforated ring-pipe and led away to a condensing arrangement, while the sediment left in the vessel is discharged by a suitable cock.—E. G. C.

*Improvements in Thermostatic Instruments.* A. G. Brookes, From G. W. Gregory, Boston, U.S.A. Eng. Pat. 11,052, June 6, 1893.

THE apparatus is intended for use in an electric circuit so as to give an alarm when the temperature becomes excessive, as in the case of fires and the like. It consists of a capsule containing a volatile liquid fastened down to a stout non-conducting base, and connected to the electric terminals of a circuit containing an alarm bell by means of strips of fusible metal passing through the solid base. When the temperature rises, the expansion of the liquid within the capsule forces the latter away from the base, and in so doing ruptures the fusible connections, thereby breaking circuit and giving the alarm. Should the capsule fail to rupture the connections, a slight increase of temperature will melt the solder and break circuit.—E. G. C.

*An Improved Machine for Straining Pulp and the like.* P. Reinicke, Coblen, Germany. Eng. Pat. 11,531, June 12, 1893.

THIS is an improved machine for straining pulp and the like, having fixed on a rotary shaft a number of discs adapted to reciprocate within splices, on both sides of which annular screens are arranged, the said shaft being caused to produce abrupt reciprocating motion in an axial direction for the purpose defined. The discs are inclined upon the shaft of the strainer, or are corrugated, so as to give one or more lateral impulses when revolved.

*A New or Improved Hydrostatic Apparatus for Automatically Mixing with Water, or other Liquid, Disinfectants, Softening or Purifying Substances, or Saturated Solutions of any Soluble Substances in Definite Proportions.* F. Spence, Newcastle. Eng. Pat. 13,146, July 5, 1893.

THE apparatus consists of a conical vessel of glass or earthenware, provided with an external channel, terminating outside the vessel near the top and communicating with the interior of the vessel below; the top of the vessel stands above the level of the water in the tank or cistern in which it is placed. While the tank is being emptied, water flows out of the vessel through the disinfectant or other substance contained therein and mixes with the water outside, while during the filling of the tank, water flows into the vessel and forms a solution of the substance ready for discharge at the next emptying of the tank.—E. G. C.

*Improved Apparatus for Cooling or Warming Liquids.* R. Ch. Baker, London. Eng. Pat. 14,145, July 21, 1893.

THIS invention relates to the shape of tubes used in open-air coolers, consisting of a number of horizontal tubes disposed in rows one above the other, over which the liquid to be cooled runs down in a thin stream, whilst the cooling liquid circulates through the tubes in an opposite direction. The new tubes are of elliptical section with the major axis horizontal, the surface of the tubes being corrugated longitudinally, and each tube carries along its underside a longitudinal depending fin or blade serrated or undulated at the lower edge.—B.

*Improvements in Bottles for Holding or Containing Poisons or Poisonous Liquids.* R. H. Quinn, Pendleton, Lancashire. Eng. Pat. 11,553, July 28, 1893.

THE bottle is constructed with a flat side upon which it can rest, and the two adjacent sides and end are bevelled so as to make the top side short and narrow. The neck is placed obliquely at the end and above the level of the top, so that

when the bottle is laid upon its flat side the contents cannot run out if it be uncorked. The word "poison" is moulded upon the upper side, and the end is so narrow that the bottle cannot stand upright. The sides of the bottle are made so narrow that to grip it properly the fingers must come into contact with the surface on which it rests.

—E. G. C.

*An Improvement in Apparatus for Drying Animal and other Matters.* W. C. Carne, Birkdale, and W. E. Rowlands, Liverpool. Eng. Pat. 16,198, August 28, 1893.

THE object of this invention is to economise first cost in apparatus, also time and fuel used in the operation. The drying cylinder (in which an agitator revolves) is enclosed in a steam boiler, either entirely, or except at the ends. By this arrangement there is little loss of heat by radiation, and no necessity for the discharge of condensed water, which is required when the ordinary steam-jacketed cylinder is employed.—J. J. K.

*Improvements in Drying Apparatus or Chambers relating to the Drying and Preserving of Animal, Vegetable, and other Substances.* T. Christy, London. Communicated by K. F. Töller, Bremen, Germany. Eng. Pat. 21,787, November 15, 1893.

THIS invention relates to an apparatus for drying (without the application of heat) organic or inorganic substances, which are liable to decompose when dried by heat, or exposed to the action of open air. The apparatus is composed of a chamber of zinc, or wood lined with zinc, the door of which is made air-tight by means of india-rubber or other fittings. The material to be dried is placed in boxes or trays, which are supported on brackets in the chamber. The water-absorbing chemicals, such as sulphuric acid, chloride of lime, or unslaked lime, are placed in a receptacle on the bottom of the chamber, and the door is then hermetically closed, when the drying process commences. The same substances are proposed for the purpose of preventing shop windows from becoming wet in cold weather, and for preserving surgical instruments and other metallic goods free from humidity.—J. J. K.

*An Improved Fluid for Preventing and Removing Incrustation in Boilers.* S. Borecký and J. Komčstik, Pardubice, Bohemia. Eng. Pat. 5431, March 15, 1894.

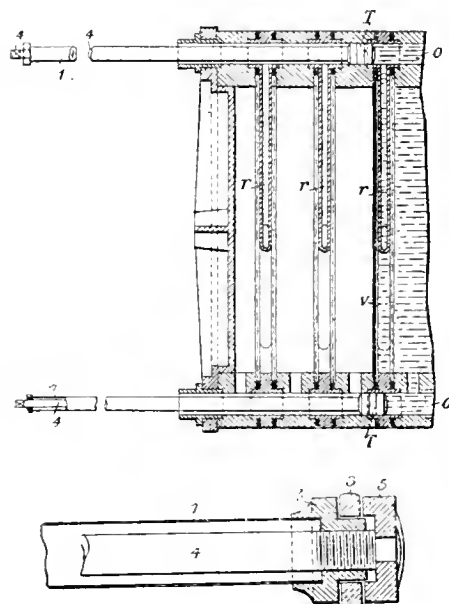
CAINED soda, oil, oil soot, lime water, and granulated zinc, are mixed together in the following approximate proportions:—Calcined soda, 60 parts; olive oil, 2 parts; oil soot, 1 part; lime water, 80 parts; granulated zinc, 1 part. One part of this fluid is added to every 200 parts of water in the boiler.—J. J. K.

*Improvements in Filter Presses.* T. F. Seitz, Kreuznach, Germany. Eng. Pat. 5973, March 22, 1894.

THE principal improvement embodied in this invention consists in the application of means for shutting off the liquid from a number of the press-plates, thereby reducing the number of plates in operation at any time as may be desired. The accompanying figures indicate the arrangement.

The filtering liquid enters by the lower pipe *o'*, and, having passed through the filtering surfaces into the hollow press-plates, is emitted through the pipes *r* into the upper collecting pipe *o*, whence the purified liquid is withdrawn. If it be desired to exclude any of the press-plates from the operation, the plugs *T* held on the end of tubes *1* are pushed forward as shown, confining the liquid to the space in front of the plugs. A metal plate *e* is further inserted behind the last press-plate in use, shutting off the liquid from the space behind. The enlarged section shows the construction of the plugs *T*, the india-rubber ring *3* being held between

the flanged caps 2 and 5 secured to the tube *1*, and to the concentric rod 4. On the rods 4 being turned after the



plugs have been pushed into the desired position, the caps 5 are screwed against the india-rubber rings, which by their expansion make tight joints within the pipes *o* and *o'*.—B.

*Improvements in and relating to Apparatus for Distilling Sea-Water and other Liquids.* H. E. Fouché, Paris. Eng. Pat. 6594, April 2, 1894.

THE improvements consist in the application of "triple effect" to the distillation of sea-water, in combination with the use of low-pressure steam for heating. The apparatus is of the tubular kind, provided with parallel tubes for the purpose of evaporation and with coils for condensation. The steam for heating the last set of tubes is taken from the third cylinder of a triple-expansion steam engine, or reduced to equally low pressure by means of a reducing valve if taken from an auxiliary boiler when the main engines are not working. The sea-water is heated to nearly boiling point before entering the apparatus, by being passed through a feed heater, and is kept in constant circulation in each department by means of special pumps. The steam given off in the final vessel is used for evaporation in the second vessel, and the vapour raised in the latter serves for heating the third. The condensed vapours are collected and returned to the fresh-water cistern, and the pressures in the various sections are maintained and equalised by means of water columns and siphons between adjacent vessels, the whole of the arrangements working automatically after the first start.—B.

*An Improved Filter for Air, Gas, Steam, and the like.* K. Moeller, Brackwede, Germany. Eng. Pat. 7523, April 16, 1894.

THE filter consists of a casing traversed by vertical tubes of filtering material opening at both ends into chambers attached to the casing. The dust-laden air enters the tubes at both ends, leaving the dust inside the tubes, whilst the purified air passes into the casing which is provided with outlet branches. Movable brushes are fitted, one in each tube, and, on being passed up and down, deposit the dust in the lower chamber, where it can be collected and removed in any convenient manner.—B.

*Improvements in or appertaining to the Pipes or Tubes of Heating, Cooling, or Evaporating Apparatus.* W. P. Thompson, Liverpool. From A. Dumas, Brévaux, France. Eng. Pat. 3182, April 25, 1894.

This invention contemplates insertion into tubes for heating or cooling apparatus of longitudinal partitions or wings of various shapes in cross section, the purpose being to increase the metallic surfaces of the tubes. The partitions are removable, and may be either straight or of helical form longitudinally.—B.

## II.—FUEL, GAS, AND LIGHT.

*Water-Gas in America.* F. Bredel. Zeits. angew. Chem. 1891, [10], 292—293.

COMMENTING on the observations of Lunge (Zeits. angew. Chem. 1894, 137) that the supply of gas-coal is failing in the United States to such an extent that ere long (perhaps even now) it will be necessary to import gas-coal from England, the author remarks that on the contrary, the United States is the richest of any country both as regards the quantity and quality of gas-coal. The following are the more important coal-fields:—Pennsylvania, whence the best gas-coal is obtained, viz., the so-called Youghiogheny coal. West Virginia; the gas-coal here is richer in sulphur than the last-named. The coal of the Indian Territory is a relatively good gas-coal and is much used in Texas. The Colorado coal-field yields a very good gas-coal. The coal of the State of Washington is also considerable in quantity, and good in quality as a gas-coal.

With the best Youghiogheny gas-coal a yield\* of 5—5.15 cub. ft. of gas per pound of coal is afforded. The lighting power of this gas is 18—20 candle-power (London Parliamentary). The author then enters into details respecting the gas and coke production of the various sorts of American coal.

Coal-gas is cheaper than water-gas when both are calculated to candle-power; the only advantage that water-gas possesses over coal-gas is that the former possesses an illuminating power of 23—28 candles per 5 cub. ft., whereas the highest limit that can be obtained with the latter, is 20 candle-power. Many manufacturers enrich their coal-gas with naphtha gas, which is made in the same way as Pintsch's gas.

In many States a mixture of two-thirds coal-gas and one-third water-gas is supplied; this gives a very beautiful light equal to 22 candle-power. The following table shows the price and quality of the gas supplied in various parts of the United States:—

	Price per 1,000 Cub. Ft.	Illuminating Power in English Candles.	
New York .....	Dols. 1.25	22	Mixed gas.
Philadelphia ....	1.60	22	"
Cleveland .....	0.80	18.75	Coal gas.
Wheeling, W. Va.	0.75	18	"
Chicago .....	{ For lighting gas 1.20 For heating and motive power 1.00 }	23	Water-gas.
Milwaukee .....			
Baltimore .....	{ For lighting gas 1.00 For heating and motive power 0.80 }	24	Water-gas (1) (Coal-gas (1/2)).
St. Louis .....			
Baltimore .....	1.25	28	Water-gas.
St. Louis .....	1.18	20	Mixed gas.

Contrary to Lunge's statement, purification of gas with ferrous oxide is more general than purification with lime. The latter is used in very few factories, and then only for the purpose of removing the carbonic anhydride from water-gas.—A. R. L.

### PATENTS.

*Improvements in and in the Mode of Utilising the Pressure in Cylinders or Reservoirs of Compressed Gases in the Application or Employment of such Gases.* The Manchester Oxygen (Bris's Patent) Co., Ltd., and W. M. Jackson, both of Manchester. Eng. Pat. 10,554, May 30, 1893.

THIS invention has reference to apparatus for utilising the pressure in cylinders containing compressed oxygen, to induce or draw in a stream of gas, such as coal-gas, so as to mix with the oxygen and thereby give a mixed jet for producing the oxy-hydrogen flame. The apparatus consists of an inner tube tapering to a very fine outlet, through which oxygen is passed under pressure, thereby inducing a current of coal-gas from the reservoir connected to the outer and enveloping cone and delivering the mixed jet into a suitable outlet pipe, as in the manner of an ordinary injector; the outlet pipe conducts the gases to a mixed jet burner. To prevent the fine outlet of the oxygen pipe being blocked up, one or more layers of gauze are placed over the oxygen inlet, and the gases, after mixing, are passed through a plug of fibrous material to prevent noise or vibration due to the injector acting injuriously upon the flame.—E. G. C.

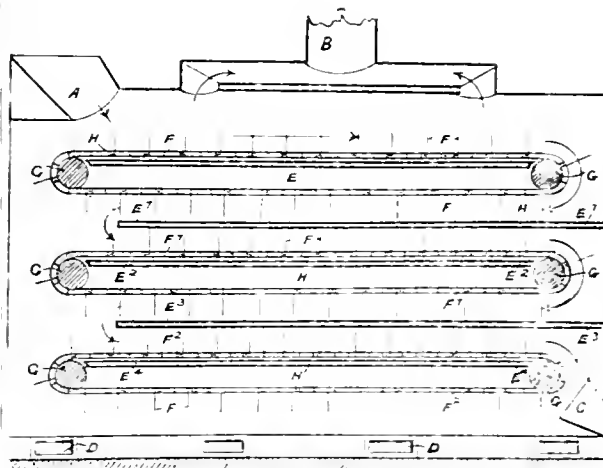
*Improvements relating to Incandescence Bodies for Incandescent Gas Lamps.* G. A. E. Schneider, Chemnitz, Saxony. Eng. Pat. 12,029, June 19, 1893.

THIS invention relates to the "hoods" of incandescent gas lamps, and consist in using a fabric of incombustible threads. The hood made from this is repeatedly dipped into a suitable liquid, in which absorbent, combustible, finely-divided fibres are suspended, whereby it is coated with a layer of them. It is then impregnated, dried, and finally burnt. The hood thus produced has, in addition to the usual skeleton of ashes, a fire-proof skeleton to support them.—R. E. P.

*Improvements in Ovens or Furnaces for Drying and Coking Peat Briquettes and the like.* E. Stauber, Hamburg, Germany. Eng. Pat. 14,203, July 22, 1893.

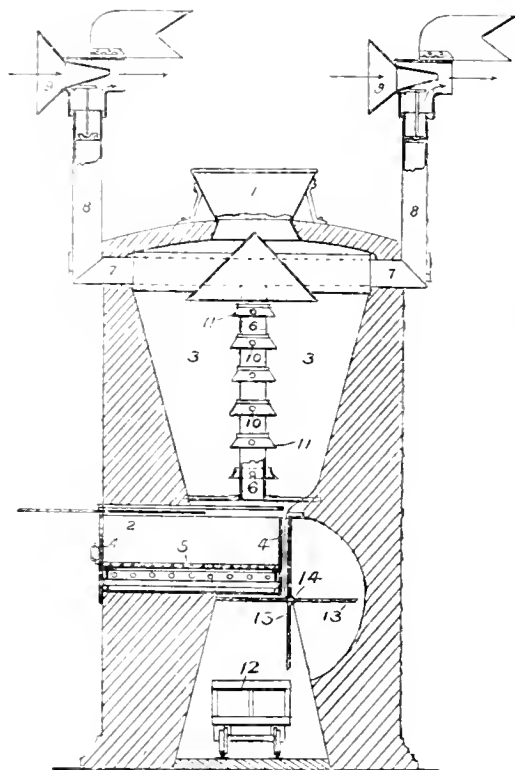
THE drying oven is constructed of iron, and has a feed hopper A, and an outlet B on top. The discharge-opening C and the air-inlets D are at the bottom. The briquettes

Fig. 1.



are fed in at A, and fall on to a hot plate E, along which a conveyor band F traverses them. They then fall on to a second hot plate E', are moved back along this by a second conveyor band F' until they fall on to the third hot plate, and so on, being finally delivered through C, thoroughly dried. The dried briquettes then go to the coking oven shown in Fig. 2, into which

Fig. 2.



they are fed through the hopper 1 until the coking space 3, which is closed by the plate 2, is filled. The hopper is then closed air-tight. The fire-box 4, filled with glowing fuel, is next pushed back into the fire-space 3, the disc 2 is withdrawn, and the coking commences. The gases drawn off are exhausted through the outlets 6, 7, 8, by the ventilators 9. After the removal of all moisture, air inlets, constructed on the exterior of the furnace, are from time to time opened. The outer air enters through small connecting tubes 10 in the caps 11, which are fastened to the central outlet 6. The air here mixes with the water-gas produced in the furnace, causing it to ignite and complete the coking process. The products of combustion pass along 6, 7, and 8 into the outside air. The fire-box being at this time drawn out by rotating the shaft 14 and the attached vanes 13 a quarter of a turn, the coked material is from time to time withdrawn into waggons 12. By these means the whole contents of the furnace are prevented from tumbling out when a waggon load is being withdrawn.

—R. B. P.

*Improvements in Apparatus for Washing and Purifying Gas.* F. D. Marshall, Copenhagen. Eng. Pat. 22,576, November 24, 1893.

The improved apparatus is two-chambered, the roof of the lower chamber, *a*, Fig. 1, being the floor of the upper one, *c*. An axial vertical tube, *d*, puts the two chambers in communication; the lower opening of this tube is flush with the roof of the lower chamber, while the tube projects from the floor to about one-third the total height in the upper

chamber. The lower chamber is about one-third full of water, and the pipe, *b*, leading the gas into the apparatus, dips into this water. The annular space between the projecting vertical tube and the walls of the upper chamber is also partly filled with water, and into this water dips a bell-like structure *f, g*, suspended against a counterpoise

Fig. 1.

Fig. 2.

Fig. 4.

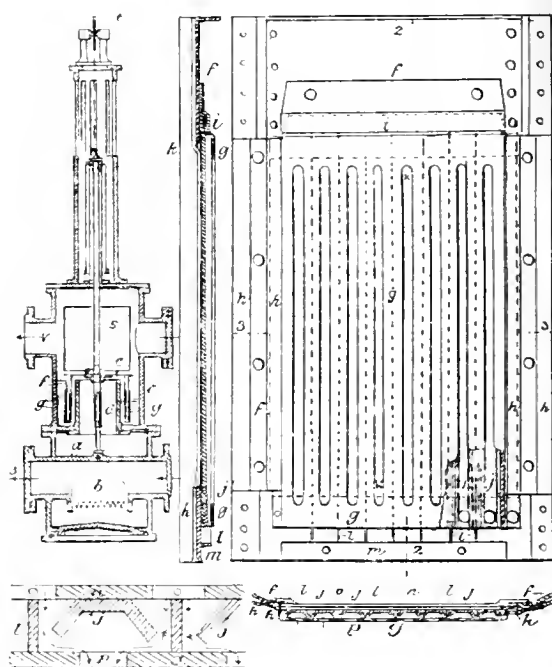


Fig. 5.

Fig. 3.

outside the apparatus, the connection between bell and counterpoise being a rod *s*, attached to the top of the bell and passing through a stuffing box in the roof of the upper chamber; the rod is attached to a flexible connection, which, after passing over a pulley *t*, suspends the counterpoise.

The bell itself is a highly complicated structure. It is double walled, each wall being built up of eight rectangular structures, joined together by their side edges; while the two walls so built up are joined to the single top plate, which completes the bell. A horizontal section of the bell therefore shows two regular and concentric octagons.

Each of the 16 rectangular structures is composed (see Figs. 2, 3, 4, and 5) of an outer plate *g*, perforated by eight vertical slits, and an inner plate *n*, similarly perforated. The perforations *p* of the outer plate alternate with those, *o*, of the inner plate. Between the two plates are a series of channelled bars *j, j* (riveted to the cross-bars *k, k*, which are attached to *g*), and a series of metallic strips *l, l*, alternating with the bars *j, j* (the strips are held in position by the angle-iron *m*, which is riveted to the framing *f*). The outer plate *g*, with its cross-bars *k, k* and their attached channelled bars *j, j*, can slide in and out under the guides *h, h* and *i*.

The action of the apparatus is as follows:—Gas enters the lower chamber, passing through the water therein and losing, therefore, part of its tar and ammonia; from the lower chamber it passes by the vertical tube to the inside of the bell, then through the apertures *o* (see Fig. 5) of the inner plates, where each stream is immediately divided into two by the strips *l*. The divided streams next pass through the narrow slits existing between *l, l*, and *g*, and thence by the apertures *p* of the outer plate, the gas reaches the space between the outer and inner walls of the bell. The outer

wall is similarly penetrated, and, passing through the water, the gas reaches the upper part of the upper chamber, in the wall of which is the exit pipe A.

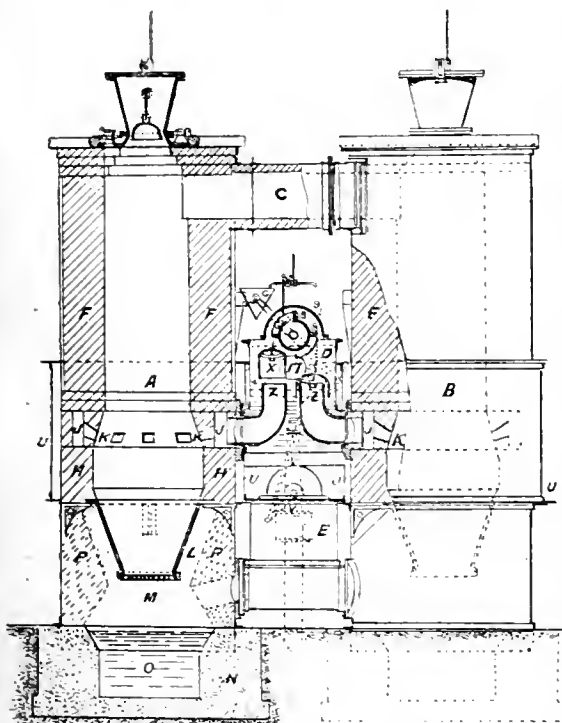
The bell is immersed to a great or small extent in the water of the upper chamber, according as the pressure of the gas overpowers the counterpoise.—E. R. B.

*Improvements in Methods and Apparatus for Generating Gaseous Fuel from Solid Hydrocarbonaceous or Carbonaceous Material.* B. H. Thwaite and G. Threlfall, London. Eng. Pat. 1690, January 26, 1894.

THE inventors describe a new form of fuel gas generator which, while it permits the use of "slack," carrying as much as 30 per cent. of volatile constituents, on the one hand, or "breeze," carrying as much as 25 per cent. of incombustible matters, on the other, and allows of the recovery of much of the radiated heat, produces permanent gas, suitable for internal combustion engines, &c.

Two generators, placed side by side and joined by three connections, as shown in Fig. 1, are employed. By means of an air-blast the fuel is rendered incandescent in each and then a definite cycle of gas manufacture is established, which consists in making the gas generated in the first generator descend through the incandescent fuel in the second, so that any steam or hydrocarbons still remaining in the gas produced by the first generator, is decomposed into H and CO in the second. The gaseous current is then reversed, the second generator now acting as crude gas producer, while the first ensures the permanent character of the gas. Throughout the process the direction of the gas current is reversed at short intervals of time. In Fig. 1, A and B are the two generators, and C, E, and K, J, Z, D, the three connecting conduits with their valves. The chamber D

Fig. 1.



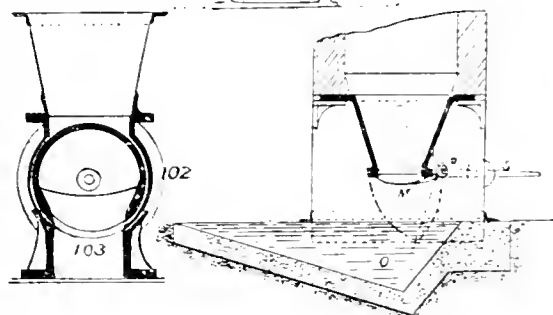
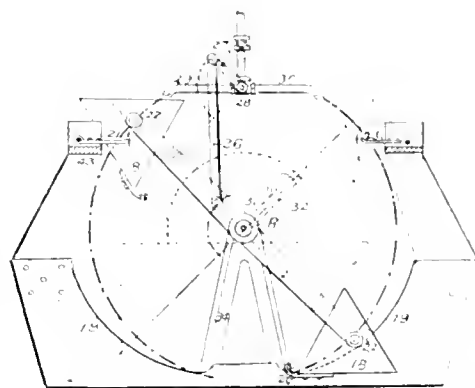
of the middle conduit contains the reversal gear, consisting of two bells, X and Y, suspended each over one of the exit passages Z Z of the respective generators, and the final gas exit, 17.

The bells forming the gas-reversal valves are suspended by chains attached to two projections, 8, 8, on the periphery of the wheel 7. The axle of this wheel carries a rocking

lever, 20 in Fig. 2, to the ends of which are suspended two accurately balanced pyramidal brackets, 18, capable of being filled from the water supply pipes, 29, 30, above them. The flow of water is controlled by the three-way cock, 28, which is operated, through the lever 27 and the link 26, by the rocking lever 20. The air-reversal valves are similarly operated by another link and lever (Fig. 1) from the rocking lever. The motion of the latter is determined by the fall of the upper bracket, which takes place immediately the amount of water it has caught overpowers the spring catch, 21, which retains it; the lower bracket is then swung up and caught by its spring catch (20), and water commences to flow into it, while, simultaneously, the direction of the water-supply alters.

Other special features of the apparatus are, 1 (see Fig. 4).

Fig. 2.

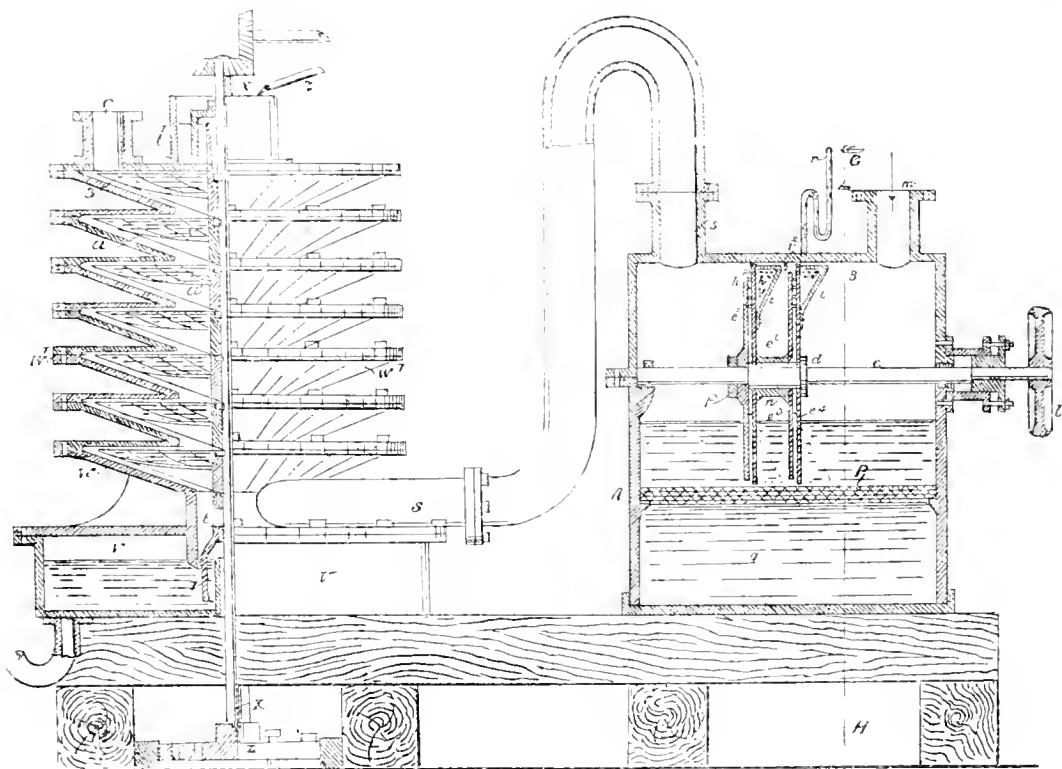


*Improvements in or connected with Hydrocarbon Burners.*  
A. Kiesow, London. Eng. Pat. 3319, February 15, 1894.

This invention relates to an improved method of inserting the wick into a lamp burning fluid hydrocarbons, and to an improved method of securing the burner to the vessel containing the fluid.—R. B. P.

*Improvements in and relating to the Purification of Gas.*  
E. Fleischhauer, Gotha, and M. Bernstein, Berlin. Eng. Pat. 4719, March 6, 1894.

This patent principally relates to a new apparatus for removing tar and ammonia from coal-gas. In the figure, A B is a vessel partly filled with water, and with gas inlet and outlet, *m* and *s* respectively; it also contains the horizontal shaft *c* driven by the pulley *d* and carrying four discs, *e*<sup>1</sup>



to *e*<sup>2</sup>. The second and fourth of these discs carry a series of pockets, *i*, *l*, having one side open, and sets of perforations, *g*, *h*, on the other sides: a set of pins or stops, *k*, keep the discs 1 and 3 apart from 2 and 4 respectively. The stop *r* prevents gas passing, above the discs, from the entrance to the exit side of the apparatus, without being intimately engaged by the discs.

The action of this part of the apparatus is as follows:—Any gas in A B is caught up by the pockets of disc *e*<sup>1</sup>, which is revolving rapidly, and is driven by centrifugal force partly through the perforations at *g* and partly through those at *h*. That passing through *h* impinges against the casing of the apparatus, while that passing through *g* impinges against disc *e*<sup>2</sup>, and thus by centrifugal force is driven through the narrow spaces between the pins *k*, and impinges against the casing. Having arrived in the space between the pairs of discs, it is caught up by the pockets of *e*<sup>2</sup>, and is treated by *e*<sup>2</sup> and *e*<sup>3</sup> as it was by *e*<sup>1</sup> and *e*<sup>2</sup>. Gas and water-sprays are therefore being constantly dashed against the sides of the apparatus, and the tar, which is constantly being deposited on the walls, is continually being washed off. The object of the hurdle *p* is to keep the bottom part of the liquid contents from agitation, so that tar which has sunk below this level may quietly separate out. The gas now passes by *s* to the second part of the apparatus.

This consists of a vertically-adjustable shaft *x*, rotating in upper and lower bearings *z*, and carrying seven pans *u*, the whole being enclosed in a casing *w*, which

resembles a series of bottomless pans joined together, and which communicates at the bottom with a reservoir *v* partly filled with water.

Gas entering by *s* passes through *v* into *t*, and then between the rotating pans and the casing to the exit *e*; its upward passage is greatly assisted by the presence, on the under side of the pans, of deep radial ribs *u*<sup>1</sup> reaching nearly to the casing, which have a fan-like action on the gas when the shaft is rotated. The pans are kept supplied by water through *2* and *t*<sup>1</sup>, which is constantly being flung against the sides of the casing, partly as consequence of the rapid rotation of the pans, and partly owing to the action of certain wings *y*, which project downwards from the horizontal parts of the casing into the water in the pans, in a slanting direction, opposite to that of the rotation. This agitation and sub-division of the water is further aided by the radial partial partitions *3* in the interior of the pans.—E. R. B.

### III.—DESTRUCTIVE DISTILLATION. TAR PRODUCTS, Etc.

*The Commercial Position of the By-Products of Gas Manufacture.* P. Mallet. Bull. Soc. Chim. 9, 218—220.

The most important factor influencing the commercial value of the by-products from the manufacture of gas is the rapidly increasing number of coke ovens at work. In 1893 there were 210 of these ovens in use in France, capable of carbonising from 180,000 to 190,000 tons of coal per annum; in England there were about 413, and in Germany more than 1,250. Were, however, the whole of the coal required for the preparation of the coke used in metallurgical operations treated in ovens, the quantities of by-products turned out would rival and sometimes exceed the amounts produced by gas manufacturers; for in France  $1\frac{1}{2}$  million tons of metallurgical coke are used per annum, which would yield 18,000 tons of ammonium sulphate, 44,000 tons of tar, and 3,000 tons of light oils; while Germany requires 74 million tons of coke, equal to 110,000 tons of sulphate, 375,000 tons of tar, and 10,000 to 30,000 tons of light oils.

Tar itself is likely to continue to fall in value, chiefly owing to the progress that has been made in designing furnaces capable of burning economically fine coal alone, this having resulted in a diminished demand for pitch for briquette manufacture. Almost all the tar products, including benzene and anthracene, will in all probability become still cheaper than they are at present, with the exception perhaps of the heavy oils, for which the railway companies keep up a constant demand for preserving sleepers, the use of copper and zinc salts for this purpose dying out by reason of the higher cost and inferior results. At the same time, the attempts that are being made to replace wooden sleepers by iron must be carefully watched.

Of ammoniacal products, the trade done in liquefied ammonia, ammonium chloride, and nitrate is very insignificant, while the price of aqueous ammonia depends on the ammonia-soda trade. In 1891, 116,323 out of 196,583 tons of carbonate of soda were made in France by this latter process, requiring, on the average of 8 kilos. of ammonia per ton of soda, 930,000 kilos. of ammonia. Thirty-five years ago ammonium sulphate was used only as a source of ammonia alum, and it continued to be exported from France till 1868. Then its use as a manure began to spread, and the price rose from 28 to 50 francs per 100 kilos., where it remained till 1882, when the competition of the South American sodium nitrate became noticeable. In 1873 the total exports of nitrate were 285,000 tons, and in spite of a tax of 6.24 f. per 100 kilos. imposed by Chili in 1880, this amount has risen to 932,000 tons in 1889, while the price has fallen in the same time from 44 f. to 19½ f. Legrand has calculated that Belgium consumes per unit of surface six times more nitrate than Germany, nine times more than France, and 11 times more than England, and that the present requirements are only  $\frac{1}{10}$ ,  $\frac{1}{30}$ , and  $\frac{1}{100}$  respectively of what they should be were the soil properly manured; but the deposits of Tarapaca and Acatama are estimated to contain 250 million tons, and this amount at a very favourable computation will last more than another 40 years, so that the only hope for better prices in the sulphate market lies in a possible union between the manufacturers.

Coke is after all the most important by-product, its value reaching half that of the gas made from the same quantity of coal. Thirty years ago there was no domestic outlet for coke at all, and it was hardly used in any manufacturing operation except lime-burning; now, however, improved forms of warming and ventilating stoves are being brought out, constructed to burn either coke or anthracite (and the latter is too expensive to use in France, costing, as it does, in Paris, without tax, 50 f. per ton), so that its use is constantly spreading among private consumers. It is being employed, too, in blast furnaces, many in the Loire district being run with 30 to 40 per cent. of coke. Outlets are also being found for the smalls, some furnaces being constructed which will burn without a blast up to 70 per cent.

of the fine powder. The outlook, therefore in this department is favourable, the chief obstacle to any increase in price being the gas-makers themselves, who are using a smaller proportion of their make, and consequently throwing more on the market. T. H. L.

*New Apparatus for Fractional Distillation.* E. Vazeille. Bull. Soc. Chim. 11—12, 1894, 289—292.

See under XVIII., page 704.

*The Removal of Sulphur from Mineral Oils at Chicago.* —Biche and —Roume. Annales des Mines, 5, 95, 1894. Proc. Inst. Civil Eng. 1894, 116, 187—89.

At the Standard Oil Company's refinery at Whiting, near Chicago, a new establishment covering 300 acres and employing 1,000 men, the crude oil of Lima, in Ohio, which was formerly used only as fuel, on account of its disagreeable odour and the sulphur gases evolved in burning rendering it unfit for illuminating purposes, is now refined by methods which destroy the sulphur compounds, leaving the hydrocarbon oils unaltered. These oils are very variable in composition, their density ranging from 0.816 to 0.950, their average yield on treatment being—

Naphtha and burning oil .....	56.88
Heavy oils .....	32.00
Residues .....	9.50
Water .....	0.75
Sulphur .....	0.65
	100.84

Two processes are used in the treatment, both being based upon the use of cupric oxide as a desulphuriser, a method used in France for desulphurising glycerin in the soap manufactories at Marseilles.

In the first method the crude oil is subjected to distillation without reagents, the oil-vapours from the still being passed into vessels containing cupric oxide, where the sulphur is separated as cuprous sulphide, while the cleaned oil passes on to a large tubular cast-iron condenser cooled with water, where it is collected for further treatment. In this way the whole of the volatile contents of the oil are subjected to the desulphurising treatment.

In the second method, which, in spite of its involving a double operation, is now becoming general, the first distillation is conducted in the ordinary way, the lighter products, spirit and burning oil, being kept apart and subsequently transferred to another still provided with a stirring apparatus, in which they are heated with finely-divided oxide of copper, the contents being kept intimately mixed by the agitator. This second distillate is perfectly free from sulphur, showing no trace of colour in a solution of acetate of lead, which is readily blackened by the crude oil, as well as by the first distillate, and as burning oil it is equal in quality to that obtained from Pennsylvania petroleum, as it burns without a sulphurous odour and does not blacken the chimney of the lamp.

In conducting the operation it is necessary to use a large excess of cupric oxide, otherwise the pipes of the still are rapidly corroded, even when heavy steel ones are substituted for those of sheet iron ordinarily used.

The residue in the still consists of a mixture of tar and cuprous sulphide. It is drained and finally squeezed on a press or roller mill, giving a thick fluid, which yields a solid cake in the filter-press. The pressed cake is broken up and heated in an inclined cylindrical furnace lined with fire-brick, with a fireplace at the lower end, the heat being just sufficient to start the combustion of the mass, which is continued by the oxidation of the sulphur, the final product being a finely-divided powder of cupric oxide, which is returned for use to the refinery after being dried in thin layers under shed in the open air.

About 10,000,000 barrels of Lima oil are refined annually, a further quantity of about 30,000 being treated in the smaller establishment at Lima, where the method was originally developed.

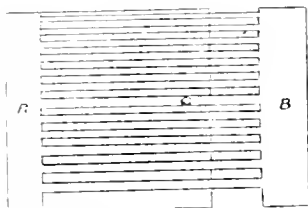


## PATENTS.

*Improvements in the Distillation of Petroleum.* J. A. Wanklyn and W. J. Cooper, both of New Malden, Surrey. Eng. Pat. 4097, February 24, 1893.

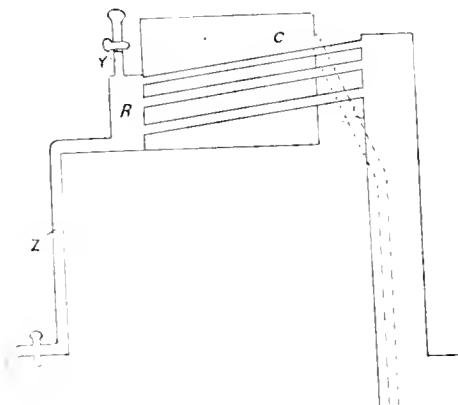
In this apparatus the boiler, or still, has a tall trunk, rising up to the condenser C. At its upper end there are a number of outlets, each connected to a tube in the condenser, which

Fig. 1.



is of the surface-condensing type. Its tubes, which correspond in number to the outlets from the still trunk, slope downwards from their inlet ends, and discharge into a common receiving chamber R. From the latter an outlet pipe Z passes downwards, of sufficient length to get a column of liquid 40 ft. high. By means of two cocks, one on the

Fig. 2.



top of the receiving chamber and the other at the lower end of the outlet pipe, the distillation can be carried on at all pressures from an approximate vacuum to that of the atmosphere. This form of outlet enables the apparatus to create and maintain its own vacuum. The cooling medium used in the condenser is the liquid to be distilled, which is thus heated to a certain extent before it is fed into the still.

—R. B. P.

*An Improved Artificial Asphaltum or Pitch, and Process for Making same.* R. Hadden, London. From G. R. Anderson, A. H. Shotton, W. Kochler, and C. H. Dorer, all of Cleveland, Ohio, U.S.A. Eng. Pat. 9550, May 12, 1893.

This invention relates to the production of artificial asphaltum, or pitch, from hydrocarbon oils or their products (especially the heavy distillates and the residuum from distillation, which may or may not have been previously treated with acids) by the addition of resinous substances, preferably those from the pine tree, and, if sufficient acid is not present, the addition of acid, preferably sulphuric acid, and subjecting the whole to heat and partial distillation. The amount of each ingredient is determined by the qualities the final product is to have, and all proportions are claimed.

—R. B. P.

## IV.—COLOURING MATTERS AND DYES.

*Acetine.* E. Kopp and E. Grandmougin. Bull. Soc. Ind. Mulhouse, 1894, 112—116.

*Manufacture.*—Heat together in an enamelled copper vessel, by means of an oil-bath, 60 parts of glycerol and 82 parts of glacial acetic acid for 12—15 hours at 120° C., then gradually raise the temperature to 160° C. to drive off excess of acetic acid. The product consists of 93 parts of concentrated acetine, a dense liquid smelling more or less strongly of acetic acid, and varying in colour from pale yellow to dark brown according to the glycerol employed.

*Valuation.*—It is evidently important that the union of the glycerol with the acetic acid should be as complete as possible; in other words, that the acetine should contain the maximum of combined, and the minimum of free acetic acid. These two factors are therefore estimated. The density, which varies from 1.1608—1.1896 may serve as a rough indication, the more free acetic acid present the less the density. The estimation of free and combined acetic acid is thus carried out:—50 grms. of acetine are diluted with water to 500 cc. In 50 cc. of this solution determine the free acetic acid with normal caustic soda and phenolphthalein indicator. Then to 10 cc. of the acetine solution add 15 cc. normal soda, heat to boiling, and boil for five minutes, then after dilution estimate excess of soda with normal acid. For example, if 4.5 cc. normal acid are required, the combined acetic acid corresponds to 15—4.5=10.5 cc. of normal caustic soda (1.5 cc. being the amount necessary to neutralise the free acid of the 10 cc.) = 9 cc. normal soda, which, for 50 grms. acetine, would mean 27 grms. combined acetic acid, or 54 per cent. It is possible also to estimate the glycerol, but the process is too long for practical use, and is, moreover, unnecessary, the above estimations indicating sufficiently the value. The following table gives the results of analysis of three samples:—

No.	Density.	Acid, Free.	Acid, Combined.	Remarks.
		Per Cent.	Per Cent.	
I.	1.1774	9.2	46.0	Acetine obtained with above proportions of glycerol and acetic acid.
II.	1.1896	6.98	55.7	.. ..
III.	1.1608	23.0	43.5	Almost colourless; smells very strongly of acetic acid.

Sample II. is good, I. of medium quality, III. contains too much free, and too little combined acetic acid.

*Employment.*—As a solvent, especially for basic colours, e.g., Induline and Perkin's violet. To dissolve induline, heat with the acetine in a copper vessel, boil for two hours, allow to cool, and pass through a silk filter.

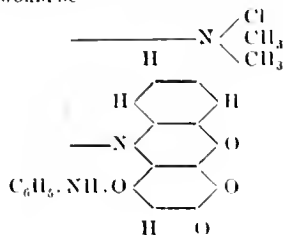
*Supplementary Note.*—Ethyl- and methyl-tartaric acids, which are used for the same purpose as acetine, are valued in much the same way, viz., by estimating free and combined tartaric acid. Acetine is to be preferred as a solvent, because acetic acid injures the fibres less than tartaric acid.—R. B. P.

*Oxindophenolic Colouring Matters from Gallanilide and Galloparatoluide.* P. Cazeneuve. Bull. Soc. Chim. 11, 1894, 85—87.

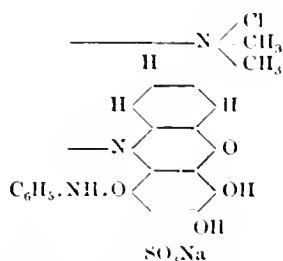
In a French patent of July 27th, 1877, Durand and Huguenin describe the preparation of a blue colouring matter obtained by the action of nitrosodimethylaniline on the condensation product of tannic acid and aniline. The olive-green product insoluble in water is converted into the sulpho-derivative by boiling with alcohol and sodium bisulphite, or by the action of fuming sulphuric acid. Both the sodium and ammonium sulpho-salts are valuable blue dyes, sold under the name of "Gallic blue" or "Tannin indigo." This compound is undoubtedly a derivative of gallanilide.

The author has obtained a similar compound from gallotannolide. The first product is an insoluble green crystalline substance; the ammonium and sodium salts of the sulpho derivative are blue colouring matters closely resembling those from gallanilide in tint and character.

The author considers these compounds to be analogous in constitution to indophenol. For the gallanilide derivatives the formula would be—



and—



(See under XX., page 755)—L. T. T.

### 2,3-Naphthylene Diamine. P. Friedländer and S. v. Zakrzewski, Ber. 27, 761—765.

THEORETICALLY 10 different naphthylene diamines can exist, of which at present only 9 have been obtained. The authors have prepared the missing isomide (2,3), employing as starting-point  $\beta$ -naphthol-disulphonic acid R, which (according to Pfitzinger and Duisberg) has the constitution 2,3,6. This was fused with 5—6 times its weight of caustic soda at  $200^\circ$ — $220^\circ$ ; the termination of the reaction is marked by thinning of the melt, and cessation of foaming. On solution in water, nearly neutralising, filtering, and faint acidulation of the filtrate, shining crystals separate, consisting of the sodium salt of dihydroxynaphthalene monosulphonic acid. On boiling this with mineral acids the sulphonic group is removed and a dihydroxynaphthalene results; since this is not the well-known 2,6 modification, it follows that it must be the 2,3. Finally, by heating this with ammonia to  $240^\circ$  for 12 hours, the hydroxyl groups are displaced by amidogen, and 2,3-diamidonaphthalene results. This melts at  $191^\circ$  and is readily soluble in alcohol, extremely so in ether, but only with difficulty soluble in water. The hydrochloride is easily soluble in water, only sparingly soluble in excess of acid. With 1 molecule of a diazo-compound it combines, forming a red diazo dyestuff. With acetic anhydride it forms a diacetyl derivative,  $\text{C}_{10}\text{H}_6(\text{NH}(\text{CO}.\text{CH}_3))_2$ . When 2,3-dihydroxynaphthalene is heated with ammonia to a lower temperature than that requisite to form the diamine, 2,3-amidonaphthol results (10 parts concentrated ammonia at  $135^\circ$ — $140^\circ$  for some hours); this melts at  $234^\circ$ . 2,3-naphthylene-diamine does not form a tetrazo derivative with nitrous acid, but produces a weakly basic azinide,  $\text{C}_{10}\text{H}_6\text{N}_3$ . Fusion with oxalic acid forms dihydroxynaphthylquinoxaline; with dihydroxytartaric acid in hydrochloric solution, naphthyl-2,3-quinoxaline orthodicarbonic acid is produced as a nearly insoluble precipitate.—C. R. A. W.

### The Manufacture of Copper-plate Ink. A. M. Villon. Bull. Soc. Chim. 9, 1893, 929—936.

THE inks employed in printing from a copper plate are compounded in a somewhat similar manner to those used

by letterpress printers, but the "varnish" is usually thinner and lampblacks are replaced by other forms of pigment. Copper-plate varnish, or "burnt oil," is prepared in three qualities, thin, medium, and strong. According to the older methods of manufacture the raw linseed oil was heated in a movable pot, set fire to, and allowed to burn for five minutes, well stirred, removed from the fire, and extinguished by means of a closely-fitting lid. Several slices of stale bread were then successively introduced with a pair of tongs and stirred round in the oil until they were browned but not burnt by the heat, this device—which is still believed in by almost all practical varnish makers—having for its object to remove the "greasiness" of the oil. Occasionally peeled onions were used instead of the bread to the same end, although the action of either substance on the oil is quite unknown. This operation being over, the oil was either allowed to cool down, or else inflamed again for a few minutes, put out, stirred up, and again set light to, till the requisite strength was reached, the strong varnish having, when cold, a viscosity resembling molasses. In large modern ink-works the linseed or nut oil is first refined or "freed from grease" by treatment with nitric acid or magnesia, then 200 litres are warmed slowly up in a pan, so that after three hours the temperature is  $180^\circ \text{C}$ , 500 grms. of potassium bichromate in fine powder are added, and the heat raised to  $300^\circ$ ; when the frothing is over it is increased to  $315^\circ$ — $320^\circ$ , and 500 grms. of Prussian blue ("pencil blue") put in, and the heat maintained—for the strong varnish—half an hour. For the medium varnish the oil is kept at  $300^\circ$ — $305^\circ$  for five minutes only, and for the thin it is not allowed to exceed  $275^\circ$ , where it is maintained from 15 to 30 minutes. The best varnishes are made from nut oil, but sufficient viscosity for the strong variety cannot be obtained without the addition 12 to 15 per cent. of linseed oil. Oxygen may be employed instead of fire heat, by warming either nut or linseed oil, or both together, to  $180^\circ$  and passing over them a current of the gas. However prepared, the varnishes must be allowed to settle for a month.

As black pigments, instead of the ordinary lamp- or "vegetable" black, the following substances are employed:—*Frankfort-black*, prepared by calcining the refuse from wine-making; *fish-black*, from oyster shells, &c.; *cork black*, and "*German*" *black*, made from a mixture of these substances together with greasy bones. The carbonisation takes place in specially designed retorts, holding from 40 to 50 kilos, each, set on tiles over the hearth, and so arranged that the acetic acid and other liquid products can be collected, and the permanent gases burnt underneath. The resulting blacks are ground with water, carefully levigated, and moulded into "drops." In compounding the inks, mixtures of these pigments are employed, as each one has a special shade of its own, a good mixing being as follows:—*Frankfort-black*, 50 parts; *bone-black* and *fish-black*, 25 parts each. On the small scale, the ink is often made with a muller and stone, the black being moistened with a few drops of thin varnish, and when thoroughly mixed, strong varnish added until the proper consistency is reached, blue being put in if desired. On a larger scale 1,000 parts of thin varnish are heated in a steam-jacketed pan provided with a mechanical agitator, 50 parts of "pure" blue added, the temperature raised to  $150^\circ \text{C}$ , and, after half an hour's stirring, 25 parts of rosin soap thrown in. The mass is cooled over night, and 500 parts of the previously-described mixed black and 200 of strong varnish mixed in, and the whole finally ground on granite or iron roller mills. The author quotes a number of formulae for the manufacture of different grades of ink, the following being specially recommended as having a deep black colour, free from "bronze" and keeping well. Thin nut-oil varnish, 1,000 parts; strong varnish, made from 50 per cent. of nut oil and 20 per cent. of linseed oil, 200; Carbauba wax, 25; paraffin, 35; rosin soap, 25; Paris-violet, 0.5; "pure" blue, 10; cork black, 100; blood black, 50; *Frankfort black*, 20; and *bone black*, 150 parts. The violet is dissolved in alcohol before being added to the hot varnish, and the blue is ground and put through a silk sieve.

—F. H. L.

## PATENTS.

*The Production of Colours on Fibres.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 11,891, June 16, 1893.

THE colours are produced from the dyestuffs referred to in Eng. Pats. 8530 of 1890, 9636 of 1891, 22,641 of 1891, 11,395 of 1892, and 15,439 of 1892 (this Journal, 1893, 238, 593, and 754), by diazotising them on the fibre and subsequently treating the fabric in a hot acid or alkaline bath. Similar shades are obtained if instead of diazotising and subsequently treating the dyed fabric as above, it is subjected to the action of weak oxidising agents. For instance, the colouring matter from one molecular proportion of tetra-*o*-ditolyl chloride and two molecular proportions of 1,3'-*n*-naphthylamine sulphonic acid is dyed on cotton in a bath containing sodium sulphate and sodium carbonate. After rinsing, the dyed fabric is immersed in an acidulated bath of sodium nitrite for about half an hour, when the bluish-black colour is turned to violet-brown. The material, after washing, is then heated at about 50° C. in a 5 per cent. bath of sodium carbonate, when a cutch-brown shade is produced fast to washing. A similar shade is obtained by dyeing the fabric as above and then treating it for some time in a weak bath of chloride of lime. The strength of the bath is about 0.25° B. and the temperature 30°–40° C. The products obtained are supposed to be azoxy compounds.

—T. A. L.

*Improvements in the Manufacture of Colouring Matters.* H. H. Lake, London. From A. Leonhardt and Co., Mülheim-on-the-Maine, Germany. Eng. Pat. 12,323, June 22, 1893.

By condensing *m*-amido-*p*-cresol, 1,2,4- $C_6H_3(CH_3)_3NH_2.OH$ , with formaldehyde, preferably in presence of a mineral acid, it yields diamido-dihydroxyditolylmethane, which, on treating with a dehydrating agent, such as sulphuric acid, and subsequently oxidising, is converted into a colouring matter. A colouring matter of the rhodamine series is also produced by heating *m*-amido-*p*-cresol, and phthalic anhydride in presence of sulphuric acid. The following quantities are employed for the preparation of the methane derivative. About 12 kilos. of *m*-amido-*p*-cresol are dissolved in 200 litres of water, containing 14 kilos. of 30 per cent. hydrochloric acid, and treated with 3.8 kilos. of a 40 per cent. formaldehyde solution. After standing some time, the solution is heated to 60° C., a small quantity of sodium carbonate is added to precipitate impurities, and the diamido-dihydroxy-ditolyl methane is precipitated from the filtrate by adding sodium carbonate. The product, when crystallised from spirit, melts at about 225° C. By heating it with five times its weight of sulphuric acid to 100° C., it forms a yellowish-red melt, and when a sample supersaturated with caustic soda gives no precipitate on adding acetic acid, it is poured into 20 times its weight of ice water, when most of the condensation product is precipitated. The mother-liquor is mixed with salt and zinc chloride, and oxidised with ferric chloride or potassium bichromate. The colouring matter is purified by dissolving in water and reprecipitating. The orange-red colouring matter from *m*-amido-*p*-cresol and phthalic anhydride is obtained by heating together 1 kilo. of the latter, 2 kilos. of the former, and 8 kilos. of sulphuric acid to 160° C. for several hours. The melt is poured into 100 litres of water, the whole boiled up and filtered, when the greater part of the colouring matter crystallises out on cooling, the remainder being precipitated with salt.

—T. A. L.

*The Manufacture of New Leuco Compounds and the Production of Colouring Matters therefrom.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 13,394, July 10, 1893.

THIS specification describes another method of obtaining the dyestuffs referred to in Eng. Pat. 11,876 of 1892 (this Journal, 1893, 595). The methods now mentioned are for

the production of the leuco derivatives of the above compounds, and these are obtained by condensing the alkylated diamido-diphenyl methanes or homologues thereof with oxazine dyestuffs, or else by condensing the alkylated diamido-benzhydrols with the leuco derivatives of the oxazine dyestuffs. The two following examples illustrate the production of these leuco derivatives. (1.) 31 kilos. of new blue R and 26 kilos. of tetra-methyl-diamido-diphenylmethane are dissolved in 250 kilos. of alcohol and kept at 80° C. for several hours. The leuco compound produced can be precipitated by adding water. (2.) The same quantities of new blue R and alcohol are taken, and zinc dust and hydrochloric acid are added until the solution is decolourised. The filtered solution is then mixed with 27 kilos. of tetra-methyl-diamido-benzhydrol, and heated at 70°–80° C. for 12 hours, when the reaction is complete, and the leuco compound is precipitated by water. The leuco compound produced according to either of the above examples is mixed with 50 kilos. of acetic acid and 125 kilos. of a 20 per cent. lead peroxide paste. Sulphuric acid is then added to precipitate the lead, and after filtering off the lead sulphate the filtrate is mixed with 25 kilos. of zinc chloride and sufficient salt to precipitate the dyestuff, which can be purified by dissolving and reprecipitating it. The colouring matter thus obtained is found to be identical with that described in the first example of Eng. Pat. 11,876 of 1892. By using twice the quantity of lead peroxide paste and working the product up as before, a product is obtained which is identical with that mentioned in example 2 of the before-mentioned specification.

—T. A. L.

*Manufacture of Colouring Matters by Reduction of Tetra-nitro-anthrachrysone and Dinitro-anthrachrysone-disulphonic Acid.* O. Inray, London. From "The Farbwerke vormals Meister, Lucius, and Brüning," Höchst-on-the-Maine, Germany. Eng. Pat. 13,395, July 10, 1893.

THESE colouring matters, which dye mordanted wool green and blue shades, are obtained from tetra-nitro-anthrachrysone and dinitro-anthrachrysone disulphonic acid by reduction in an alkaline or acid solution. The following substances may be employed as alkaline reducing agents:—Sodium or ammonium sulphide, glucose, sulphate of iron, and similar reagents, and the method employed is to dissolve the tetra-nitro-anthrachrysone in about 20 times its weight of water containing rather more carbonate of soda than corresponds to four hydroxyl groups. Sodium sulphide (for four nitro groups) is then added, and after standing some time the solution is warmed on the water-bath for an hour. After cooling, the colouring matter is precipitated by salt or acid and is used for dyeing as a paste. It dissolves in hot water with a blue colour, in dilute caustic soda with a violet, and in ammonia with a blue colour, and is precipitated easily from these solutions by salt or acid. The solution in concentrated sulphuric acid is violet. On chromed wool it gives deep green to blackish-green shades fast to soap and fulling. In order to carry out the reduction in an acid solution the tetra-nitro-anthrachrysone is mixed with 30–40 times its weight of diluted hydrochloric or sulphuric acid, and rather more zinc is added than is required for four nitro groups. After about 12–24 hours the colouring matter separates in flakes, and the reaction is completed by warming the melt on the water-bath, when the dyestuff is filtered off and purified by precipitating its dilute alkaline solution with salt. The colouring matter forms a greyish-blue paste, which dries to a black powder with a metallic lustre, dissolving in concentrated sulphuric acid with a brownish-red colour. On chromed wool the shades range from bluish-grey to blue, and are said to be perfectly fast to fulling, soap, and light. In a similar manner colouring matters can also be produced from dinitro-anthrachrysone disulphonic acid. These, however, are also capable of dyeing unmordanted wool from an acid bath on unmordanted wool are blue, whilst on chromed wool they are green, and on wool mordanted with alumina they are bluish-green. The colouring matter obtained by employing acid reducing agents gives pure violet shades on

unmordanted wool from an acid bath, on chromed wool pure blue shades, and on alumina mordants the shade is violet-blue. On mordanted wool the colours are fast to fulling.—T. A. L.

*Improvements relating to the Production of Amidophenol and Amidocresolsulpho-Acids, and to the Manufacture of Colouring Matters therefrom.* H. H. Lake, London. From K. Ochler, Offenbach-on-the-Maine, Germany. Eng. Pat. 13,402, July 10, 1893.

The sulphonic acids referred to are obtained by heating the disulphonic acids of aniline and of *o*- and *p*-toluidine with caustic alkalis to 200–230° C. So-called amidophenol sulphonic acid III. is obtained by heating 1 kilo. of sodium aniline *o*-disulphonate with 2 kilos. of 50 per cent. caustic soda lye to 200° C., and precipitating the cold melt with hydrochloric acid. When heated in the water-bath with sulphuric acid it is converted into the isomeric amidophenol-sulphonic acid IV. By fusing 12 kilos. of the acid potassium salt of  $\alpha$ -aniline *m*-disulphonic acid with 36 kilos. of 50 per cent. soda lye for 7 hours to 220° C., the so-called amidophenol sulphonic acid V. is produced by precipitating the melt with hydrochloric acid. In a similar manner the sodium salt of *p*-toluidine disulphonic acid (obtained by sulphonating *p*-toluidine), when heated with twice its weight of 50 per cent. caustic soda lye for 5 hours to 210–230° C., and precipitated with hydrochloric acid, gives amidocresol-sulphonic acid. The same acid is obtained by sulphonating *p*-nitrotoluene, reducing, again sulphonating, and then heating with caustic soda lye. By treating *o*-nitrotoluene in this way an amidocresol sulphonic acid IV. is produced, which on heating with acids gives an amidocresol melting at 157° C. All these compounds will combine with diazo compounds, or the compounds themselves may be diazotised and combined with amines and phenols and their derivatives. The shades of the colouring matters obtained range from scarlet to yellow, whilst the hexazo compounds obtained by combining a diazo compound with the combination from tetrazodiphenyl and amidophenol, or amidocresol sulphonic acid and resorcinol, or *m*-phenylene diamine, gives darker shades.—T. A. L.

*Improvements relating to the Production of Colouring Matters.* A. Ashworth and J. Burger, Bury. Eng. Pat. 13,419, July 11, 1893.

The *o*-nitroso-naphthols,  $C_{10}H_6(OH)NO$  [1.2 or 2.1] or  $\alpha$ -amido- $\beta$ -naphthol, can be condensed with gallic acid or with tannin by warming with sulphuric acid, giving colouring matters which dye on chromium mordants. A solution of 18.8 kilos. of crystallised or 17 kilos. of dried gallic acid or 32 kilos. of tannin in 200 kilos. of sulphuric acid of 66° B. has gradually added to it 17.3 kilos. of  $\alpha$ -hydroxy- $\beta$ -nitroso-naphthalene or  $\beta$ -hydroxy- $\alpha$ -nitroso-naphthalene, the temperature being kept below 25° C. When all is dissolved, the temperature is raised to 40° C. for an hour, then in another hour to 60–70° C., and finally for two hours to 85–90° C. The melt is then cooled down, poured on to ice, and the colouring matter, which precipitates in flakes, is filtered off. When dry it forms a black powder, sparingly soluble in cold water and alcohol, soluble in hot water, and very slightly soluble in ether. The colour in dilute caustic soda is brown, and purplish-brown in sulphuric acid, giving brown flakes on dilution. The colouring matters are used preferably in the form of paste, and when dyed on chromium mordants give brown shades fast to light and soap.

—T. A. L.

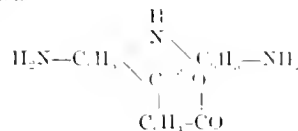
*Manufacture of Mordant-dyeing Colouring Matters.* C. D. Abel, London. From "The Actien Gesellschaft für Anilin Fabrikation," Berlin, Germany. Eng. Pat. 13,833, July 17, 1893.

The colouring matters described are azo-dyestuffs derived from  $\alpha$ -amido- $\beta$ -naphthol and the two sulphonic acids of the same,  $C_{10}H_5NH_2 \cdot OH \cdot SO_3H$  [1.2.6] and [1.2.7], which contain a hydroxyl and an amido group in the ortho position. The colouring matters obtained by combining diazo and tetrazo compounds with these substances dye wool mordanted

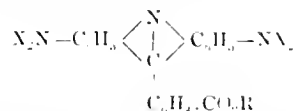
with a chromium salt in shades fast to fulling and light. The diazo compound from 9 kilos. of sulphamic acid is combined with 8 kilos. of 1.2 amido-naphthol dissolved in dilute acetic acid. The colouring matter separates as a blackish-green crystalline precipitate, which dyes wool mordanted with chromium salts a blackish-violet. The diazo solution from 21 kilos. of *p*-nitraniline, in which the free acid has been neutralised by the addition of sodium acetate, is added to a cold solution of 11 kilos. of 1.2.6-amido-naphthol sodium sulphamate. The colouring matter forms a brownish-violet powder and dyes chromium-mordanted wool a deep violet-blue shade. Tetrazo-diphenyl or Triolyl may also be combined with the substances referred to, and in this way mixed diazo compounds may be obtained. In this case, if amido-naphthol be one of the components, either the other component must contain sulphonic or carboxylic acid groups, or the *p*-ol amine must be a sulphonic acid, such as tetrazo-benzene-disulphonic acid, in order that the final product may be sufficiently soluble in water.—T. A. L.

*The Manufacture and Production of New Basic Colouring Matters.* J. Y. Johnson, London. From "The Badische Anilin and Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 14,014, July 19, 1893.

These dyestuffs are acridine derivatives, and are obtained by alkylating the compound formed by treating fluorescein with ammonia having the empirical formula  $C_{12}H_8N_2O_2$  and the constitution—



On alkylation, by heating with an alcohol and hydrochloric or sulphuric acid at about 100° C., or by carefully treating with an alkyl halogen compound, there results a product of the general formula—



where X represents either hydrogen or an alkyl group, and R is an alkyl group. The bodies obtained are yellow and orange dyes, and the following quantities are given for the production of a dye-stuff which much resembles phosphine. About 1 kilo. of the compound  $C_{20}H_{13}N_3O_2$  is mixed with 10 kilos. of 96 per cent. ethyl alcohol and heated under an inverted condenser on the water-bath, a slow current of hydrochloric acid gas being passed through the liquid until no further production of the dye-stuff takes place. The alcohol and hydrochloric acid are then distilled off on the water-bath and the residue is dissolved in boiling water. After filtering, the dyestuff is precipitated with salt, and is purified by repeating this treatment. The dyestuff is an orange-coloured powder soluble in water and alcohol, giving reddish-yellow solutions having a yellowish-green fluorescence. An autoclave may be used instead of an upright condenser, and by carrying the alkylation further, redder products are obtained corresponding to an alkylation in the amido groups.—T. A. L.

*Improvements in the Manufacture or Production of Colouring Matters derived from Anthraquinone.* H. E. Newton, London. From "The Farbwerke vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 11,315, July 25, 1893.

By the action of sulphuric acid on di-*o*-nitroanthraquinone no valuable dyestuffs have hitherto been obtained, but by the addition of boric acid to the melt, colouring matters which give brilliant shades are formed. It is assumed that the hydroxyl groups in the hydroxyanthraquinone derivatives produced, form boric ethers which are stable in

presence of concentrated or fuming sulphuric acid, even at high temperatures. The following example illustrates the method employed:—A mixture of 10 kilos. of di-*o*-nitro-anthraquinone, 10 kilos. of crystallised boric acid, and 200 to 300 kilos. of sulphuric acid of 66° B. (containing 92 to 93 per cent. of pure sulphuric acid), is heated in an enamelled pan with constant agitation. At about 230° C. a reaction sets in and the temperature rises to 250° or 260° C. The melt is then allowed to cool, poured into 3,000 litres of cold water, boiled up, filtered, and washed free from acid. The dye-stuff so formed consists principally of the hexahydroxyanthraquinone described in Eng. Pat. 17,712 of 1890 (this Journal, 1891, 917), together with a small quantity of a compound containing nitrogen similar to that formed by the action of ammonia on hexahydroxyanthraquinone, as described in Eng. Pat. 8702 of 1891 (this Journal, 1892, 514). It also contains small quantities of the alizarin hexacyanine of Eng. Pat. 4871 of 1891 (this Journal, 1892, 513). On wool mordanted with chromium salts the dyestuff produces intense blue shades. The proportions of the different components vary according to the strength of the sulphuric acid employed: for instance, by using a weaker sulphuric acid than that mentioned above, the product consists almost entirely of hexahydroxyanthraquinone. The same process may also be applied to the isomeric dinitroanthraquinones, or to the crude mixture of nitroanthraquinones obtained by nitrating anthraquinone or anthracene, or the sulphonic acids of these nitroanthraquinones may be similarly treated.—T. A. L.

*The Manufacture and Production of a New Amido Sulpho Acid and of New Azo Dyes therefrom.* J. Y. Johnson, London. From "The Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 14,678, July 31, 1893.

THE new acid is *m*-phenylene diamine disulphonic acid obtained by the action of fuming sulphuric acid on *m*-phenylenediamine or one of its salts, sufficient acid being taken that at least two molecular proportions of  $\text{SO}_3$  are present for every one molecular proportion of *m*-phenylenediamine. The sulphonation is complete when on combining a sample with diazotised primuline in an alkaline solution an orange-yellow dyestuff for cotton is produced. The presence of *m*-phenylenediamine or of the monosulphonic acid produces a much browner shade. The quantities employed are as follows:—One kilo. of *m*-phenylenediamine hydrochloride is mixed with 5 kilos. of fuming sulphuric acid containing 40 per cent. of free anhydride, and the temperature is gradually raised to 120° C. until the reaction is complete as described above. The melt is then poured into water, limed, filtered, and converted into the sodium salt, or the free acid may be separated by concentrating the solution of the calcium or sodium salt and adding an excess of strong hydrochloric acid. By combining the acid with diazobenzene chloride in presence of sodium acetate a colouring matter is obtained which dyes wool yellow from an acid bath. An orange-yellow colouring matter for unmordanted cotton is produced by diazotising 112 kilos. of primuline with 16 kilos. of sodium nitrite and 140 kilos. of 30 per cent. hydrochloric acid, and combining this with an ice-cold solution containing 55 kilos. of *m*-phenylenediamine-disulphonic acid and 112 kilos. of calcined soda in 700 litres of water. The whole is agitated for 24 hours, and the colouring matter precipitated with salt, filter-pressed, and dried.—T. A. L.

*Improvements in the Manufacture of Colouring Matters.* C. D. Abel, London. From "The Actien Gesellschaft für Anilin Fabrikation," Berlin, Germany. Eng. Pat. 14,895, August 3, 1893.

DIRECT-dyeing colouring matters for unmordanted cotton giving very dark shades from a single bath, without the necessity for diazotisation and combination on the fibre, are obtained by combining one molecular proportion of a tetrazo compound with one molecular proportion of the amido-naphthol disulphonic acid of Eng. Pat. 15,175 of

1889 (this Journal, 1890, 854), rediazotising the intermediate compound thus formed, and combining it with two molecular proportions of *m*-tolylene diamine, *m*-phenylene diamine or resorcinol. With regard to the quantities employed, 21.2 kilos. of tolidine are converted into a tetrazoditolyl salt and combined with a solution containing 32 kilos. of amido-naphthol disulphonic acid and 30 kilos. of sodium carbonate. When the formation of the intermediate compound is complete, the product is diazotised by adding 90 kilos. of hydrochloric acid of 20° B. and 7 kilos. of sodium nitrite. The new tetrazo compound thus produced is run into a solution containing 30 kilos. of *m*-tolylene diamine and 30 kilos. of sodium carbonate. After agitating for some time the solution is boiled, the colouring matter salted out, filter-pressed, and dried. It dyes unmordanted cotton a deep black from a soap bath, fast to acids and alkalis.—T. A. L.

*Improvements in the Manufacture of Colouring Matters.* H. H. Lake, London. From A. Leonhardt and Co., Mühlheim-on-the-Maine, Germany. Eng. Pat. 15,080, August 5, 1893.

GREENISH to reddish-blue basic colouring matters may be obtained from *m*-amidoeresol ( $\text{CH}_3 : \text{NH}_2 : \text{OH} = 1 : 2 : 4$ ) or its mono-alkylated derivatives—(1) by reacting with the nitroso or azo derivatives of dialkylated *m*-amidophenol or *m*-amidoeresol on the amidoeresols; (2) by converting the amidoeresols into azo compounds and combining these products with amidoeresols or aromatic amines; and (3) by condensing the amidoeresols with the *p*-nitroso derivatives of aromatic amines, with dichlorimides, with aromatic *p*-diamines or with *p*-amido-azo compounds. The process generally consists in heating the substances together in a medium, such as spirit or acetic acid, and precipitating the colouring matter by means of salt and zinc chloride. For instance, a mixture of 6 kilos. of amidoeresol, 16 kilos. of nitroso-dimethyl-*m*-amidoeresol hydrochloride, and 40 litres of spirit, is heated under a condenser until the nitroso compound has disappeared. The melt is then diluted with 200 litres of hot water, any impurities present are precipitated by adding 7 kilos. of sodium acetate, and, after filtering, the colouring matter, which dyes mordanted cotton blue, is precipitated by adding salt and zinc chloride. As an example of the second method, 6 kilos. of benzene-azo-ethylamidoeresol, 2.5 kilos. of *m*-amido-dimethyl-*p*-toluidine, 4 litres of 30 per cent. hydrochloric acid, and 15 litres of spirit, are heated under an inverted condenser for 24 hours until a sample diluted with water shows the formation of a bluish-violet colouring matter. After dilution with 200 litres of boiling water and addition of 3 kilos. of sodium acetate, the colouring matter is precipitated from the filtered solution with zinc chloride and salt. The process described in Eng. Pat. 13,565 of 1890 (this Journal, 1891, 760) for the preparation of colouring matters from dialkylated amidoeresols may also be applied by substituting for them amidoeresol or mono-alkylated amidoeresol. The following example illustrates the method for obtaining a colouring matter by oxidising a *p*-diamine together with *m*-amidoeresol or its alkyl derivative according to this invention. A solution of 6 kilos. of amidoeresol, 20 kilos. of sodium acetate, and 10 kilos. of *p*-tolylene diamine hydrochloride in 500 litres of boiling water, is gradually oxidised by adding 140 litres of a 15 per cent. ferric chloride solution. After cooling, the liquid is filtered and the colouring matter precipitated as before. It dyes mordanted cotton violet-blue.—T. A. L.

*Improvements in Colouring Matters.* R. G. Williams, Heywood, Manchester. Eng. Pat. 15,509, August 15, 1893.

THIS is an improved method of obtaining colouring matters similar to those produced according to Eng. Pat. 1593 of 1888 (this Journal, 1889, 279). In the present process one molecular proportion of *m*-phenylene diamine or one of its homologues is mixed with two molecular proportions of

sodium nitrite and one molecular proportion of a phenol, resorcinol, or a naphthol, and dilute hydrochloric acid is run into the solution at a low temperature. The colouring matters dye unmordanted cotton from an alkaline or neutral bath. The dyestuff from *m*-phenylene diamine and resorcinol is produced as follows:—A solution of 10.8 lb. of *m*-phenylene diamine, 60 lb. of caustic soda, 14 lb. of sodium nitrite, and 11 lb. of resorcinol in 35 galls. of water is cooled with ice to about 5° C., and dilute hydrochloric acid is run in slowly until the reaction is complete, caustic soda being added from time to time if necessary so as to keep the solution "strongly alkaline during the whole operation." An excess of hydrochloric acid is then added to precipitate the colour, which is filtered off, washed, and dried at a low temperature with sufficient caustic alkali to render it soluble in water. It dyes mordanted cotton brown from an alkaline bath with or without the addition of salt.

—T. A. L.

*Manufacture of Colouring Matters applicable to Unmordanted Fibres or Fabrics.* J. Murray, London. From "La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis," Paris, France. Eng. Pat. 23,578, December 7, 1893.

BLACK, blue-black, and blackish-green colouring matters for unmordanted cotton are obtained by heating certain aromatic diamines, amido phenols, and other bodies with sulphur and caustic soda. The colouring matters are divided into two series, to the first of which belong those derived from *p*-phenylene diamine, *p*-amido-dimethylaniline, Bernthsen's mercaptan (Annalen, 251, 23), azophenine, and indamines, amido-azo-benzene, and generally azo compounds which, on reduction, give a *p*-diamine. The colouring matters of this series are very soluble in water, but insoluble in acids, and dye yellowish or greenish-black shades. Precipitated by acid, they are only slightly soluble in cold alkalis, but are easily soluble in sodium sulphite, and dye best from this solution, and on exposure to air or treatment with an oxidising agent the colour develops on the fibre. The colouring matters of the second series, which are obtained by the action of sulphur and caustic soda on *o*- or *p*-amido-phenol, oxy-azo-benzene, azoxybenzene, and azo colouring matters which, on reduction, give *o*- or *p*-amido-phenol, are very soluble in water, alkalis, or alkaline sulphites, are insoluble in acids, and, like those of the first series, are best applied from a sodium sulphite solution. For obtaining a colouring matter from *p*-phenylene diamine, a mixture of 1 kilo. of the diamine, 0.5 kilo. of sulphur, and 1 kilo. of caustic soda of 40° B., is heated for 10 hours to 180°–210° C. The bronzy-black mass thus obtained is powdered, and can be used directly for dyeing. By substituting the same weight of *p*-amido-phenol for the *p*-phenylene diamine in the example just given a dark reddish-brown mass is obtained, which, after grinding, can be used for dyeing as described above.—T. A. L.

*Manufacture of Colouring Matters Dyeing Unmordanted Cotton, and their Salts and Sulpho Derivatives.* F. Petersen-Müller, Bâle, Switzerland. Eng. Pat. 8536, April 30, 1894.

VIOLET to blue colouring matters for cotton are produced according to this specification by heating rosaniline with benzidine, telidine, diamido-methylbenzidine, diamido-diphenylalkylether, diamido-hydroxydiphenylalkyl ether, or diamidostilbene, together with benzoic or acetic acid, to 130°–185° C. The rosaniline is substituted to a lower or higher degree according to the proportions of the constituents, the temperature and the length of time the melt is kept on, but in no case is a homogeneous product obtained, although by suitably varying the conditions the product may consist principally of the component desired. Thus, in order to obtain a substituted rosaniline containing three diphenyl rests, 10 kilos. of rosaniline are heated with 100 kilos. of benzidine and 2 kilos. of benzoic acid for two hours to 150°–185° C. The melt is then extracted with dilute hydrochloric acid, and the colouring matter precipitated as hydrochloride by salt. It forms a brown powder, and

dyes unmordanted cotton blue from a slightly acid bath. In place of the benzidine, other diamines may be employed, or a mixture of these with aromatic monamines such as aniline or toluidine, or with diamines such as *o*, *m*, or *p*-phenylene diamine, tolylene diamine, or a triamine such as triaminobenzene. On treatment with sulphuric acid or other sulphonating agents, mono-, di-, and tri-sulphonic acids of the colouring matters can be obtained. —T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

### PATENTS.

*An Improved Paste or Size for Use in the Pasting of Cap Bottoms and the Sizing of Cotton Goods.* W. A. Brown, Manchester. Eng. Pat. 12,345, June 23, 1893.

The patentee employs for above purpose a mixture of starch or farina, pulverised soap, and sugar. These ingredients are taken in the following proportions:—Farina or starch, 16½ ozs.; soap, 1 oz., and sugar, 1 oz.; and dissolved in two gallons of water. After being boiled, the paste is ready for use.—H. S.

*Improvements in or connected with the Producing or Printing of Designs upon Waterproof Fabrics.* J. Frankenburg, Salford, and C. O. Weber, Manchester. Eng. Pat. 12,367, June 23, 1893.

POWDERED alpha-oxy-cellulose is applied to the adhesive rubber surface of waterproof cloth by passing the latter under a gauge, in front of which the dry powder is placed. In this manner an even film is obtained, which, owing to the great affinity of alpha-oxy-cellulose for dyes is printed upon like an ordinary calico. Finally the material is thinly coated with a rubber solution, and the whole vulcanised.

—H. S.

*Improvements in the Manufacture of Flax or other Vegetable Fibre Roofing.* G. McTear, Belfast. Eng. Pat. 14,551, July 28, 1893.

See under IX., page 737.

*Improvements in or relating to the "Retting" of Fibres.* J. C. Pennington and W. O. Allison, New York. Eng. Pat. 22,808, November 28, 1893.

ACCORDING to the patentee the "retting" of flax and other fibrous materials is a partially putrefactive fermentation brought about by the agency of microbes, which destroy the gum and extractive matters. He therefore adds to the retting liquid, tepid water containing pchizomyces microbes and such soft salts and oxides as shall furnish alimentation to the microbes, and promote their growth and multiplication. It is stated that by this treatment flax straw may be retted thoroughly in from 36 to 48 hours.

—H. S.

*A New or Improved Process for Rendering Textile Materials Waterproof and Flame-proof.* H. Rous and G. N. West, London. Eng. Pat. 5718, March 19, 1894.

Two solutions are prepared, one of sugar of lead (½ lb. to 1 gall. of tepid water), and the other of alum (¼ lb. to 1 gall. of tepid water). Both solutions are allowed to stand for 6 hours, and are then mixed together, well stirred, and allowed to stand for 36 hours. As a precipitate results, the liquid is drawn off, and the material to be treated soaked in this liquid for 48 hours. Then the fabric is dried, and afterwards immersed for 36 hours in a solution of tungstate of soda and bromide of potassium (½ lb. of the former and ¼ lb. of the latter to every gallon of water). Finally the material is steamed, dried, submitted to hydraulic pressure, and finished.—H. S.

*An Improved Process for Producing Artificial Whalebone, and Method for forming the same into Strips.* G. Fehrenbach and L. Prud'hon, Paris. Eng. Pat. 6221, March 27, 1894.

HITHERTO the caustic baths employed in the manufacture of artificial whalebone from hair, bristles, or wool have been composed of a mixture of caustic lime and caustic potash. It is proposed to omit the lime, using either caustic potash or caustic soda of a concentration determined by the conditions of temperature and duration of steeping. The hair or other material which has been causticised in this bath is next steeped in very dilute acid, whereupon "the proteinous matter appears and remains adhering to each hair." The material is then washed, the fibres are arranged parallel, and in piles to a given thickness, and submitted to hydraulic pressure whilst hot. The strips thus obtained are laminated, dried, polished, and cut to size.—A. G. B.

## VI.—DYEING, CALICO PRINTING, PAPER-STAINING, AND BLEACHING.

*On the Nature of the Forces which play a part in the Process of Dyeing.* M. A. Rosensiehl. Bull. Soc. Chim. 1894, 11—12, 44—48.

THE author (like others before him) shows, on the basis of well-known facts and simple observations, that the supporters of both the mechanical and chemical theories of dyeing err in their one-sidedness, and he defines the process of dyeing as an operation consisting in covering the textile fibre with a perfectly adherent layer of a coloured substance, this layer being sometimes a compound of a part of the substance of the fibre with a colouring matter.—F. M.

*New Dying Process.* Presented in Competition for a Prize of the Society by M. Bonnet, with report on the same by E. Kopp, E. Noelting, and E. Grandmougin. Bull. Soc. Ind. Mulhouse, 1894, 82—99.

FOUR new processes are described, followed by a report on the correctness and practical utility of the author's observations, the report being based on numerous and detailed experiments carried out by MM. Kopp, Noelting, and Grandmougin.

A. New method of producing manganese brown, and its application in the production of aniline black. The property possessed by tannin of reducing permanganate is made use of. Cotton is boiled in a tannin solution containing at least 60 grms. per litre, or in a decoction of elina galls at 5°—6° B. Allow the cotton to cool in the bath, fix with tartar emetic, and dye in permanganate solution containing not less than 5 grms. per litre. The method may also be applied in printing, the tannin being printed on the goods, which are subsequently passed through permanganate.

Aniline black is produced on cotton dyed as above, by successive treatment with aniline and bichromate according to well-known processes. Blacks may be dyed on mixed silk and cotton goods by making use of the fact that treatment with tannin prevents the formation of manganese brown on silk. By adjusting the amounts of tannin and permanganate used previous to the aniline bath, it is possible to obtain a black on the cotton only, on the silk only, or on both fibres alike.

*Report on above.*—The only novelty in the author's process is the use of tannin: as an almost identical process, in which Cachou de Laval was employed, was in use some years ago. The defects of the process—leaving out of the question the price of tannin and of permanganate—are rapid decomposition of the permanganate bath, difficulty of washing, attacking of wooden vessels, lack of brilliancy and evenness in the aniline black. In printing it is difficult to

produce a discharge on account of the large quantities of tannin used. Clear whites are only obtained by the use of sodium bisulphite, and this injuriously affects the black.

Experiments made with wool show that it behaves almost exactly as silk: however, on chlorinated wool dark shades can be produced with tannin and permanganate.

B. New method of mordanting with lead, and its application for chrome yellow, &c. The mordant used is potassium plumbate, made by boiling lead dioxide with concentrated caustic potash. It is applied by alternately passing the cotton through plumbate solution, and fixing by washing in running water, the operations being repeated several times. For chrome-yellow, mordant in plumbate solution (100 grms. per litre) 15 minutes at 50° C., then pass into bichromate. For brown or black, mordant with plumbate, dye with ammonium sulphide; a black may be thus produced which is fast to soap and weak acids. For mixed silk and cotton fabrics, mordant with plumbate; the cotton only is affected, and on treating with bichromate is dyed yellow, while the silk remains white and can be shaded at will with coal-tar colours. On mordanting with plumbate and passing through permanganate, oxide of lead is fixed on the cotton and oxide of manganese on the silk.

*Report.*—The plumbate may be prepared as follows:—150 grms. caustic potash are dissolved in 125 cc. water, the solution boiled gently, and 130 grms. lead dioxide gradually added. A greyish crystalline deposit of potassium plumbate is formed, which on cooling appears as a solid deliquescent mass, 340 grms. of which are obtained from the quantities given. For use it is dissolved in dilute caustic soda; the resulting solution stands at 31° B., or can be further diluted to 19°, 10°, or 5° B. Its use depends on the fact that on dilution lead dioxide separates out. One minute's immersion in boiling water is a better way of effecting this than the author's method of washing in running water.

The treatment with plumbate is carried out cold, owing to the alkalinity of the liquid: even then the fibre is mercerised if the solution is strong. This mordant has no advantage over other lead compounds for chrome yellow or for sulphide browns and blacks, nor does it show any advantage over permanganate in preparing cotton for aniline black.

A reaction with tannin is worthy of notice and very delicate. 4 cc. of a 0.1 per cent. solution of tannin give a distinct currant-red coloration with plumbate at 31° B. The colour becomes brown on standing.

C. Fixation of lead oxide by alkaline plumbites. Litharge or massicot is dissolved in excess of boiling caustic soda at 26° B., the solution diluted as required, the fibre steeped in the solution and fixed in running water.

*Applications.*—(1.) Mineral colours.—Sulphide browns and blacks, mordant as above, develop in sodium sulphide solution. Chrome yellow: mordant as above, treat with bichromate cold. Metallic oxides: oxides of iron, copper, or manganese are deposited by passing through solutions of salts of the metals.

(2.) Dyewoods.—The black with logwood is worth notice.

(3.) Cachou de Laval and tannin yield very dark shades.

(4.) Artificial colours.—Alizarin gives a violet, naphthol brown a mahogany brown, &c.

(5.) Aniline black.—Mordant with plumbite, chrome, dye with aniline, chrome again. Or mordant with plumbite, pass into manganese chloride, then into permanganate, dye with aniline, chrome. Or pass successively through plumbite, tannin, or cachou de Laval boiling, permanganate, aniline, and bichromate.

(6.) Printing with plumbite.—The plumbite may be discharged with oxalic or nitric acid.

*Report.*—The plumbite is best prepared from lead acetate. To 130 grms. lead acetate dissolved in 2,000 cc. water, are added 1,700 cc. caustic soda at 38° B. heated to 60° C. On adding to this 6,300 grms. water, 10 litres of a solution at 12° B. are obtained. To mordant, pass twice through this solution cold, and after 12 hours wash well with water.



The plumbite is better fixed by cold running water than by immersion in boiling water. It is not readily precipitated on dilution.

**Applications.**—(1.) Mineral colours. — The sulphide colours are fast to soap, but lacking in richness. For chrome yellow use bichromate at 100 grms. per litre. The fixation of metallic oxides offers no point of interest.

(2.) As mordant for dyestuffs. — The shades produced are of little value and fugitive to light. With cochineal a violet appears to be produced, and not a red, as stated by the author. The formation of a violet with alizarin is also doubtful. Basic coal-tar colours can be dyed on cotton prepared with plumbite and tannin, but the method offers no advantage, and the colours are blackened by sulphuretted hydrogen.

(3.) Aniline black. — The plumbite is not practically useful.

**D. Method of obtaining two different shades on mixed fabrics of silk and wool.** Mordant boiling with bichromate; the wool only is mordanted, and acquires the property of oxidising aromatic amines, thus becoming dyed, while the silk remains white, and can be afterwards dyed with suitable coal-tar colours.

**Report.** — In order to apply this process for aniline black, 50 per cent. of bichromate must be used and the mordanting repeated three or four times. Mordant boiling, and dye cold with nitrates of amines, *e.g.*, aniline, ortho- or para-toluidine,  $\alpha$ -naphthylamine, &c. Repeat two or three times. By dyeing cold we obtain the minimum of colour on the silk, but even in this case a good white on the silk does not result. The method can, however, be applied in connection with phenolic colouring matters. Mordant with 3 per cent. bichromate, boiling, dye with anthracene brown, chromotrop, &c. The wool only is dyed. The process is of little practical interest. — R. B. B.

#### *An Application of Silicate of Soda.* G. Geisenheimer. *Comptes rend.* 118 (1894), 192–194.

HOWEVER pure may be the chemicals employed in bleaching linen, one is never sure of getting it perfectly clean, even if the soda crystals are quite white, the soap rich in fatty matters, and the caustic soda free from sulphides and silicates. Often the linen remains reddish, or is covered with large yellow spots, or retains all its original stains. To remove these blemishes the bleacher increases the quantity of caustic or the time of boiling, and even if he succeeds in his endeavours the linen is damaged in the process. These mishaps are usually attributed to the chemicals, whereas they should be attributed to the nature of the water used. Some waters are well spoken of, whereas others are rejected as unsuitable for bleaching. This distinction ought to disappear. The only salts which can usually exercise any effect are those of calcium and magnesium. On boiling, or on addition of soda, calcium bicarbonate forms a precipitate which adheres to the linen; acting as a lake, it fixes in the tissues the yellowish colour of the alkaline solution, and the stains thus produced are not removed by subsequent washings or by oxidising agents. Calcium sulphate acts in a similar way.

When distilled water is used, or water which has been simply purified, these mishaps do not occur; and a chalky water can be rendered harmless by adding to it an alkaline silicate. The silicate must be completely soluble in water, and can be prepared by mixing powdered anhydrous carbonate of soda with 15 or 20 per cent. of a saturated solution of silicate of soda  $\text{NaO}(\text{SiO}_2)_2$ . The salt thus prepared is easily transported and retains its solubility, whereas no silicate obtained directly by fusion is wholly soluble. When added to the lye the silicate produces with the salts of lime and magnesia a flocculent precipitate which settles quickly, is not at all adhesive, and which on boiling becomes as powdery as sand. Experience shows that when the water has been purified in this way very little caustic soda is required. The method here recommended is in accordance with the practice, frequently employed, of using wood-ashes in

bleaching, for when the extract from such ashes is analysed it is found to contain only carbonate and silicate of soda or of potash. — D. E. J.

#### *Discharges and Resists on Silk.* H. Knochlin. *Bull. Soc. Ind. Mulhouse*, 1894, 107–111.

**Fat Resist** consists of resin, pitch, yellow wax, stearin, and rectified petroleum. The fabric, after printing, is dyed in the cold with a coal-tar colour and passed through benzine to dissolve the resin, after which it may be steamed to better fix the colour. For black, mordant the printed pieces with ferric nitrosulphate and dye with logwood. The mordants should always be applied after printing the resist, *e.g.*, a deep blue is obtained as follows:—After printing the resist, work in pyrolignite of iron, pass through chalk water, and dye hot with alizarin blue in alkaline bath, wash, and soap.

**Tin and Zinc Resists and Discharges.** Print with acetate of tin or hydrosulphide of zinc; the resist may be coloured with methylene-blue, phosphine, safranin, &c. Then dye with a "direct cotton" colouring matter. Or dye with the direct cotton colour, *e.g.*, diamine-blue, and print the tin or zinc compound with thickening as a discharge.

**Tartar Emetic Resists under Tannin Colours.**—The resist may contain tartar emetic, gum, oxide of tin, magnesium acetate, zinc sulphate, acetic acid, and a colouring matter if required. After printing this, apply by means of a roller a tannin colour; *e.g.*, for blue use a basic violet and malachite green with tannin, gum, acetic, and tartaric acids; steam and wash. The tannin colours are fixed on silk readily by steaming, but not by dyeing.

**Discharges on Aniline Black.**—Prepare with antimony tannate, pass through aniline ferrocyamide and potassium chlorate, and print acetate of soda and colouring matter, steam for half an hour, treat with weak bichromate, and wash.

**Chromic Acid Discharge on Indigo.**—Applied to silk, this process, which consists in printing on indigo a colour together with bichromate, and passing into oxalic and sulphuric acids, does not give a white, but a light brown; dilute boiling sodium bisulphite serves, however, to destroy this brown colour.

**Resists under Vat Blue.**—Only employed in England. Copper resists are printed on to the silk, which is dyed in the indigo vat and exposed to air to re-oxidise the indigo.

—R. B. B.

#### *The Formation of Resorcinol-green on Cotton, Wool, and Silk.* M. Pokorny. *Bull. Soc. Ind. Mulhouse*, 1894, 116–119.

RESORCINOL-GREEN is usually fixed by dyeing on an iron mordant. It can also be formed in the four following ways:—

I.—Mix together resorcinol and an iron salt, steep the fibre in the solution, and then pass into an acidified solution of sodium nitrite, wash, and dry. Ferric salts are better than ferrous, and the best proportions are 1 molecule of resorcinol to  $\frac{1}{2}$  molecule ferric chloride.

II.—In the above process part of the green colour remains in the nitrite bath, and in this bath mordanted wool can be dyed. A soluble iron salt of dinitrosoresorcinol appears to be formed, somewhat resembling Naphthol-green B (Cassella); the latter, however, contains the  $\text{SO}_2\text{H}$  group.

To prepare the colouring matter take 1 mol. resorcinol,  $\frac{1}{2}$  mol. ferrous sulphate, 2 mols. sodium nitrite, and 2 mols. acetic or hydrochloric acid. A green solution is obtained, and a precipitate which dissolves on prolonged boiling with water. Common salt precipitates the colouring matter from its aqueous solution. This colouring matter dyes mordanted wool and silk, and the bath may be completely exhausted. Copper sulphate completely precipitates the colour from its solution. The dyed wool treated with hot copper sulphate solution acquires a much deeper shade. The resorcinol may be replaced by naphthol, phenol, &c.



111.—A soluble green colouring matter can also be prepared by mixing dinitrosoresorcinol with ferrous or ferric salts under certain conditions. Here also the proportion 1 mol. ferrous sulphate to 1 mol. dinitrosoresorcinol is the best. A precipitate is formed which dissolves on heating with water for some time, the solution having a more bluish shade than 1. or 11. Ferric salts may replace the ferrous. Mordanted wool is readily dyed in the solution.

In all the above methods the iron compounds may be replaced by salts of cobalt, nickel, or copper.

IV.—If dinitrosoresorcinol is heated with solutions of potassium ferro- or ferricyanide a green solution is obtained in which wool can be dyed. The best proportions are  $\frac{1}{2}$  mol. potassium ferricyanide to 2 mols. dinitrosoresorcinol; e.g., 0.4 gram. ferricyanide and 3.5 grams. dinitrosoresorcinol are dissolved in 250 cc. water, and this solution on boiling forms dye-bath.—R. B. B.

#### PATENTS.

*Improvements in, or in connection with, "Hawking" Machines for Indigo Dyeing.* G. and J. Stubley, Wakefield. Eng. Pat. 9938, May 18, 1893.

In addition to the usual "nipping rollers" rotating in the vat, through which the fabric passes, the inventors have added one or more pairs of similar rollers, acting above the vat, and consequently out of the liquor. These rollers are fixed so as to clear the serays, when raised from the liquor. Suitable means are provided for adjusting the pressure between the rollers. It is claimed that there is a great saving of dye material and time by working with this arrangement.—W. P. D.

*Improvements in the Manufacture of Coloured Waterproof Fabrics.* T. Birnbaum, Bow. Eng. Pat. 11,110, June 6, 1893.

THE fabrics are coated in the usual manner with india-rubber, the coating material being tinted or coloured by any suitable method before or after application to the fabric. This coloured ground is then treated with a coating of dyed or tinted farina, the latter being obtained by impregnating farina with a solution of colouring matter with addition of mordants or the like, as required. The process is finished by fixing the farina-coating by any of the well-known vapour or liquor processes, or by a combination of both. Thus a shot effect is obtained by means of the contrast between the coloured ground and the superposed dyed farina.—H. S.

*Improvements in the Bleaching or Cleansing of Vegetable Yarns and Fabrics.* J. Riley and the Thornliebank Company, Limited, Glasgow. Eng. Pat. 11,929, June 17, 1893.

VEGETABLE yarns and fabrics are bleached and cleansed, by the simultaneous action of steam, and alkaline, or other suitable agent. The goods are carried in loops, or festoons, on transverse supports, which are caused to travel through a chamber similar in construction to the ordinary steaming apparatus. The steam fills the chamber, and at a convenient point the cleansing liquor is sprayed down on the goods, so as to thoroughly saturate them. The waste liquor may be collected at the bottom of the chamber and used again.

—W. P. D.

*Improvements relating to the Dyeing of Cotton and other Textile Fibres, and to Apparatus therefor.* H. B. Lake, London. Communicated from Diego Mattei, Genoa, Italy. Eng. Pat. 12,466, June 24, 1893.

THIS invention refers to the dyeing of fibres in the form of sliver, slub, or roving. The arrangement consists of sets of several pairs of rollers, placed above suitable receptacles for collecting the expressed dye liquor. The lower rollers have on their periphery, a rectangular groove to receive the sliver, the bottom of the groove being covered with india-rubber. Before the sliver passes through the rollers it is carried on an endless band, under a special injector, having an interior nozzle of transverse section, changing gradually

from a circular to a rectangular form. The sides of the nozzle are pierced with holes inclined to the longitudinal axis of the nozzle itself. The method of working is as follows:—The fibre to be treated enters the first injector, while the dye liquor passes through the holes in the nozzle under pressure, so as to thoroughly soak the sliver. Then after leaving the first injector it passes through the rollers, the liquor flowing into the first vat, and so on. The sliver, after leaving the last pair of pressing rollers, is dried in the usual manner. By means of suitable pumps, the waste dye liquor can be re-used after the addition of further dye-stuff.—W. P. D.

*Improvements in or relating to Dyeing Apparatus.* J. O. Obermaier, Rheinpfalz, Germany. Eng. Pat. 14,154, July 21, 1893.

THIS refers to improvements in the cop-dyeing machine described in Eng. Pat. 15,000 of 1892. The dye vessel is provided with longitudinal and cross partitions, and the latter are divided into two parts by a central vertical opening, arranged to permit the insertion of a wedge-shaped block. By this means the frames or blocks containing the cops may be wedged very tightly into the dye vessel. Or the cross partitions may be dispensed with, and the frames set closely against one another, to obtain the same effect of tight packing.—R. B. B.

*Improvements in the Means and Method of Preparing Vegetable and Animal Fibres, Fabrics, or Piece Goods for the Reception of Aniline Black.* J. Clapham and W. W. L. Lishman, Bradford, Yorks, and J. Pieard and C. Villedieu, Paris. Eng. Pat. 15,028, August 5, 1893.

THE process of preparation consists of five operations, the goods being steeped successively for a few minutes in each of the following baths:—(1.) Water at 15°–20° C., with a sulphonated or soluble oil, or soap, with or without glycerin, glucose, &c. (2.) Slightly acidulated water. (3.) Water, acid, and a manganate or permanganate, with or without an alkaline chloride, sulphate, nitrate, &c. (4.) Acidulated water. (5.) Water to wash off. The goods are now prepared for the reception of aniline black.

Bath No. 3 may be also used in connection with the dyeing of logwood black on vegetable yarns, and is best applied immediately before the logwood bath.—R. B. B.

*A New or Improved Method of Oxidising Indigo Blues and the like, and Apparatus employed therefor.* J. Grime, Busby, N.B. Eng. Pat. 5870, March 21, 1894.

A MOVABLE endless band made of cotton, metal, or other suitable web is fitted over and driven by two rollers with suitable adjustment for securing correct tension on the band. The goods, after passing through the dye vat and over a draw roller in the usual way, fall in a series of loose folds on to the endless band. This carries the cloth forward at a suitable rate. At the further end of the band, the cloth is again caught up over another draw roller, and then enters the second dye vat, and so on, until the desired shade is obtained.—W. P. D.

*Improved Process for Scouring, Cleansing, Bleaching and Disinfecting Wool and other Fibres or Fibrous Materials.* W. H. and S. F. Hughes, Liverpool. Eng. Pat. 7041, April 9, 1894.

THE material to be treated is first immersed in a hot bath of an infusion of quillain bark in order to dissolve and separate the grease and other adhering matters; then it is transferred into a second bath containing a solution of permanganate or bichromate of potash or soda, and finally passed through an aqueous solution of sulphurous acid, to which a small percentage of oxalic acid is added. The patentee states that this process is more efficient and expeditious than the usual method with alkali and soap.

—H. S.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Thirtieth Annual Report on Alkali, &c. Works, by the Chief Inspector.* Proceedings during the Year 1893, presented to the Local Government Board and to the Secretary for Scotland.

THE passing of the Amending Act in 1892 has considerably increased the work of the inspector under the Alkali, &c. Works Act. The number of works registered in England, Ireland, and Wales up to the close of 1893 was 1,016, showing an increase of 136 on the preceding year. In Scotland there were registered 127 works, concerning which a separate report is made. The number of processes under inspection gives a better indication of the wider scope of the inspectors' work. There are now 1,495 processes of manufacture under inspection, as against 1,225 in 1892, an increase of 270. Adding the 208 processes under inspection in Scotland, the total number in the United Kingdom is 1,703.

Tables are given showing the number of alkali and other works registered in each of the seven districts into which the country is divided for the purposes of inspection, and also of the number of separate processes in each district. From the first of these tables it is seen that there are 101 alkali works registered in 1893, as against 100 in 1892, and 945 other works, as against 810 in 1892. A table is also given showing the average result of all the tests of the acidity of chimney, condenser, and chamber-exit gases made during the year in each district. In all 4,824 visits to works were paid and 4,613 tests made. The tests point out that the condensation and absorption of acid vapours have been maintained at the high degree of efficiency in 1893 that has prevailed during recent years. It has only been found necessary to prosecute two works for contravention of the provisions of the Act. Two works were also prosecuted for non-registration.

**Alkali Works.**—Of the 101 works registered under this head only 58 carry on the old alkali or soda-making process. The remaining 43 are so registered because the work done in them comes within the original definition of an alkali work made use of in the Act, though no soda-making is carried on in them. The ammonia-soda process still continues to gain ground upon the Leblanc process, as shown by the following table:—

SALT DECOMPOSED IN THE LEBLANC AND AMMONIA-SODA PROCESS (INCLUDING SCOTLAND).

	1893.	1892.	1891.
	Tons.	Tons.	Tons.
Leblanc process.....	467,562	519,533	567,983
Ammonia-soda .....	349,609	501,897	278,528
Total.....	817,171	1,021,430	846,511

It is a matter of surprise that the Leblanc process, "which has flourished for more than a century, but seemed now to have received its death-blow, has during the last 18 months shown such renewed vitality that during that time five new Leblanc works have been started, all of which are now in operation."

In 1890, the salt decomposed for alkali-making in the United Kingdom reached the total of 855,029 tons. Since then the total make of alkali has steadily decreased. In 1893, however, there would have been an increase instead of a further decrease had it not been for the scarcity of coal, caused by the long-continued strike of the coal miners during the year.

The new alkali process by Mr. F. Gossage, of Widnes, mentioned in the Chief Inspector's Report for 1892 (this Journal, 1893, 675), has made some progress, but still awaits further development.

A drawing is given of a contrivance which has been devised by Mr. W. L. Renoldson, of Allhusen's works, for

distributing the water in hydrating acid condensers, but which may be of more general application for the condensation of gases. The device consists of a simple means of gauging the quantity of water flowing into the tower at any time. This is done by allowing the water first of all to flow into a small tank, out of which it runs through a number of short nozzles, each of which is fixed at a lower level than the one before it. The quantity of water flowing out of the tank can therefore be gauged by counting the number of nozzles through which it is being discharged. By altering the main supply cock, more or less water can be sent into the tank, and so regulated that the water flows out through more or fewer nozzles. From this small gauge-tank the water flows into a tumbling box below, from which it is thrown alternately on one side or the other of the tower, filling the pipes, which distribute it over every portion.

**Cement Works.**—No radical improvement has been made in the working of cement kilns to prevent the dense white vapour and diminish the foul smell which arises from them, especially in those places where clay, highly charged with organic matter, as in the Southampton district, is used.

An important step is being made at the works of the Oldbury Alkali Company in an endeavour to manufacture cement from the waste carbonate of lime from the Chance-Claus process. The cement already produced stands well all the tests usually applied, and it is to be hoped that longer experience will prove its good quality. This would offer a useful application for a very large quantity of material now entirely waste.

**The Nitric Acid Chlorine Process.**—Last year this process for manufacturing bleaching powder was undertaken in Ayrshire and in Cheshire. In Ayrshire the difficulties met with from the outset prevented the work from being fairly started, and for a time it has been abandoned. In Cheshire the attempt, under another patent, has also not yet been successful.

**Sulphate of Ammonia Works.**—No complaint has been brought during the past year against these works. Although formerly this was one of the most offensive manufactures, it is now "carried on at 368 works in England, &c., and at 59 in Scotland, and yet the public may be said to be quite ignorant of their presence." The following table shows the amount of sulphate of ammonia produced in the United Kingdom, and the sources from which it is obtained. It will be seen that the production is increasing year by year, but there has been a falling off in the quantity made in the iron and carbonising works in consequence of the stoppage of many of these works during the recent coal strike:—

AMOUNT OF SULPHATE OF AMMONIA PRODUCED IN THE UNITED KINGDOM.

	1893.	1892.	1891.
	Tons.	Tons.	Tons.
Gasworks .....	112,179	110,748	107,370
Ironworks .....	8,833	11,000	6,289
Shale works .....	28,485	23,105	26,690
Coke and carbonising works....	3,275	1,071	2,766
Total .....	152,772	144,924	143,115

**The Chance-Claus Process.**—Many improvements have been made since the process was first adopted in 1889, chiefly in details of construction. Owing to great care in the construction of the numerous cocks and pipes which form a part of the apparatus, the complaints of nuisance arising from the escape of sulphuretted hydrogen may be said to have ceased, and no accident arising from its inhalation by workpeople has been reported during the year. In all, about 35,000 tons of sulphur can be produced annually

when the whole of the plant is in operation. Last year the production was 31,350 tons. Most of the tank waste produced on the Tyne, at Oldbury, and at Wednesbury, is treated by this process. In the St. Helen's and Widnes district the plant for treating the whole of the waste made by the United Alkali Co. has been erected, but, as is stated in the report of the district inspector, Dr. Adleck, for some reason or other this company have not yet found it practicable to treat the whole of the waste made. About 200,000 tons of waste were made in their Widnes works, of which 144,000 tons, or 72 per cent., were treated by this process, and yielded 12,835 tons of sulphur and about 1,000 tons of sulphuric acid of 123 Tw. The weak point of the Chance-Claus process is still the imperfect decomposition of the sulphuretted hydrogen. The gases escaping at the final exit contain both sulphurous acid and sulphuretted hydrogen, equivalent to nearly 15 per cent. of the sulphur contained in the waste. These gases are passed through a fire or through heated flues with sufficient air to completely burn the sulphuretted hydrogen before being discharged into the atmosphere.

**Venetian-Red Works.**—In these works red oxide of iron is usually prepared by the roasting of sulphate of iron, whereby a mixture of sulphurous and sulphuric acid is discharged, which has usually been allowed to escape into the air. The number of these works now registered is 15. There is considerable difficulty in dealing with these acids, as no means may be employed that would interfere with the furnace draught. As the production of particular shades of colour is aimed at, no interference with the working of the furnace is permissible. It has been found possible to separate the sulphuric acid by cooling and condensation, and by washing with water; but the sulphurous acid is only sparingly soluble in water and is not condensed on cooling. A method proposed by the chief inspector is now on trial and appears likely to be successful. It is that of leading the mixed hot gases through a tower or chamber filled loosely with light scrap iron down through which water percolates. A solution of sulphate of iron is formed which may be evaporated to the crystallising point by the waste heat of the Venetian-red furnaces, and the crystals recovered can then be used for the further production of Venetian-red.

**Arsenic Works.**—These are of two kinds. Firstly, those in which arsenious acid is made by roasting arsenical pyrites or as a by-product in the roasting of tin and copper ores. Secondly, works in which arsenic acid is made by the oxidation of arsenious acid with nitric acid or a nitrate. Of the former class there are 35 works registered, and these are all situated in Cornwall and the western portion of Devonshire. The difficulty to be overcome in these works is the prevention of the discharge of the arsenic into the atmosphere. The employment of wash towers is only partially successful, as water does not readily take hold of finely-divided dry solid matter suspended in air. By this means also the arsenic is only transferred from the air to the water, which, passing into a neighbouring stream, might do much damage. A dry filter of brushwood, gorse, &c. has therefore been advised and in several cases adopted quite successfully. As brushwood is, however, too quickly destroyed by the acids which accompany the arsenic, some more durable material is sought for. The effect of such filters has been to arrest almost the whole of the arsenic, bringing it below one-tenth of a grain per cubic foot of the chimney smoke on the average.

In the second class of arsenic works there is no danger of the escape of arsenic, but of nitric or nitrous acid. One method of dealing with these nitrous vapours is to lead them into a sulphuric acid chamber, but an objection to this is the intermittent nature of the process. In other cases the fumes are absorbed by strong sulphuric acid in a tower packed with flints, and the nitrated sulphuric acid may be then used in the Glover tower. Another and more common method is to lead the red fumes together with steam and air into columns packed with coke. The acid is thus re-oxidised and is ready for use in a further operation. This method is so nearly perfect that the loss of nitric acid from all causes is less than 5 per cent.

**Hydrochloric Acid Works.**—These works include a number of processes by which hydrochloric acid is produced by other methods than by the decomposition of common salt in the soda manufacture. Prominent among these is a process for the recovery of hydrochloric acid from galvanisers' dippings, devised by Mr. Thomas Turner, of Birmingham. The chloride of iron liquors are allowed to flow gradually upon the hearth of a reverberatory furnace: the chloride of iron is decomposed by the heat, and the hydrochloric acid that is driven off is caught in a wash tower such as is used in alkali works.

**Fibre Separation Works.**—In these works rags consisting of mixtures of cotton and wool are treated with hydrochloric acid gas, which destroys the cotton in the fabric. By cleansing and then mechanically separating the woollen fibres a new white wool is produced, which may be compared not unfavourably with the original wool in appearance. Under the name of "shoddy" this is used in the fabric of new cloth. In the report of the East Lancashire and Yorkshire inspector, Mr. R. Forbes Carpenter, some account is given of the apparatus used in these works. In the first and oldest class of works the rags are suspended in chambers built of brick, tarred inside and out, and heated below by a stove. Hydrochloric acid gas, in proportion to the cotton to be destroyed in the rags, is turned into the closed chamber and the action is allowed to continue through the night; in the morning the chambers are draughted to the condenser by chimney suction, and the fresh air admitted at an opening in the chamber door sweeps the surplus gases before it. In the second class of works the rags are treated in revolving machines of rectangular section placed in a heated stove, the hydrochloric acid gas being introduced at the axis of the machine. Great economies of space, time, and labour are obtained by this method, but it is not suitable for every description of rags, and the old method finds more favour where the least amount of "tendering" and loss of colour are essential. Another step in advance has been to make the operation a continuous one, but this can only be carried out where the rags are uniform in quality. Another process that has recently been proposed, which is being tried experimentally, is the first departure from the usual lines to meet the question of nuisance from the escape of gaseous HCl, and at the same time bring it into use without absorption by a condenser. The surplus gases from the machine, together with the fuel gases from the stove, fired for this purpose with coke, are drawn by a fan through a second machine filled with fresh moist rags to be dried and subsequently carbonised. The second machine acts as a dry condenser; the HCl is kept gaseous by the hot stove gases, and performs its function by partly carbonising the rags in the machine. The completion of the carbonisation is afterwards much more rapid, and a saving of 20 per cent. of acid is claimed. Manufacturers have all of them been informed of the duties laid upon them by the Alkali Acts, and a few have responded promptly and willingly, but the inspector has met with a great deal of opposition and passive resistance from the majority of those engaged in this industry, arising from their profound ignorance of even elementary chemical facts.

**Tar Works.**—The inclusion of these works under the Alkali Act has added most to the work of the inspectors. 118 of these works have already been registered. In preventing nuisance during the distillation of tar the escape of noxious vapours from the ends of the still worms and from the pitch bays has been found to be the chief difficulties to overcome. The gases from the ends of the still worms contain sulphuretted hydrogen, particularly at the beginning and end of tar-distillation, and also a considerable amount of light hydrocarbon vapour which does not condense at the usual temperature of the worms. In some works the gases are passed through a bed of oxide of iron or slaked lime. In other works they are burned in a furnace fire, various methods being adopted to prevent firing back, such as reducing the diameter of the pipe as it approaches the fire, the introduction of a water seal between the still worm and the furnace, or the production of a strong draught by means of a steam jet or a fan. In the report of Mr. Ballard, the inspector for Cheshire, North Wales, and part of

Lancashire, the firing back was found to be quite prevented by the inrush and cooling effect of the air on the end of the pipe by allowing it to enter the fire with an open space all round it instead of building it in. Details of various methods of dealing with these gases are described in the reports of the inspectors. In many cases, owing to the differences in the character of the tar to be distilled, the means to prevent nuisance applicable in some cases cannot be used in others. Thus, in the case of a tar distilled in the eastern counties a carbonaceous deposit has frequently been found to form in the still; this from time to time separates suddenly from the bottom of the still, and exposes an over-heated surface which causes a violent disengagement of vapour. These eruptions would be dangerous were any obstruction offered such as would be caused by a bent pipe or closed box at the end of the still worm. The most serviceable arrangement there is the plain worm end fitted with a loose cap from which a pipe rises to lead off the noxious gases.

The second source of nuisance which has called for attention are the vapours given off during the running of the pitch. The usual custom in tar works now is to distil the tar until "hard" pitch is obtained. It is then mixed with a proportion of creosote oils or waste anthracene oils so as to make it into "medium soft" or "soft" pitch, as required. It is then run from the still into a cooling vessel, and thence it is run into the pitch bay. It would be very desirable that all pitch bays be covered in, but in some cases this is not practicable because of a deposition of a highly carbonaceous hydrocarbon which ignites on exposure to air. It is deposited under protection of a dense cloud of tar-vapour, but when this is blown away by a slight gust of wind the carbon has become ignited and has transmitted the fire to the mixture of tar-vapour and air, thus causing explosion with burst of flame. Much interesting information with regard to tar works will be found in the report of Mr. F. Napier Sutton, the inspector for the south-eastern counties. An account will also there be found of a continuous tar still, the invention of Mr. F. Lennard, of the firm of Forbes, Abbot, and Lennard. This still is capable of treating from 15,000 to 20,000 gallons of tar per 24 hours, and has been at work successfully for the past 12 months, during which time about 4,000,000 gallons of tar have been passed through it. The chief inspector has tabulated the information so far collected with regard to the number and capacity of stills used in different works, the type of pitch-cooler used, the temperature at which pitch is run, the means used for destroying still-gases, &c. These tables occupy six pages of the report.

**Felt Works.**—In these works tar is heated in large open pots and conveyed to a small tank through which an endless sheet of felt passes; on leaving the tank the felt then travels between heated rollers. This operation is carried on in a closed workroom, in which many women and lads are employed, and the atmosphere of the place is charged with tar-vapour to an extent which must be highly injurious to the operatives. It has not been found easy to confine this vapour, as there must be free access for the workmen to the tar tank and machine, but it is hoped that a satisfactory plan may soon be arrived at.

**Zinc Works.**—The complaints brought against these works for nuisance are due to the sulphurous acid driven off in roasting blende, and to vapours of oxide of zinc which escape at the nozzles of the retorts in which the zinc is smelted. As in many other metallurgic processes whereby sulphurous acid is evolved from furnaces, no effectual means of arresting it is known. The difficulty of dealing with the vapours of oxide of zinc is increased by the fact that they rise from numerous small sources and are not enclosed in a furnace or flue. An attempt has been made to collect them by draughting them away through a projecting hood of sheet iron, but these hoods reflected too much heat on the heads of the men, or if removed further they failed to collect the fumes. A partial success has been obtained by enclosing the nozzle ends behind loose iron plates, which can be removed when necessary for the working. From above this enclosure the fume is draughted away to the flues, where the greater part of it is deposited.

For treating dust-laden air of this character the chief inspector suggests the use of a dry filter of such materials as are best suited to the occasion. In the case of fumes of zinc, lead, copper, &c., light waste scrap iron, such as thin sheet clippings, waste wire &c., might be useful. A hood of 5 or 6 ft. in thickness and of considerable superficial area might be supported on iron bars placed in a brick chamber. The smoke, admitted into the cavity below, would rise up through the filtering material and thence to the chimney, and if suitable holes were left in the sides of this chamber, iron bars might from time to time be inserted so as to shake up the filtering medium and cause the lead or zinc oxide to fall into the cavity below, thus keeping the filter clear.

**Alkali, &c. Works in Scotland.**—The Amending Act of 1892 has released 11 small saltworks from the obligation to register, and three new works, viz., one Venetian-red work, one muriatic acid work, one nitrate of iron work, have been registered. There is an increase of 30 in the number of processes under inspection. The total number of works registered is 127, and the number of processes is 208. The details of the inspection of these are given in the report of Mr. W. S. Curphey, the inspector. With one unimportant exception the infractions of the Act, reported during the year, were such as occurred from temporary unavoidable causes.

A general index of the first 30 reports under the Alkali, &c. Works Regulation Act is appended to this year's report.—H. S. P.

*The Concentration of the Mother-Liquors obtained in the Manufacture of Alum.* J. Wiernik. *Zeits. angew. Chem.* 1894, [10], 289—290.

THE concentration of the mother-liquors forms an important phase in the manufacture of alum. In the first place, the excess of sulphuric acid (which acid is employed for the decomposition of the aluminous minerals—bauxite, alunite, kaolin, &c.) remains in the mother-liquors, and can, by concentrating the latter, be recovered and turned into account. Secondly, the washings of the decomposed mineral residues, as well as the washings of the crude alum, consist of more or less dilute solutions of alum (generally very dilute), and to recover the alum it is necessary to concentrate them to the point of crystallisation.

The mother-liquors and washings are mixed in a wooden cistern lined with lead, and evaporated with steam to a density of 40° Beaumé. The lyes thus concentrated are, before cooling, mixed with an additional quantity of sulphuric acid (account being taken, of course, of the amount of sulphuric acid which they already contain) and employed for the decomposition of a fresh amount of aluminous mineral.

By means of this *modus operandi* nothing, so to speak, is lost from the mother-liquors. When, however, ferriferous minerals (bauxite, alunite, and the like) are made use of, the iron accumulates in the mother-liquors, until at last these contain so much iron that they not only render a fresh quantity of alum highly impure, but hinder its crystallisation. Such ferriferous mother-liquors form a troublesome by-product which is not easily disposed of; they cannot be run into the sewer on account of the not inconsiderable amount of acid which they contain.

It might be supposed that such ferriferous lyes could be utilised as a source of iron sulphate; this is, however, impossible, since, when they are evaporated, nothing but a muddy mass is obtained, which cannot be separated from adhering mother-liquor.

By the following process it is possible to free the mother-liquors from the greater portion of the iron which they contain:—The concentration of the lyes is taken to 50° Beaumé instead of 40° Beaumé, and after turning off the current of steam, a precipitate of iron salt forms, which varies in composition according to circumstances. This precipitate quickly settles; the supernatant liquor contains considerably less iron and can be used again for crystallising purposes. The

decrease in the amount of iron with increasing concentration is shown in the following tabulated analyses :—

	Fe Grms. per Litre.
Mother liquor of 90° Beaumé .....	16.80
Same mother-liquor evaporated to 12° B. and diluted to 30° B. ....	13.20
Same mother-liquor evaporated to 15° B. and diluted to 30° B. ....	11.56
Same mother-liquor evaporated to 17° B. and diluted to 30° B. ....	8.80
Same mother-liquor evaporated to 19° B. and diluted to 30° B. ....	8.04

The composition of the precipitates above mentioned varies according as the iron exists in the mother-liquor in the ferrous or ferric condition. By evaporating completely oxidised (ferric) lyes, the precipitate is yellowish-white, crystalline, and insoluble in water. It forms hard incrustations on the steam worm and the concentrating cistern, which are very difficult to remove. A point of supreme importance, however, is that the formation of this precipitate is attended with a loss of alumina and potash (also of ammonia when "mixed alums" are manufactured). A specimen of this precipitate was withdrawn, and, after washing and drying, submitted to analysis. From the mean values of two analyses it appeared to be a basic ammonium potassium aluminium sulphate of the formula  $2\text{Fe}_2\text{O}_3 \cdot 5\text{SO}_3 \cdot \text{Al}_2(\text{SO}_4)_3 \cdot \text{K}_2\text{SO}_4 \cdot (\text{NH}_4)_2\text{SO}_4$ . The salt is not only insoluble in water, but also in acids, and is decomposed with the evolution of ammonia and separation of ferric oxide and alumina when treated with alkalis in the cold. It follows from these observations that the constituents of this salt can no longer be turned to account in the manufacture, and therefore the formation of the salt must, if possible, be avoided. With this object in view, it is advisable to keep the major portion of the iron present in the mother-liquors as ferrous salt; this is accomplished by reducing the lyes (the reduction need not be complete) in the evaporating tank. After numerous experiments, the author finds that thin chips of green poplar wood form the best and cheapest

of reducing agents. The chips are packed into a high leaden sieve, which is then lowered into the concentrating tank. Under the influence of the hot acid lyes the chips are dissolved without carbonisation (apparently forming in the first stage, oxalic acid, which then decomposes into carbonic oxide and carbon dioxide), with the simultaneous reduction of the ferric oxide to ferrous oxide. When, under these circumstances the lyes are evaporated to a density of 50° Beaumé, most of the iron separates on the bottom of the tank as a black, muddy, amorphous precipitate, also in other parts of the apparatus (on the steam worm, for example) in small black crystals; the mass is readily soluble in water. Analysis of a portion of the dried substance points to the formula  $3\text{FeSO}_4 \cdot 2\text{Fe}_2(\text{SO}_4)_3$ , thus :—

	Found.	Calculated.
	Per Cent.	Per Cent.
FeO .....	16.98	17.29
$\text{Fe}_2\text{O}_3$ .....	25.76	25.48
$\text{SO}_3$ .....	57.68	57.32

The removal of this precipitate from the evaporating tank after running off the concentrated lyes is, on account of its nature, quite easy.—A. R. L.

#### *Reactions of Acid Potassium Arsenite with Metallic Salts.* C. Reichard. Ber. 1894, 27, 1019—1036.

THE author has filled up several gaps in the known series of metallic arsenites. The salts were prepared by the interaction of acid potassium arsenite with the metallic sulphate, chloride, or nitrate; in many cases some of the acid of the salt was liberated.

The following list of arsenites epitomises the author's work :—

Composition.	Prepared from	Acid liberated.	Description.	Originally prepared by
$\text{H}_2\text{O} \cdot \text{As}_2\text{O}_3$ .....	$\text{HgNO}_3$ .....	.. ..	Yellowish, decomposed by light.	Berzelius
$2\text{CuO} \cdot \text{As}_2\text{O}_3$ .....	$\text{CuSO}_4$ .....	Sulphuric acid	Green powder	Bloxam
$2\text{CdO} \cdot \text{As}_2\text{O}_3$ .....	$\text{CdSO}_4$ .....	Sulphuric acid	White amorphous powder	..
$2\text{HgO} \cdot \text{As}_2\text{O}_3$ .....	$\text{HgCl}_2$ .....	Hydrochloric acid	Yellowish-white, decomposed by light.	Berzelius
$2\text{PbO} \cdot \text{As}_2\text{O}_3$ .....	$\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ .....	Acetic acid	White	Filhol
$3\text{Au}_2\text{O} \cdot \text{As}_2\text{O}_3$ .....	$\text{AuCl}_3$ .....	Hydrochloric acid	Purple-red powder, blackened by warming.	..
$3\text{Ag}_2\text{O} \cdot \text{As}_2\text{O}_3$ .....	$\text{AgNO}_3$ .....	Nitric acid	Yellow microscopical needles, decomposed by light.	Filhol, Bloxam, Pasteur, &c.
$3\text{PbO} \cdot \text{As}_2\text{O}_3$ .....	$2\text{PbO} \cdot \text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ .....	.. ..	White	Streng
$3\text{MgO} \cdot \text{As}_2\text{O}_3$ .....	$\text{MgSO}_4$ .....	Sulphuric acid	White powder	Stein
$3\text{ZnO} \cdot \text{As}_2\text{O}_3$ .....	$\text{ZnSO}_4$ .....	Sulphuric acid	White crystals	Bloxam
$5\text{SnO}_2 \cdot 2\text{As}_2\text{O}_3$ .....	$\text{SnCl}_4$ .....	Hydrochloric acid	Yellowish-white	Berzelius
$5\text{TiO}_2 \cdot 2\text{As}_2\text{O}_3$ .....	$\text{TiO}_2 \cdot \text{SO}_3$ .....	Sulphuric acid	White crystals	..
$\text{PtO}_2 \cdot \text{As}_2\text{O}_3$ .....	$\text{PtCl}_4$ .....	Hydrochloric acid	Bright yellow	Simon
$\text{PdO}_2 \cdot \text{As}_2\text{O}_3$ .....	$\text{PdCl}_4$ .....	Hydrochloric acid	Bright yellow	..
$\text{UO}_2 \cdot \text{As}_2\text{O}_3$ .....	$\text{UO}_2(\text{NO}_3)_2$ .....	.. ..	Bright yellow	..
$\text{Fe}_2\text{O}_3 \cdot \text{As}_2\text{O}_3$ .....	$\text{FeCl}_3$ .....	Hydrochloric acid	Rusty yellow powder	..
$\text{Al}_2\text{O}_3 \cdot \text{As}_2\text{O}_3$ .....	$\text{Al}_2(\text{SO}_4)_3$ .....	Sulphuric acid	White	Thorey
$\text{Cr}_2\text{O}_3 \cdot \text{As}_2\text{O}_3$ .....	$\text{Cr}_2(\text{SO}_4)_3$ .....	Sulphuric acid	Dark green, soluble in KOH.	..
$\text{FeO} \cdot \text{As}_2\text{O}_3$ .....	$\text{FeSO}_4$ .....	.. ..	Greenish-white, becomes brown in air.	..
$\text{BaO} \cdot \text{As}_2\text{O}_3$ .....	$\text{Ba}(\text{NO}_3)_2$ .....	.. ..	White, soluble in much water.	..
$3\text{CoO} \cdot 2\text{As}_2\text{O}_3$ .....	$\text{Co}(\text{NO}_3)_2$ .....	Nitric acid	Amethyst-coloured powder.	Girard
$3\text{NiO} \cdot 2\text{As}_2\text{O}_3$ .....	$\text{Ni}(\text{NO}_3)_2$ .....	Nitric acid	Bright green.	Girard

Composition.	Prepared from	Acid liberated.	Description.	Originally prepared by
$3\text{CaO} \cdot 2\text{As}_2\text{O}_3$ .....	$\text{CaCl}_2$ .....	Hydrochloric acid	White powder, soluble in much water	"
$3\text{SrO} \cdot 2\text{As}_2\text{O}_3$ .....	$\text{Sr}(\text{NO}_3)_2$ .....	Nitric acid	White powder, soluble in much water.	"
$3\text{MnO} \cdot 2\text{As}_2\text{O}_3$ .....	$\text{MnCl}_2$ .....	Hydrochloric acid	White, becoming red to brown in air.	Strom
$3\text{SnO} \cdot 2\text{As}_2\text{O}_3$ .....	$\text{SnCl}_2$ .....	Hydrochloric acid	Yellowish white, decomposed by acids and alkalis with separation of arsenic.	Berzelius

—A. G. B.

**Leadon Pipes in Vitriol Manufacture.** A. Lambert.  
Bull. Soc. Chim. 11, 1894, 343—351.

WHEN considerable lengths of piping are requisite for the conveyance of vitriol from one part of a factory to another, it is convenient to know what dimensions will suffice to obtain a sufficiently rapid flow without entailing the locking up of capital in leaden installations more solid and of greater magnitude than is necessary. The formulae applicable in the case of water cannot be employed in such

cases; the practical formula  $V^2 = \frac{2gh}{1+R}$  may, however, be used, where  $V$  is the velocity of the fluid,  $h$  the "head" or elevation from which it flows, and  $R$  a coefficient referring to the resistance to passage of the fluid, and composed of four parts, viz.,  $Z_1$ , applying to the resistance at the entrance of the fluid into the pipe;  $Z_2$ , to sharp angles in the pipe;  $Z_3$ , to less abrupt changes of direction; and  $Z_4$ , the resistance due to friction; so that  $R = Z_1 + Z_2 + Z_3 + Z_4$ . The value of  $Z_1$  was determined for acid at 60° B. by noting the time requisite to run the acid down through a certain difference of level in a large reservoir, using orifices respectively of 35, 40, 45, and 50 millimetres in diameter; figures varying between 0.67 and 0.70 were obtained, giving an average 0.686, or more than 10 times the value corresponding with the flow of water under the same conditions. As regards  $Z_2$ , pipes bent to sharp angles (elbows) of 60° and 90° gave respectively the average values 0.925 and 2.525, whereas where water is in question, the resistance is given by a formula of the character—

$$Z_2 = A \sin^2 \frac{\delta}{2} + B \sin^4 \frac{\delta}{2}$$

Applying this formula to sulphuric acid the equation becomes—

$$Z_2 = 2.366 \sin^2 \frac{\delta}{2} + 5.224 \sin^4 \frac{\delta}{2}$$

For curved pipes with radius of curvature 15 cm., and interior diameters 35, 40, and 45 mm. respectively, the corresponding values of  $Z_3$  were 0.814, 0.797, and 0.798. The resistance due to friction when a fluid passes along a pipe is proportionate to the diameter, so that  $Z_4$  may be put  $= \lambda \frac{1}{d}$ , whilst  $\lambda = \frac{V}{0.042001}$ , where  $V$  is the velocity.

Putting together these various valuations, tables may be calculated for various cases, based on the general formula—

$$h = (1 + Z_1 + Z_2 + \dots + \lambda \frac{1}{d}) \frac{V^2}{2g}$$

—C. R. A. W.

**Specific Gravity of Calcium Chloride Solution.** S. U. Pickering. Ber. 27, 1372—1385.

IN the course of an experimental examination of the physical properties of calcium chloride solution, the following figures were obtained (*inter alia*) as regards the

relationships between the strength, the freezing point, and the density

Specific Gravity at 17.925° C.	Percent- age of $\text{CaCl}_2$	Freezing Point.	Specific Gravity at 17.925° C.	Percent- age of $\text{CaCl}_2$	Freezing Point.
1.151778	51	-20.43	1.259339	26	-32.89
1.150676	50	-20.34	1.21918	24	-27.27
1.148459	48	-20.28	1.19901	22	-22.57
1.146238	46	-20.16	1.17910	20	-18.57
1.144097	44	-20.76	1.15923	18	-15.22
1.141770	42	-17.60	1.14016	16	-12.22
1.139189	40	-10.88	1.12139	14	-9.78
1.137242	38	+ 1.45	1.10288	12	-7.72
1.134956	36	- 9.85	1.08467	10	-5.89
1.132689	34	-23.25	1.06680	8	-4.31
1.130461	32	-29.60	1.04951	6	-3.03
1.128271	30	-48.09	1.03278	4	-1.94
1.126022	28	-39.65	1.01548	2	-0.95

—C. R. A. W.

**Spontaneous Formation of Pyruvic Acid from an Aqueous Solution of Tartaric Acid.** R. Otto. Ber. 27, 838.

AN approximately 25 per cent. solution of tartaric acid contained in a stoppered flask of colourless glass was exposed to light at a western window for a year. The solution at the end of this time was of a dark yellow colour, smelt strongly of caramel, and was formed by the phenylhydrazine reaction to contain pyruvic acid.—A. R. L.

**Spontaneous Formation of Pyruvic Acid from an Aqueous Solution of Tartaric Acid.** R. Otto. Ber. 27, 1264—1265.

AN examination of the dark-coloured solution containing pyruvic acid (last abstract) has shown that it is quite free from micro-organisms, so that the observed conversion of tartaric acid into pyruvic acid cannot, as was at first supposed, be due to the agency of such organisms.

—A. R. L.

**Formation of Aluminium Sulphide.** F. Gruy. Zeits. angew. Chem. 1894 [10], 299.

See under XI., page 742.

*Effect of Heat on Iodates and Bromates.* E. H. Cook.  
Proc. Chem. Soc. 1894 [139], 119—129.

POTASSIUM iodate is difficult to purify; repeated crystallisations from water fail to give a reliable product. The method adopted was, therefore, to crystallise from water once or twice, then to precipitate the solution by means of alcohol, collect the precipitate and recrystallise from water. The first effect of heat on the iodate is to cause the substance to become brown. This brown substance has the same composition as the original iodate, and it is formed without any alteration taking place in weight. When the heating of the iodate is continued up to the time of its ceasing to lose weight, the following changes are observed: the brown substance fuses and becomes a colourless liquid; during the fusion, iodine is evolved; after continued and regulated heating, the liquid becomes a white solid, and when thoroughly solid, ceases to lose weight; the residue is potassium iodide.

The experiments having failed to produce any oxy-compounds, it was thought that these compounds might be formed if the heating were effected under reduced pressure; but at a pressure of 70 mm. the results were precisely the same as under normal conditions.

Finally, in order to discover at what stage the chlorine known to be evolved when chlorate is heated is produced, experiments were made with that substance. It was found that the chlorine was evolved just at the same period as the iodine from the iodate, viz., during the melting of the salt. The amount formed is 0.03 per cent.

It would therefore appear that the halogen is liberated early, but that it is not actually set free until the structure of the salt is broken down—in the case of the iodate by fusion, and in that of the bromate by decrepitation (at about 180° C.).

#### PATENTS.

*Improved Still and Apparatus for the Distillation of Ammoniacal Liquor and other Fluids.* G. Pettigrew, Middlesbrough. Eng. Pat. 10,171, May 23, 1893.

ABOUT a third of the upper part of a vertical still is fitted with shallow trays, "helical in form," communicating each with that next below, by an overflow pipe into a walled receptacle, forming a water joint, so that the liquor continuously overflows on to that tray. The ammoniacal liquor is run continuously into a centrally-placed pipe at the top of the still, into the centre circumference of the first tray, from which it flows spirally outwards, and is discharged into the tray beneath, in the manner described, near the circumference, whence it flows spirally towards the centre, to descend by an overflow pipe to the centre of the next tray, and so through the series. The fluid is taken from the last tray nearly to the bottom of a compartment below, whence it passes by a siphon into a lower compartment to meet milk of lime entering in "specified quantity" by a siphon arrangement. Steam is admitted to the bottom of the still in the usual manner, and, with accompanying gases, passes upwards through hooded short pipes placed about equidistant along the circumvolutions of each tray. The bottom edges of the hoods are serrated, so as to be always covered with liquor when the still is in action, the exit of the vapour and gases being through such serrations into the liquor. The gases are led to the saturator through a suitable outlet pipe in the hood. The spent liquor is run off intermittently.—E. S.

*Improvements in Evaporating Saline Solutions.* Sir L. Bell, Middlesbrough. Eng. Pat. 13,709, "June" 14, 1893.

A STEAM boiler furnace is provided with duplicate flues, for alternate use. When one set of flues has become highly heated the products of combustion are caused to enter the other set, and the spent steam from the engine is passed through the heated flues set at liberty, from which the

steam, now superheated, is admitted beneath the salt or brine pan, and allowed exit as moist steam from the far end of the pan. Or a series of pans may be similarly heated.—E. S.

*Improvements in the Production of Nitric Acid, Caustic Alkali, and Ferric Oxide.* G. Lunge, Zürich, Switzerland, and F. Maxwell-Lyte, London. Eng. Pat. 13,654, July 13, 1893.

PURPLE ore, or other form of ferric oxide, finely powdered, is mixed with sodium nitrate in such proportion that the mass may remain porous when heated to the temperature required to effect decomposition. The proportion preferred is two parts ferric oxide to one part of sodium nitrate, for which potassium nitrate may, if desired, be substituted. The mixture is heated in a current of mixed air and steam to an incipient red heat, say between 450° and 550° C., though the effective range is somewhat wider. The air and steam is preferably heated to the same temperature as that of the mass. The nitrous fumes evolved are collected and recovered as nitric acid by the use of air and water. The residue is a dirty green mass, consisting essentially of sodium (or potassium) ferrite, which, on treatment with water, yields caustic alkali and ferric oxide. The ferric oxide is levigated to separate any coarse particles of unattacked ore, and is then fit for use as a paint or polishing substance. The differences between this process and that patented by A. Vogt, Eng. Pat. 22,018 of 1891 (this Journal, 1893, 43), are pointed out.—E. S.

*Improvements in the Production of Caustic Alkali and Chloride of Lead for Conversion into Chlorine and Refined Lead.* F. Maxwell-Lyte, London, and G. Lunge, Zürich, Switzerland. Eng. Pat. 13,655, July 13, 1893.

THE present invention is an improvement upon Eng. Pat. 8692 of 1891 (this Journal, 1892, 686), according to which lead nitrate (obtained by dissolving lead oxide in nitric acid) is decomposed by an alkali chloride, such as sodium chloride, to form sodium nitrate and lead chloride. On then heating the sodium nitrate with ferric oxide, sodium ferrite is formed, with evolution of nitrous fumes, which are collected and recovered as nitric acid. About equal quantities were taken of the alkali nitrate and ferric oxide, and at the high temperature required to effect the decomposition, the material fluxed, and the nitrogen oxides evolved were so far split up as to be irrecoverable as nitric acid.

To overcome the defect in the former process, the proportion of ferric oxide is so far increased as to form a porous mass with the nitrate when heated. When ferric oxide, in the form of "purple ore," is used, two parts are taken to one part of the nitrate or thereabouts, and the heated mixture is subjected to the action of a previously similarly heated mixture of air and steam. The quantity of steam is carefully regulated, and the air is kept in due excess for converting the lower nitrogen oxides into nitric acid by means of a "Lunge tower," or otherwise. The resulting ferrite is decomposed by water to obtain caustic alkali and ferric oxide.

The liquor filtered off from the lead chloride obtained on mixing solutions of an alkali chloride and lead nitrate, still contains dissolved lead chloride. As much as possible of this is recovered by concentration and crystallisation, and the lead remaining in solution is precipitated by an alkali or alkaline earth.—E. S.

*An Improved Process for Producing Basic Lead Salts and Obtaining certain By-Products.* G. Lunge, Zürich, Switzerland, and C. H. Maxwell-Lyte, London. Eng. Pat. 13,656, July 13, 1893.

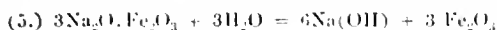
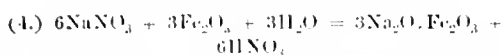
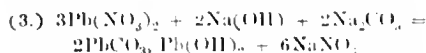
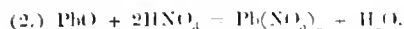
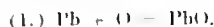
CRUDE pig lead is oxidised to form litharge, which is dissolved in nitric acid, and finely-divided lead is added to precipitate silver and other foreign metals. The silver lead obtained is a by-product of the process, when crude lead is first operated on. Litharge obtained from refined lead may,

\* Probably a misprint for July 14.



however, be used, though with less advantage. Basic lead carbonate is precipitated from the lead nitrate solution by adding sodium hydroxide and carbonate in the proper proportions, which are approximately 33 per cent. and 67 per cent. respectively. The sodium nitrate solution separated from the precipitate is, while being dried up, stirred with so much ferric oxide as will prevent fluxing of the mixture when this is subsequently heated with air and steam to recover nitric acid, caustic alkali, and ferric oxide, in the manner already described in the abstract of the immediately preceding patent.

The reactions taking place are represented by the following equations:—



The operations are similar when any other basic lead salt is required. For instance, basic lead chloride and caustic soda are produced by using brine instead of sodium carbonate. A chloro-carbonate may be obtained by using a mixture of sodium carbonate and chloride. "Sulphate, oxalate, or any other salt of soda may be employed so long as on addition to the lead nitrate it produces a sufficiently insoluble basic salt of lead and a residual nitrate from which the nitric acid is capable of being recovered in the manner specified, for use over again."—E. S.

#### *Improvements in the Treatment of Bicarbonate of Soda.* Sir L. Bell, Middlesbrough. Eng. Pat. 13,708, July 11, 1893.

SUPERHEATED steam is passed over moist sodium bicarbonate contained in a close chamber. Dry sodium carbonate then remains, while the mixed gas and vapour are collected in a cooler, where water is deposited, and nearly pure carbonic acid recovered, suitable for use in the soda process.—E. S.

#### *A Process for Utilising Acid Sulphates of Soda.* R. E. Chatfield, Sewardstone, Essex. Eng. Pat. 19,530, October 17, 1893.

RESIDUES obtained of acid sodium sulphates in the manufacture of nitric acid, as in the process described in Eng. Pat. 16,512 of 1893, or resulting from other processes, are saturated in aqueous solution with ammoniacal compounds or vapours from gas-liquor or other sources. If the residues consist of normal sodium bisulphate, the neutralised and filtered solution may be treated by either of two processes, (A) or (B). According to process (A), the solution, 48 per cent. of the salts in which are ammonium sulphate, is concentrated up to 76° Tw., and the sodium sulphate which falls during the concentration is fished out, to be afterwards dissolved in water and crystallised. The liquor will then contain salts enriched in ammonium sulphate to about 65 per cent. Process (B). The solution of salts containing 48 per cent. ammonium sulphate is brought to a specific gravity of 55°—60° Tw. at 90° F., or to corresponding density for other temperatures, and allowed to cool. The hydrated sodium sulphate (Glauber's salts) then crystallises out, and, after draining off the mother-liquor, is covered with water. These washings, when run off, are used for soaking subsequent lots. The salts in the well-cooled mother-liquor may contain as much as 77 per cent. ammonium sulphate.

Liquors having salts in solution containing 65 per cent. and upwards ammonium sulphate, as is the case with the mother-liquors obtained by either of the described processes, are evaporated, and the mixed sulphates fished out are agitated or ground with sodium sulphate washings, cold mother-liquors or water, until the density of the liquor ceases to rise, even though more salts are added. Such

density will be about 57° Tw., and the salts present will contain about 88 per cent. ammonium sulphate to 12 per cent. sodium sulphate, or a proportion of 8 equivalents of the former to 1 equivalent of the latter. From this liquor ammonium sulphate can be fished out on concentration up to a density of 76° Tw., when as much as possible of the liquor is run off before re-charging with fresh liquor of 88 per cent. ammonium sulphate. The liquor run off can be brought round up to the circle again. The processes are obviously applicable to sodium sulphates containing any excess of sulphuric acid.—E. S.

#### *Improvements in the Manufacture of Caustic Soda and Potash, out of Nitric and Sulphuric Acids.* W. Garraway, Netherfield, Lancashire, N.B. Eng. Pat. 7412, April 14, 1894.

SODIUM nitrate, preferably dried, is mixed with about two and a half times its weight of burnt lime, and is heated in a cast-iron retort into which superheated steam is passed and a little hot air when required. Nitric acid is evolved, and may be conducted to a vitriol chamber or Glover's tower, or may be condensed directly. The mixture of caustic soda and lime left in the retort is lixiviated, with aid of steam if necessary, and the solution filtered or decanted off; the lime is boiled down to produce caustic soda. The lime residue is dried and heated to redness to fit it for re-use. Potassium nitrate may be similarly treated to obtain caustic potash and nitric acid. Magnesia, baryta, or strontia may be substituted for lime in the process. The apparatus shown in the drawings for carrying out the invention consists of a pair of horizontal iron retorts, with drawing-off and charging doors, set over pyrites burners, and heated by the flame of the burning pyrites. The superheated steam-supply pipes and pipes leading the nitric acid to a Glover's tower are also shown. This pair of retorts is adapted for use in combination with a set of six chambers, so that one retort may be discharged while the other is still working. If not intended for use in connection with the manufacture of vitriol, the retorts may be heated from ordinary fuel in any suitable furnace.—E. S.

## VIII.—GLASS, POTTERY, AND ENAMELS.

### PATENTS.

#### *Improvements in the Manufacture and Production of Stoneware Sanitary Pipes and the like.* H. Tagby, Ashby-de-la-Zouch. Eng. Pat. 11,098, June 6, 1893.

THE object of the patent is to prepare stoneware pipes in such a manner that the joints between successive lengths of pipe may be more secure and impervious than is usually the case. To this end both the spigot and socket end of each length of pipe are protected by rings of dry sand or clay, so that the glaze applied to the pipe may be prevented from adhering to the spigot and socket. The surface being thus kept rough and unglazed, affords a good hold for the cement used in jointing the pipes. Additional security of joint may be obtained by the use of longitudinal or circumferential grooves on the spigot and socket, or by the use of a double joint arranged so that the included annular space may be filled up with cement.—B. B.

#### *New or Improved Substitute for Pounce-stone, and Process of Manufacturing the same.* H. Salmon and J. E. Whiting, Leeds. Eng. Pat. 13,386, July 10, 1893.

ONE hundredweight of "crushed meanwood sandstone" is mixed with 7 lb. of bay-salt, ground and sifted, freed from any particles of iron that may be present by passage



through a magnetic separator, mixed with 1 peck of soft-wood sawdust, previously wetted, the whole mass moulded and fired. The product consists of siliceous grains held together to form a porous pumice-like mass. Substances equivalent to those mentioned above may be substituted.

—B. B.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Slag Cement Experiments.* R. W. Mahon. J. Franklin Inst. 137, 1894, 184—190.

SLAGS low in silica and high in lime and alumina are best suited for cement making. Examples:— $\text{SiO}_2$  25.30—30.00,  $\text{CaO}$  48.00—47.50,  $\text{Al}_2\text{O}_3$  20.10—17.00. The best cement made from such slags is prepared by the addition of about 25 parts of slaked lime to 100 parts of slag by weight. Mortar made of one of cement to three of sand from such cement binds two bricks firmly together, even in the dry air of the laboratory. Test-pieces of such mortar, hardened one day in air and 27 days in water, compare favourably with such mortar for good bought cements. Neat cement test-pieces from the best slags show no shrinkage in the moulds in dry air. These cements set slowly. Their hardness is satisfactory. By far the most complete set of comparisons made was the study of tensile strength at 28 days of neat cement test-pieces, 1 sq. in. in cross section, hardened in dry air, moist air, and water, both for slag cements and bought cements. Four examples selected from the principal table of tensile strain after 28 days, follow. They are the English Portland and the American natural rock No. 1, both in extensive use in this country, and the best two of the slag cements.

Cement.	Tensile Strain.	Kept in
English.....	266	Dry air.
	333	Moist air.
	333	Water.
American, No. 1 .....	88	Dry air.
	109	Moist air.
	79	Water.
Slag cement .....	375	Dry air.
	210	Moist air.
	253	Water.
Slag cement .....	353	Moist air.

—W. S.

*Execution of Masonry and Brickwork during Frost.* A. Griel. Zeits. des Österr. u. d. arch. Ver. 1894, 95. Proc. Inst. Civil Eng. 116, 1894, 9—11.

See under I., page 713.

*Modern Street Pavements.* O. B. Gunn. Journ. Assoc. Eng. Soc., Chicago, 1893, 477. Proc. Inst. Civil Eng. 116, 1894, 41—43.

THE author lays down the following rules for an ideal pavement:—

- 1st. It must be constructed of durable materials.
- 2nd. It must be as free as possible from noise.

3rd. It must present the least resistance to traction (*i.e.* must be smooth).

4th. It must cause the least wear and tear to vehicles and horses.

5th. It should be easily cleaned.

6th. The wearing-surface must be practically non-absorbent.

7th. The cost must be reasonable.

In the United States, as in Europe, street pavements in large cities continue in a period of evolution. Having commenced with native earth as roadways, gravel was next used, and then, successively McAdam, Telford McAdam, cobble-stone, and stone-blocks were evolved; but the constant and intolerable noise arising from the contact of millions of wheels and horses' feet with the granite blocks, which for generations were looked upon as the acme of all pavements, has become such a dreadful nuisance as to cause a general demand in the larger cities for a smoother and less noisy pavement, and the choice now lies mainly between wooden blocks, vitrified brick, or asphalt.

Wooden blocks, 6 in. deep and 4 in. thick, were first introduced in the United States about 40 years ago by Samuel Nicholson, of Boston, and 20 years ago the "Nicholson pavement" was in very general use, especially in Chicago and the cities of the North-West, each layer being separated from the next by a half-inch space which was filled with coal-tar and gravel. In London and in Paris the wooden-block pavements are laid in the most thorough manner, with creosoted or otherwise prepared wood, are kept in perfect repair, and give great satisfaction; but the cost of repairs is greater than with any other pavement.

The busiest streets in London, Paris, and Berlin are formed with asphalt, and it has been also successfully used in New York, Washington, Boston, Buffalo, and other cities in the United States. In Buffalo about 150 miles have been laid during the last 11 years. It is durable, free from noise, presents the least possible resistance to traction, and can easily be kept perfectly clean; but is unsuitable for steep gradients, and can only be laid and repaired by experts who are thoroughly skilled in the manipulation and handling of asphalt. Where it is especially desirable to get rid of noise, if the gradients are steep and the traffic heavy, wooden blocks or vitrified bricks should be preferred to asphalt; their cost is much less than that of granite blocks. In the city of New York about half the asphalt that has been laid is over old stone-block pavements, and several macadamised streets have been covered in the same way.

Vitrified brick is the latest paving material, and is now on its trial with a fair prospect of becoming a prime favourite. It is durable, not very noisy, easily cleaned and repaired, and is quite smooth, presenting small resistance to traction, and is not hard upon vehicles and horses; very close-jointed and nearly non-absorbent. The clay requires careful manipulation before burning, as also improved kilns and methods of burning. Lately some interesting experiments have been made in burning the bricks by electricity, and it is claimed that, in this way, with material of the best quality, a perfect vitrified brick can be produced. The author, however, recommends that instead of using bricks of the ordinary size, their width should be increased to 3½ in.

This material is singularly applicable for the pavement of cities in the prairie States of the West, where neither timber nor stone for paving are at hand, and asphalt would be too expensive; good brick material abounds almost everywhere. In Kansas City its cost would be about two-thirds that of asphalt and only a little more than half that of stone blocks; and, judging from the results obtained from an experience of about four years, such a pavement would carry the traffic of a residential street for 20 years.

No pavement, however perfect in itself, is suitable for all conditions of traffic, and it must be chosen with due consideration to the circumstances; but, on the whole, the author would be inclined to give the first place to asphalt, and the second to vitrified brick.

## PATENTS.

*An Improved Composition of Materials to be used for Making Blocks for Paving, Cement for Walls, and other Purposes of the like kind.* A. Edwards, London. From J. W. Oyster, Louisville, U.S.A. Eng. Pat. 12,791, June 29, 1893.

The composition patented consists of two-fifths its weight of powdered menaccanite, ilmenite, or other titanite mineral; two-fifths of sand, ashes, or other powdered mineral matter; and one-fifth of asphalt or bitumen, with or without a small quantity (about 5 per cent.) of coal-tar. The materials are crushed, mixed, and pressed hot to form blocks suitable for paving, especially for the floors of stables and farm buildings.—B. B.

*Improvements in the Manufacture of Flax or other Vegetable Fibre Roofing.* G. McFearn, Belfast, Eng. Pat. 11,551, July 28, 1893.

VEGETABLE fibre is fed upon a carding drum and thence on to a wire-gauze cylinder, the interior of which is partially exhausted, so that the fibre is formed upon it into a sliver or bat, which is pressed, saturated with bitumen, and faced with a layer of Manilla paper, adhesion being secured by means of bitumen. The product has the merit of possessing a non-absorbent backing which prevents the bitumen soaking out of the fibre and into the woodwork of the roof when exposed to a hot sun.—B. B.

## X.—METALLURGY.

*On the Allotropic Transformation of Iron under the Influence of Heat.* G. Charpy. *Comptes rend.* 113 (1894), 418—421.

UNDER the influence of a permanent deformation in the cold, iron appears to undergo an allotropic transformation, indicated by the existence of a rectilinear step in the curve, serving to indicate the relation between the elongation and the load, when the metal is tested. M. Osmond designates the two modifications as  $\alpha$ -iron and  $\beta$ -iron. If the rod of iron tested is in the state  $\beta$ , the curve will be a continuous one. On the other hand, if it is in the state  $\alpha$ , the curve will contain the step above referred to. If it contains both modifications simultaneously, we can get an idea of the relative proportions of the modifications  $\alpha$  and  $\beta$  present by comparing the length of the step with that given by another bar of the same metal in the state  $\alpha$ . The author has applied this method in investigating the transformation of iron under the influence of heat. Iron in the state  $\alpha$ , when heated to a sufficiently high temperature, becomes transformed into the state  $\beta$ ; but the transformation is reversible, and disappears in cooling. In order to maintain it, the cooling must be done suddenly by plunging the metal into a cold liquid (as in the process of hardening). The author finds that the rapidity of the transformation increases with the temperature. Thus, in the case of hard steel (containing 0.8 per cent. of carbon) no change is produced after heating for an hour to 700° C., or for five minutes to 750°; but the transformation is complete after heating for 30 minutes to 750°, or for five minutes to 800°. Other kinds of steel give similar results. It is clear that in metallurgical operations such as hardening, attention should be paid not only to the temperature, but also to the time during which the metal is submitted to this temperature.—D. E. J.

*Action of Aluminium on Cast-Iron.* *Bull. Soc. Chim.* 11, 1894, 374—379.

It was shown in 1890 by Keep that in order to dissolve ferro-aluminium or pure aluminium in cast-iron, the latter must be melted and run on to the former, which then rapidly

melts and dissolves; it, on the other hand, lumps of ferro-aluminium be thrown on the molten cast iron, they swim up and become covered with a crust which prevents proper fusion and admixture. The addition of aluminium to cast-iron produces an effect different from that observed in the case of steel; instead of increasing the fluidity and lowering the melting point, the mass becomes more and more pasty as the aluminium present increases; even when only low percentages are present there is difficulty in running into moulds, the mass becoming lumpy. Further observations on the subject have been made by Lesclapart and Borsig; commercial aluminium of 92—98 per cent. was plunged into graphite crucibles containing molten cast-iron, by means of tongs, samples of the metal being previously taken for comparison, after skinning. Less than 20 seconds sufficed to effect fusion and solution of the aluminium; quickly-cooled ingots were then prepared in iron moulds 0.5 cm. thick and 12 broad; length variable; and ordinary ingots in sand moulds. The aluminium found in the final product was very much less than the quantity actually added, owing to oxidation; a larger proportion disappeared in the grey iron than in the case of white or mottled; thus in a series of experiments where the aluminium actually used amounted to 0.5, 1.0, and 2.0 per cent. respectively, the following figures were obtained on analysis of the final castings:—

Cast-Iron.	Aluminium added.			Aluminium existing per 100 parts composed.
	0.5 per cent.	1.0 per cent.	2.0 per cent.	
Grey .....	0.027	0.007	0.016	5.0
White .....	0.06	0.085	0.06	9.1
Mottled .....	0.05	0.112	0.06	9.4

As the result of various observations made with the object of investigating the relative effect of re-melting on the material of the crucible, and of the direct oxidising action of the air, the conclusion is drawn that aluminium protects silicon, manganese, and carbon from oxidation by virtue of its own superior oxidisability, and that it is capable of reducing their oxides, and likewise iron oxides; whence, the crucible material is not a matter of indifference. The castings are less liable to contain cavities and blisters due to escape of gases; thus with a white iron, naturally very porous, 0.5 per cent. of aluminium caused nearly all the cavities to disappear, whilst 1.0 and 2.0 per cent. entirely removed them; on the other hand, with 2.0 per cent. the mass thickens notably, so that the metal must be run very quickly in order to fill the moulds. Moreover, the contraction during cooling is apt to cause holes and blisters.

The alteration in resistance to flexure and traction caused by the introduction of aluminium could not be measured at all in the case of white iron; no perceptible effect could be traced with grey iron on comparison with samples of the metal used, drawn before addition of aluminium; with mottled iron, however, a regular increment was noticeable, more especially as regards resistance to flexure; thus when the test bars (30 mm. square in section, 1 metre between the supports) contained 0.05 per cent. of aluminium, the resistance was 28 kilos. per square mm.; whilst at 2 kilos. was the corresponding figure for bars containing 0.16 per cent.

On the whole, the conclusion is drawn that in general aluminium acts on cast-iron like silicon, but less energetically. Its use in iron foundling is but slight, excepting when small quantities can be introduced without considerable loss; for not only is any considerable degree of waste by oxidation costly, but also it brings about various other inconveniences, some of grave character.—C. R. A. W.

*Desilverisation of Lead by an Alloy of Zinc and Aluminium: the Roessler-Edelmann Process.* M. Knortzer. *Bull. Soc. Chim.* 1893, 9 [3], 1033—1037.

THE presence of zinc oxide in the dross obtained by desilverising lead by means of zinc is highly objectionable,

nilitating as it does against the removal and recovery of the zinc by distillation. The ideal product of the process would be an alloy of zinc and silver, free from lead.

The quantity of zinc which will remain dissolved in melted lead is 0·6—0·8 per cent. at 400°, and rather more than 3·0 per cent. at 700°. Zinc at 400° dissolves 1·5 per cent., and at 700°, 5·6 per cent. of lead. In the desilverising process the zinc-silver alloy cannot be freed from lead by liquation because of the presence of oxides, although on a small scale, where oxidation is very limited, such a separation is possible.

An alloy of 95 of zinc and 5 of silver was mixed with lead, in the proportion of 2·6 parts to 100 parts of lead, at 600°. The lead dissolved much of the zinc and but little of the silver, so that the 0·6 part of zinc which rose to the surface contained 20 per cent. of silver; it remained liquid at 600°. An alloy of zinc containing 10 per cent. of silver melts at 500°, and one containing 5 per cent. is nearly as fusible as is pure zinc.

The system devised from the foregoing facts is as follows:—The lead to be desilverised, is heated to 600 and successive portions of zinc, of decreasing silver-content, are added, and skimming effected after each addition. In this way the bath becomes poorer in silver and the alloy more fusible at each skimming; the temperature is allowed to fall from 600° to 400°. The following figures are quoted in illustration of the process:—

100 PARTS OF LEAD CONTAINING 150 GRMS. OF SILVER  
PER 100 KILOS.

#### Additions.

2·6 parts of zinc containing 150 grms. of silver.	
2·1     "     "     "     21     "	
1·6     "     "     "     3     "	
1·1     "     "     "     0     "	

#### Skimmings.

0·6 parts of zinc with 216 grms. of silver, or	Zn = $\frac{3}{1}$
2·4     "     "     53     "     "	45
1·8     "     "     19     "     "	94
1·5     "     "     10     "     "	150
	1

The desilverised lead contained 0·6 per cent. of zinc and 1 gm. of silver per 100 kilos.

The first skimmings can be distilled or electrolysed; the second serves for the first addition in the next operation, and so on.

Whilst the process works smoothly with pure argentiferous lead, the presence of copper, antimony, &c., renders the metal so much more oxidisable that the skimmings contain much oxide and show irregularities in their silver-content. The addition of aluminium, together with the zinc, greatly enhances the resistance of pure lead to oxidation, but its effect on impure lead, particularly if it contain copper, is not so marked; removal of the copper by a preliminary treatment with zinc, will, however, render the aluminium effective.

When the lead is free from copper it is possible to desilverise it in one operation. The zinc-aluminium alloy (0·5 per cent. Al) is melted at a red heat and mixed with lead obtained in liquating previous skimmings; this mixture is then run into the argentiferous lead, heated to the right temperature.

The proportion of alloy added for 0·1, 0·2, 0·4, and 0·7 per cent. of silver are 1, 1·2, 1·4, and 1·7 per cent. respectively, the appropriate temperatures being 450°, 480°, 510°, and 550°. The skimmings are liquated and the resulting crusts contain—

	Per Cent.
Silver .....	20—40
Lead .....	3—4
Copper .....	1·2—2·0
Zinc .....	76—54

They are melted and cast into slabs, which serve as anodes in an electrolytic bath. The electrolytic zinc con-

tains: zinc, 99·94 per cent.; iron, 0·009 per cent.; lead, 0·034 per cent., and a trace of silver. The residues of the anodes contain: silver, 74—80 per cent.; lead, 10—12 per cent.; copper, 1·5—5 per cent.; zinc, 0·2—0·5 per cent.

—A G. B.

*Vanadiferous Liguite found in the Argentine Republic, with Analysis of the Ash.* Prof. Kyle. U.S. Consular Rep., June 1894, 176—180.

The coal is lustrous black, even when finely pulverised, without fibrous structure, and remarkably brittle. On being heated it softens, emits much gas, which burns with a smoky flame, yielding a light, spongy coke, which, after combustion, leaves a singularly small amount of a greenish-coloured ash. The specific gravity of the mineral = 1·173. Its proximate analysis gave:—

Loss on drying at 100°, 2·05; volatile matters, 49·51; fixed carbon, 47·81; ash, 0·63; total, 100.

In the elementary analysis, the carbon and hydrogen were determined by combustion with lead chromate, the nitrogen by combustion with soda lime, and the sulphur by Eschka's method. Its composition, deducting hygroscopic water and use, was found to be as follows:—

Carbon, 60·59; hydrogen, 8·63; nitrogen, 1·43; sulphur, 4·23; oxygen, 25·12; total, 100.

The remarkably small percentage of ash and its peculiar greenish colour naturally attracted attention. On treating it with hydrochloric acid there appeared a deep-red coloration, which, on dilution and heating, changed to a greenish-blue. On addition of ammonia in excess the colour disappeared, showing it had not been due to traces of copper, as was at the first supposed. On further investigation the author satisfied himself that the ash contained vanadium.

The following plan was adopted for quantitative analysis. It is based on a method described by Dr. Adolfo Doering on the "Separation and Quantitative Estimation of Vanadic Acid," published in the Boletín de la Acad. Nacional de Ciencias en Córdoba, Rep. Arg. (Vol. V., p. 117), especially with regard to the separation of vanadic acid from iron and alumina and its precipitation as mercurous vanadate.

One gram. of the ash was repeatedly digested in hot dilute nitric acid; the insoluble silicates and carbon were washed on a tared filter, dried at 120°, weighed, ignited to remove carbon, and the weighed silicates reserved for further investigation.

The nitric acid solution, after removal of excess of acid, was nearly neutralised by sodium carbonate, acetate of sodium added, and on boiling, iron and aluminium phosphates and vanadates were thrown down. The precipitate was dissolved in nitric acid, some sodium phosphate was added, and the phosphates again precipitated by sodium acetate, the vanadium being retained in solution. The mixed phosphates were collected and weighed, the iron contained in these converted into sulphide and weighed as peroxide. The phosphoric acid was estimated, and its amount, plus that of the iron oxide, deducted from the weight of the total phosphates, the difference being regarded as alumina. As the determination of the  $P_2O_5$  included part derived from the added sodium phosphate, that corresponding to the ash was separately determined. The filtrate containing vanadium and excess of sodium phosphate was precipitated by ammoniacal magnesium nitrate, and as the precipitate retained some  $V_2O_5$ , it was redissolved, and the treatment repeated until the magnesium and ammonium phosphate was colourless, the mixed filtrates containing the  $V_2O_5$  were acidified with acetic acid, and the  $V_2O_5$  precipitated by mercurous nitrate. The mercurous vanadate was washed with hot water, collected, and carefully ignited to expel mercury, the residue = 0·034 gm.  $V_2O_5$ .

The filtrate from the mixed precipitates of phosphates and vanadates, thrown down by sodium acetate as previously mentioned, was nearly neutralised by sodium carbonate, and the  $V_2O_5$  precipitated in the hot liquid by mercurous nitrate; the mercurous vanadate on ignition weighed 0·25 gm. On fusion with sodium carbonate and digestion of the melt with hot water, there remained a residue which weighed 0·008 gm. which being deducted = 0·242 gm.

$V_2O_5$ , not combined with iron and alumina. After getting rid of excess of mercury by  $H_2S$ , the filtrate on being neutralised with ammonia assumed a dark colour, and after repose there was formed a very slight precipitate which contained traces of vanadium and manganese sulphides, which were not estimated; the filtrate from these was treated by ammonium oxalate, and the lime estimated as usual; traces of magnesia were found in the filtrate, but not estimated.

It being now apparent that only a small fraction of the vanadium was in combination with iron or alumina, the author treated a fresh portion of ash by digestion in warm ammonia solution, so long as anything appeared to dissolve. The alkaline solution was evaporated in a tared capsule, the residue consisting of calcium sulphate, ammonium vanadate, and vanadates of the alkaline metals. By drying and weighing the residue insoluble in ammonia, which was collected on a tared filter, the amount soluble in ammonia was ascertained. The filter was then ignited and the residue weighed, thus controlling the previous carbon determination. The residue was digested in hot nitric acid, and the solution employed to determine the phosphoric acid by means of molybdate of ammonium.

The mixed vanadates and calcium sulphate left on evaporation of the ammoniacal extract were treated with hot water, much of the calcium sulphate remaining undissolved; the  $V_2O_5$  was precipitated by lead acetate, the precipitate collected on a tared filter and washed with hot water, a few drops of ammonium acetate being added to insure removal of any lead sulphate; the lead vanadate was dried at  $100^\circ$  and treated as recommended by Roscoe (Roscoe and Schorlemmer, "Treatise on Chemistry," Vol. 2, Part 2, 299). As in this method the  $V_2O_5$  is estimated by difference between the weight of the dried lead vanadate and the contained lead weighed as sulphate, the estimation was checked by titration with permanganate of the sulphuric solution, the  $V_2O_5$  having been reduced to  $V_2O_4$  by sulphurous acid. After removal of the excess of lead in the filtrate from the lead vanadate, and of the lime by ammonium oxalate, the filtrate was evaporated to dryness, ignited to expel ammonia salts, the residue moistened with sulphuric acid, again ignited, and the sulphates weighed. The residue dissolved completely in water, the contained  $SO_3$  was on estimation found to correspond to the sulphates regarded as potassium sulphate; the sodium revealed by the spectroscope was therefore a mere trace. As the ash contained no carbonate the lime was calculated as sulphate.

The insoluble silicates were fused with alkaline carbonates, and the melt, being practically free from vanadium, was treated as usual for the estimation of silica and bases.

The composition of the ash after deduction of the carbon (which two separate determinations had shown to amount to 27.8 per cent. in the ash as analysed) may be stated as follows:—

#### Soluble in acid:

Vanadium pentoxide, 38.22; phosphorus pentoxide, 0.71; sulphuric anhydride, 12.06; calcium oxide, 8.44; ferric oxide, 4.98; aluminium oxide, 3.32; potassium oxide, 1.73.

#### Insoluble in acid:

Silicic anhydride, 13.7; ferric oxide, 9.42; aluminium oxide, 5.26; magnesium oxide, 0.83; undetermined traces of manganese, magnesia, chlorine, and loss in analysis, 1.33. Total, 100.

As the analysis of the extract obtained by digesting the ash in hot ammonia water showed that of the total  $V_2O_5$ , viz., 38.22 per cent., no less than 30.6, say 80 per cent., exists either as a vanadium oxide or as a soluble alkaline vanadate, only the remaining 20 per cent. can exist in combination with iron and alumina. When heated on platinum foil to the highest temperature of the blowpipe flame, the ash undergoes partial fusion and acquires a dark-red colour and metallic lustre.

On being boiled with distilled water, a yellow solution is obtained, the yellow colour being probably due to the change of the potassium metavanadate into tetravanadate,

by far the larger part of the vanadium compounds remaining insoluble in pure water, although, as previously mentioned, readily soluble in hot solution of ammonia.

There appears to be no doubt that the vanadium exists in the ash chiefly as vanadic pentoxide, in a free state, partly as potassium vanadate, and also some as iron or aluminium vanadates.

From the foregoing, it is evident that the San Rafael coal is one of great interest, being not only of fair quality as a combustible, but being also available as a source of vanadic acid for industrial purposes, seeing that each ton of the mineral, supposing it to be equal to the sample, will produce over 14 lb. of pure ash, containing  $\frac{1}{2}$  lb. of the vanadic pentoxide, of which 2 lb. may be extracted by simple treatment of the ash with an alkaline liquor, while the remainder is, of course, susceptible of extraction from the insoluble part as in the case of the basic slags of Creusot (Comptes rend. 95, 12-14), which contain but 1.5 per cent.

The ash of the San Rafael lignite is thus the richest known material utilisable as a source of vanadium compounds. Information regarding the carboniferous strata whence the sample of mineral was derived, indicate that the seam is about one metre in thickness.

The Argentine provinces of Córdoba and San Luis are already well known as sources of vanadium minerals, such as vanadite, descloisite, and psittacinite. The province of Mendoza must now be added to the list of localities in the Argentine Republic where vanadium is to be found.

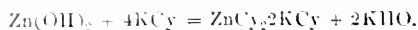
#### The Cyanide Process. W. H. Virgoe. Eng. and Mining J. 1894, 533-534.

THE determination of the sources of waste due to reactions in the leaching vats is a complex and difficult matter. Some ores consume more cyanide than others, and the consumption of this salt is always very greatly in excess of that required by the amount of gold in the material treated. The sources of waste in the leaching vats are similar to those in the storage tanks. A case occurred in Montana, where the leaching of the ore was conducted in covered tanks. The gas volatilised from the solution was so large in amount as to overpower two men engaged in removing the cover after the expiration of the time allowed for the leaching. But, in addition, there are the losses due to consumption of cyanide by base metals in the ore treated. In regard to hematite and limonite ores the consumption of cyanide is usually low. This is also the case with sulphide of iron ores, provided the latter be free from all oxidation. The manganese gold ores of California consume a little more cyanide than the corresponding ferruginous ores. Galena and zinc blends are unattacked. Great difficulty has always been experienced in treating copper ores containing more than 2 per cent. or 3 per cent. of copper, owing to the very heavy consumption of cyanide. A quarter of 1 per cent. solution is invariably the best to be adopted, if ores containing copper must be treated by this process.

Pyritiferous ores are liable to contain, especially when mined above the permanent water level, soluble salts of iron and small amounts of free sulphuric acid. It is necessary, therefore, in treating ores of this description, to first leach them with water in order to dissolve the ferrous and ferric sulphates out, and afterward to decompose the insoluble basic sulphates with a solution of soda-lye or lime water. All mill work should be based upon carefully-conducted laboratory experiments, determining the requisite mesh at which the ore must be treated, the strength of solution necessary, and the length of time for such solution to remain in contact with the ore for the attaining of an economical extraction.

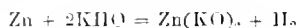
In the zinc precipitation boxes not only does an amount of zinc enter into solution proportionate to the amount of gold and silver and base metals precipitated, such as Cu, Sb, As, &c., thus,  $2KAuCy_2 + Zn = ZnCy_2 + 2KC_2 + Au_2$ , but far more, and the excess is proportionate to the decomposition of the potassium cyanide. Looking at the reactions occurring in an ordinary zinc box, reactions in part purely chemical, in part electro-chemical, involving a

waste of cyanide, it is seen that as soon as the gold is deposited on the zinc shavings, galvanic action is at once established between the electro-negative gold and the electro-positive zinc, hydrogen is vigorously evolved at the negative pole and the zinc converted into hydrate, which in turn is immediately attacked by the excess of potassium cyanide, forming zinc potassium cyanide and potash—



As this reaction is incessantly occurring, the solution becomes very strongly alkaline in the lower compartments of the zinc boxes. This galvanic action is a source of a large loss of cyanide, not only by causing the formation of the double salt of zinc and potassium cyanide, but undoubtedly by causing the oxidation of a certain quantity of potassium cyanide to cyanate.

The majority of mills make a mistake in not discarding screens made of fine iron wire in the compartments of the precipitation boxes, because there is a strong galvanic action established immediately between the zinc and the iron so soon as the cyanide solution flows through them, hindering the deposition of gold and silver and augmenting to a marked degree the loss of cyanide through solution of the zinc, besides largely increasing the zinc slimes in the product and making them more difficult to handle. Commercial zinc is only slightly soluble in potassium cyanide, but in contact with iron it is readily dissolved, especially in the lower compartments of the precipitation boxes, where the solution is most strongly alkaline. This reaction is thus expressed:—



Again, the majority of boxes in use are far too long. There is no advantage to be gained in using boxes 20, 30, and 40 ft. in length.

Precipitation takes place mainly in the upper compartments, after which the solution is unnecessarily exposed to the action of the zinc. Short boxes and plenty of them is the right motto.

These are, in a general way, the merits and disadvantages connected with the cyanide process. That there is a great future before it in America the writer has every confidence, and in the interests of mine owners and millmen, it is to be hoped that under the present organisation success may be achieved.

*Notes on the Cupellation of Bismuth-Silver Alloys.* E. A. Smith. Proc. Chem. Soc. 1894 [139], 117.

See under XXIII., page 765.

*The Conditions in which Carbon Exists in Steel.* J. O. Arnold and A. A. Read. Proc. Chem. Soc. 1894 [138], 105—106.

THE authors have carried out experiments on the condition in which carbon exists in normal, annealed, and hardened steel on a series of five steels containing 0.26, 0.57, 0.33, 0.16, and 0.6 per cent. of carbon respectively.

The method they have used in their determinations is a modification of the process of Binks and Weyl, originally proposed for the estimation of carbon in iron.

Two bars of polished steel of suitable size were suspended in separate beakers containing dilute hydrochloric acid (rel. dens. 1.02), placed in the same circuit, and connected with the positive pole of a single Bunsen cell, the cathodes being plates of platinum placed in a flat porous cell. At the end of the time allowed for solution the carbide was collected on a smooth filter paper and washed with water, alcohol, and finally with ether. The residue was washed off the filter paper into a weighed boat, and then dried in a vacuum. The carbon was estimated by direct combustion, the residue afterwards being used for the estimation of the iron.

From the results of a number of experiments the authors draw the following conclusions:—

1. The existence is confirmed of a carbide of iron possessing the formula  $\text{Fe}_3\text{C}$ , originally discovered by Abel and Müller by independent methods.

2. The normal carbide exists in two different forms—identical in chemical composition.

(a.) A diffused carbide is scattered in granules or very small plates throughout the iron in normal steel. When isolated it is a greyish-black powder.

(b.) A crystalline carbide, arranged in comparatively large distinct plates in well-annealed steel; these plates are chiefly in the form of well marked striae, and consist of pure  $\text{Fe}_3\text{C}$ . They are identical with the microscopical laminae of Sorby's "pearly" constituent, and they may be isolated as bright silver plates.

3. The percentage of the total carbon obtained as carbide is greater in hard than in soft steels.

	Per Cent.	Per Cent.	Loss per Cent.
In iron containing ....	0.1	92	8
" " ....	0.5	87	13
" " ....	0.25	74	23

4. The above-mentioned loss does not appear to be due to decomposition, but rather to the presence of a readily decomposed sub-carbide of iron (containing less carbon than the normal compound) existing to the extent of about 25 per cent. in mild steel, and capable of existing to the same amount in cold, hard steel after the latter has been heated for some time at a white heat.

5. The loss being practically the same in well-annealed steel as in the normal steel, it cannot be due, as supposed by Ledebur, to the presence of "hardening carbon."

6. The carbon in hardened steel exists chiefly as an extremely attenuated carbide, or in solution, leaving on isolation a residue consisting mainly of "hydrate of carbon," mixed to a slight extent with carbide of iron. Whether the large loss of carbon (about 50 per cent. of the total) occurring during the galvanic decomposition of the hardened steel is due to the presence of a large percentage of sub-carbide or to evolved hydrocarbons, formed by the action of nascent hydrogen on the finely-divided free carbon, there is no conclusive evidence to show.

The authors have also experimented on a steel containing 1.73 per cent. of manganese and 0.55 per cent. of carbon, and find that a portion of the iron is replaced by manganese, the double carbide apparently having the formula  $\text{Fe}_2\text{MnC}_3$ .

In the discussion following, Prof. Roberts-Austen observed that this was only a portion of a long series of experiments, many of which appeared to have been very carefully made. He regretted, however, to say that he entirely differed from the conclusions at which Mr. Arnold had arrived. He was not prepared to raise objections to the method employed, which, under the circumstances, was probably as good as any that could have been adopted; it was, moreover, a method which the speaker had himself used 30 years ago. He quite believed in the existence of Abel's carbide of iron,  $\text{Fe}_3\text{C}$ , but he considered that the authors of the paper had not offered any evidence which could be accepted in support of the presence in the steel of a new sub-carbide of iron. Without the exercise of the utmost care and absolute uniformity of conditions, nothing is easier than to lose carbon when steel is dissolved by electrolytic action.

## PATENTS.

*Process of and Apparatus for Separating Metals and similar Substances.* J. A. Mays, London. Eng. Pat. 2639, February 7, 1893.

THE metals or other substances are caused by centrifugal force to arrange themselves within a revolving chamber, while molten, in layers concentric to the axis of rotation, those of greatest specific gravity being farthest from such axis and *vice versa*. The metals may either be melted in

the revolving chamber itself or before their introduction thereto. In some cases traps, siphons, or passages are disposed in or around the chamber whereby each separate substance is let off into a suitable collector. Devices for cooling, granulating, and condensing are sometimes added.

—J. H. C.

*Improvements in the Purification of Iron for Casting and General Foundry Purposes, as also in the Production of Steel and Wrought Iron.* J. B. Cochrane, Stourbridge, and T. H. Taylor, Dudley. Eng. Pat. 11,709, June 14, 1893.

In treating iron for foundry and general casting purposes, a floating ball or brick composed of calcium fluoride or other halogen compounds, such as calcium, sodium, or ferric chlorides, together with an oxide, carbonate, or hydrate of an alkali, metallic iron and chloride of ammonium, is thrown into the ladle at the commencement of or during the filling, and the metal is desulphurised as it rises.

In treating iron for conversion into steel a similar ball is placed either in the ladle or in the furnace or converter, and in converting crude pig iron into wrought iron the ball is introduced at the melting or other period of the conversion.

The following proportions answer very well with a moderately sulphurous iron:—

	Per Cent.
Iron.....	35-50
Fluoride of calcium.....	28-12
Lime.....	50-15
Sal ammoniac.....	1-2

The materials are thoroughly ground or mixed together with water, moulded into the required size and shape, and dried.—J. H. C.

*Improvements in the Treatment of Lead Fumes.* G. Bischof, London. Eng. Pat. 12,034, June 19, 1893.

THE gases which carry the fumes are made to pass through a deep fire of carbonaceous material, or they are mixed while hot with combustible gas or vapour and passed through porous refractory material, such as fragments of coke or of brick free from silica. In either case metallic lead will be deposited.—J. H. C.

*Improvements in the Metallurgy of Metallic Sulphates.* W. Mills, London. Eng. Pat. 12,647, June 28, 1893.

THE invention has for its object the preparation of metals such as magnesium, calcium, aluminium, barium, potassium, and sodium by decomposing their salts in the presence of sulphuric anhydride and a reducing metal such as zinc, iron, tin, or lead.—J. H. C.

*An Improved Process for Treating Complex and Refractory Ores and Materials, and Obtaining the Metals and Pigments therefrom.* J. W. Hall, Bristol. Eng. Pat. 12,979, July 3, 1893.

THE ores are subjected to a strong blast, preferably hot, upon an open Scotch hearth or other suitable furnace, and the fumes containing the volatilised metals are drawn by a fan or otherwise into a settling tower or chamber through sheet-iron tubes or other cooling media into strainers made of suitable textile fabric. The fumes so obtained are utilised direct or further treated. For certain classes of ore rich in silver or gold, lead material is added, and for ores poor in the precious metals a cupola furnace is sometimes used instead of the Scotch hearth.—J. H. C.

*Improvements in the Manufacture of what is known as Manganese Steel.* R. A. Hadfield, Sheffield. Eng. Pat. 13,847, July 17, 1893.

THE improvement consists in bringing the ferro-manganese and the ordinary steel together, and mixing them in a ladle or vessel such as is adapted by suitable weighing mechanism

to show the weight of its contents, the ferro-manganese and the steel being added separately and successively so that the weight of each, and consequently the proportions thereof, can be easily controlled.—J. H. C.

*Improvement in Dissolving Leaching, and Filtering Ores or Minerals, and Apparatus therefor.* J. F. Phipps, O. Tinsley, London. Iron B. F. 4,637, Soc. London, and J. Storey, Sydney. Eng. Pat. 15,940, August 23, 1893.

STEAM or other elastic fluid is applied to cause the liquid solvent to pass repeatedly through the material to be dissolved or the solution to pass through a filter.—J. H. C.

*Improvements in the Art of Producing Metallic Zinc.* P. C. Choate, New York. Eng. Pat. 8985, April 24, 1894.

ZINC is volatilised as fume by heating its ores with a reducing agent in a furnace in the presence of air. This fume is heated with carbon at a moderate temperature, so as to eliminate any metallic constituents which are more volatile than zinc. It is finally reduced, distilled, and cast into anodes of suitable form.

The zinc anodes so prepared are immersed in a zinc sulphate solution containing a little soluble chloride; the cathodes may be plates of zinc, copper, or other suitable metal. On passing a current of electricity pure zinc is deposited, while any antimony, arsenic, copper, silver, gold, &c., present will be separated and will settle in the bath.

—J. H. C.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

*Electrical Smelting.* Consul-General Mason. U.S. Consular Report, June 1894, 237-240.

ONE of the most interesting processes, by reason of its simplicity and its probable value in practical application, is one invented by Edward Taussig, of Bahrenfeld, Germany, for the smelting and casting of metals by electrical heat, under the influence of rarefied air. The operation consists in fusing ores or metals in a closed furnace or chamber, the hearth of which is connected with moulds, into which the fused metal flows by gravitation, and is cast into any desired form, and in both of which (moulds and furnace) the air has been rarefied as far as practicable through exhaustion by air pumps or other mechanical means.

The apparatus includes a long, air-tight smelting channel or chamber, the hearth of which inclines to a central orifice, through which the fused metal passes into the moulds. This chamber is filled with the metal to be smelted, and heat is generated by passing an adequate current of electricity through the mass without the use of any kind of fuel, or the employment of carbon electrodes.

The furnace is lined with glazed fire bricks, which are so efficient a non-conductor as to insulate quite perfectly the contents of the chamber, so that the heat is developed where it is required evenly throughout the mass, and very little, if any, loss of current is experienced. From the testimony of experts who have examined this process in operation, it appears that a degree of heat sufficient to smelt pig iron in 15 minutes can be generated and maintained without unduly heating the other parts of the circuit, and this is equally true of the smelting of easily-fused metals like lead and zinc, and of Siemens-Martin steel of Swedish origin, which is known to be one of the most refractory of all the useful metals.

The entire absence of coke, coal, or carbon electrodes secures to the fused metal nearly absolute purity, and the

continued exhaustion of air and the gases generated by smelting increases the fluidity of the molten material and wholly prevents oxidation and blistering, and produces castings of a dense, smooth quality, which have shown, under elaborate tests, the highest mechanical qualities of which cast iron is capable.

For smelting raw ores, the most obvious advantage claimed for the process is that it can be worked wholly by water power and without fuel in mountainous and remote districts, which produce ores and have abundant water, but no coal.

(1.) It has been shown that by this process a degree of heat sufficient to smelt a ton or more of iron within 15 minutes may be generated, sustained, and applied at will without the use of carbon electrodes and without excessive heating or injury to other portions of the circuit. This alone is a fact of dominant and far-reaching importance.

(2.) Castings of iron and steel, up to a weight of 200 pounds or more, have been made which have shown all the desirable qualities—smoothness of surface, fineness of texture, and freedom from many of the impurities which are always more or less incident to smelting by ordinary methods.

(3.) Pig iron of excellent quality has been produced in small quantities by direct smelting from Swedish ores. The tests of this iron, as made by the Royal School of Mines at Berlin, have been published in several technical journals. The tables are too abstruse and complicated to be of general interest, but the net results may be summarised in the statement that the tractile strength of a round rod 10 millimetres in diameter was 3,410 pounds, and the resistance to compression of a rod 301 millimetres in length and 30 millimetres in diameter was 57,850 kilos., or 127,270 pounds.

*On the Minimum Electro-motive Force required for the Electrolysis of dissolved Salts of Alkalis.* C. Nourisson. Comptes rend. **118** (1894), 189—192.

BERTHELOT has shown that in discussing electrolytic actions there is no need to separate the primary from the secondary reactions: all that is necessary is to take into account the initial and final states of the solution. Taking, for example, sodium chloride, and assuming that there is total decomposition and then total combination (starting with the elements), we distinguish four reactions, which together constitute the electrolytic action:—

	Calories.
(1) $\text{NaCl} = \text{Na} + \text{Cl}$ , with absorption of...	96.4
(2) $\text{H}_2\text{O} = \text{H}_2 + \text{O}$ " " "	68.4
	<hr/> 164.8
(3) $\text{Na} + \text{O} + \text{H} + \text{Aq} = \text{dissolved NaOH}$ , with evolution of.....	117.8
(4) Formation of oxy-compounds of Cl.....	6.0
	<hr/> 117.8

The difference is  $164.8 - 117.8 = 47$  calories, and the current must therefore supply work corresponding to 47 calories, in order to decompose one equivalent of sodium chloride (it being of course assumed that it does nothing but electrolytic work). The electro-motive force would thus be  $\frac{47}{23.2} = 2.02$  volts, which is known to be sufficient for the electrolysis of sodium chloride. Similarly for sodium bromide we find 1.75 volts, and for sodium iodide 1.46 volts.

Taking next an oxy-salt, the data for sodium sulphate are as follows:—

	Calories.
(1) $\text{Na}_2\text{SO}_4 = \text{S} + \text{O}_4 + \text{Na}_2$ , with absorption of....	329.6
(2) $2\text{H}_2\text{O} = 2\text{H}_2 + \text{O}_2$ " " "	137.8
	<hr/> 467.8
(3) $\text{S} + \text{O}_3 + \text{Aq} = \text{dilute H}_2\text{SO}_4$ , with evolution of...	112.5
(4) $\text{Na}_2 + \text{O}_2 + \text{H}_2 + \text{Aq} = \text{dissolved NaOH}$ , with evolution of.....	223.6
	<hr/> 346.1

The difference is 99.7 calories. According to these equations we have left,—hydrogen at the cathode and oxygen at the anode. The result is the same if we assume that the only effect of the current is to decompose the water and separate the sulphate into free base and acid; thus:—

	Calories.
Decomposition of water.....	68.4
Neutralisation of sulphuric acid.....	31.4
	<hr/> 99.8

As sulphuric acid is dibasic, we must divide this result by two. Thus, an E.M.F. of  $\frac{99.8}{2 \times 23.2} = 2.15$  volts, is the lowest which will decompose sulphate of soda. Similarly, both the nitrate and the chlorate of soda are shown to require 48 calories or 2.07 volts.

Applying to the above results the laws of electrolysis, and also certain well-known thermo-chemical data, the author deduces the following general law:—The minimum electro-motive force required for the electrolysis of a dissolved salt of an alkali is constant, on the one hand for all oxy-salts, and on the other hand for all the haloid salts derived from the same acid.

The author has experimented on a large number of salts (using the current from a secondary battery, with a voltmeter graduated to fiftieths of a volt), and he gives data confirming the law above quoted.—D. E. J.

*On the Minimum Electro-motive Force required for the Decomposition of Electrolytes.* Max Le Blanc. Comptes rend. **118** (1894), 411—412.

THE author refers to M. Nourisson's paper (preceding abstract), and points out that he had arrived at the same results (starting from Arrhenius's theory of electrolytic dissociation) some three years ago, and had verified them experimentally.—D. E. J.

*Observations on the Preceding Note (on the Limits of Electrolysis).* M. Berthelot. Comptes rend. **118** (1894), 412—415.

REFERRING to the papers by MM. Nourisson and Le Blanc, M. Berthelot draws attention to the fact that their experiments are in exact accordance with those in his own memoir "On the Limits of Electrolysis," published in 1882. In this memoir he gave experimental proof that the E.M.F. required for the electrolysis of a salt of an alkali is the sum of two quantities: one equivalent to the heat absorbed in the separation of the acid and base in dilute solutions, the other equivalent to the heat of decomposition (into hydrogen and oxygen) of the water in which the substance is dissolved. This result is not based upon any theory as to the constitution of electrolytes; it is an experimental law, independent of all hypothesis. Of course it only holds good provided that neither the acid nor the base is oxidised or reduced during the operation. It proves that, under these conditions, the electro motive force depends upon the difference between the initial and final chemical states. The author in especial points out that his results were obtained quite independently of Arrhenius's hypothesis of electrolytic dissociation.

—D. E. J.

*Formation of Aluminium Sulphide.* F. Gruy. Zeits. angew. Chem. 1894 [10], 290—291.

BACHERER has shown (this Journal, 1893, 272; 1894, 42) that aluminium sulphide is a convenient compound for the preparation of pure aluminium by electrolysis. It has many advantages over the oxide, the chief of which is that considerably less energy is required for its decomposition than for that of the oxide (*loc. cit.*).

Hitherto aluminium sulphide has only been prepared on a small scale, as it had no technical value. A mixture of carbon and alumina is heated to a white heat and sulphur vapour passed over it. This process is not a convenient one for the preparation of the sulphide in quantity. The oxide employed must be free from iron and from silica. The



use of sulphur increases the cost of the manufacture as it is impossible to prevent considerable loss of this substance. A very good bath for the electrolytic decomposition of aluminium sulphide is obtained in the following way:—When the double chloride of sodium and aluminium is added to fused sodium sulphide, aluminium sulphide and sodium chloride are formed. The fluoride of aluminium behaves in the same manner as the chloride, thus:—



In this way it is possible to obtain an aluminium bath direct from cryolite. By the use of cryolite, however, the process is not cheapened; this can only be attained by employing other substances as raw material. Among other substances clay may be used as a raw material. By decomposing clay with sulphuric acid, aluminium sulphate is obtained. When this is fused with sodium fluoride, aluminium fluoride or cryolite is formed, thus:—



or



The isolation of the aluminium fluoride is impracticable; it is expedient to reduce the sodium sulphate to sulphide, which is accomplished by dropping carbon into the molten mass. The sodium sulphide thus formed interacts with the aluminium fluoride, forming aluminium sulphide and sodium fluoride. The separation of the latter from aluminium sulphide is quite superfluous, as this mixture can obviously in the molten condition be used as an electrolytic bath.

It is to be observed, however, that both sodium fluoride, as well as sodium sulphide, attack all fireproof materials, thereby rendering the melt impure. This may be avoided by employing a furnace, that portion of which coming into contact with the melt being of iron, and furnished with a water-cooling arrangement. In this case a crust forms on the surface of the iron, which protects the latter from the corrosive action of the melt.—A. R. L.

*Desilverisation of Lead by an Alloy of Zinc and Aluminium, the Roessler-Edelmann Process.* M. Knoertzer. Bull. Soc. Chim. 1893, 9—10, 1033.

See under X., page 737.

*The Electrolytic Separation of Gold from Silver.* C. Schnabel. Zeits. des Ver. deutsch. Ing. 1893, 653. Proc. Inst. Civil Eng. 116 [ii.], 1894, 86—87.

At the works of the Saint Louis Smelting and Refining Company an electrolytic method of refining has been substituted for the method of parting by sulphuric acid in the treatment of auriferous silver, containing  $\frac{1}{10000}$  of gold (28.8 grains per lb. troy). The silver is cast into plates measuring 10 by 8 by  $2\frac{1}{2}$  inches, two of which are placed together in a linen bag to form one anode, with a rolled plate of fine silver for a cathode. The dissolving vessels are wooden vats, divided into seven cells and rendered impermeable by a coating of bitumen, each vat containing 70 pairs of electrodes arranged in series. The electromotive force required for each bath is  $1\frac{1}{2}$  volts, with a current of 350 amperes per square metre, which is furnished by a dynamo of 100 volts and 200 amperes driven by a 30-h.p. Westinghouse engine. The electrolyte used in the first instance is water containing one tenth per cent. of nitric acid, which is made up with very weak solution of silver nitrate as the operation progresses.

The anodes are entirely dissolved in from 30 to 40 hours; the reduced silver separates in a crystalline form, shooting across from the anode to the cathode, so that short-circuiting very frequently occurs. To meet this difficulty an apparatus is used for scraping the deposit off the cathodes at intervals, which, at the same time, keeps up the circulation of the solution in the bath. The deposited silver so removed falls to the bottom of the bath into boxes with double bottoms, perforated and covered with linen, for collecting the precipitate, which is removed once a day. The finely-divided gold is kept back in the anode bags, where it is allowed to accumulate for a week before cleaning out. It is then boiled with nitric acid, washed, dried, and

melted with a little sand or borax, giving a brilliant 999 fine. The silver, after washing and drying, is found to be free from gold, and assays 999.5 fine. The silver nitrate obtained from the final treatment of the gold is used to keep up the bath to the proper strength. At Saint Louis 933 kilos. silver (about 30,000 ozs.) are parted by this method daily, and at Pittsburgh a still larger plant of 40,000 ozs. capacity is in use. At the latter place the finely divided gold is melted with a small quantity of silver before being subjected to the final boiling with nitric acid.

As compared with acid parting, the electrolytic method, which has lately been adopted in Europe by the Frankfurt gold and silver refinery, is cheaper and quicker, the whole operation being completed in 48 hours; causes less loss of silver, and can be worked without producing the injurious gases which are unavoidable when sulphuric acid is used. It can be applied with profit to silver containing  $\frac{1}{2}\%$  of gold as a minimum.

## PATENTS.

*A Process for Maintaining Positive Plates of Secondary Voltaic Batteries in Good Operative Condition.* E. Epstein, East Twickenham, Middlesex. Eng. Pat. 4106, February 24, 1894.

This invention relates to a process whereby, when the oxides upon positive plates become soft and liable to be washed off when the battery is agitated, they are hardened by removal from the cell, drying them, and then subjected to reduction and reoxidised electrolytically and replaced in the cell. Where both positive and negative plates are capable of such treatment they may be similarly treated by drying and reversal of current.—J. C. R.

*Improvements in the Manufacture of Elements or Electrodes for Voltaic Batteries and Electrolytic Decomposition Cells.* D. G. FitzGerald, London. Eng. Pat. 9906, May 17, 1893.

These improvements consist in treating lead oxides with a solution of one or both of the fixed hydrates of the alkalis—caustic soda and potash—and carbonating the alkali by exposure to an atmosphere containing carbonic acid, by which a dense, coherent, and non-disintegrating mass may be obtained.

The following are slightly abridged examples:—

A. Litharge, massicot, or minium, alone or mixed; caustic potash solution, sp. gr. 1.100 to 1.150, sufficient to form into a stiff paste, and then moulded; subjected preferably to a current of air for three days, which carbonates the alkali, partially dries the plates, and precipitates any oxide in solution, and the material "sets." The set plates are dipped, at least twice, for a few seconds in a solution of sulphate of magnesia, half saturated or more dilute, at intervals of a day or more, and dried by exposure to air after each immersion. The solution for last immersion more dilute and slightly acidulated with sulphuric acid, the plates left in this solution for some hours to dissolve out precipitated magnesium hydrate, and then stood in racks to drain, dry, and "season." Caustic soda may be employed instead of the potash, but undesirable on account of efflorescence, and if used with caustic potash the latter in larger proportion.

B. For plates of greater porosity. Lead oxides (as above) in a slightly damp condition pressed in a mould between linen or felt containing enough moisture to cause the surface particles to cohere, to allow of the subsequent treatment of the mass within the mould, or supported on a plate of glass, with an alkaline solution, as above, gently poured over the moulded mass. Subsequent exposure to carbonic acid and treatment with sulphate solution, as above described.

C. A mixture of any lower oxide or oxides of lead with peroxide of lead is used, containing 25 per cent. or more of the latter, made into a paste with caustic solution, or moulded dry and treated with caustic solution. Treatment with alkaline solution causes the plate or mass to set hard



and porous, even without the intervention of carbonic acid, so that exposure to that gas for a longer period than a few hours, and subsequent treatment with sulphate of magnesia, are not in this case essential. To produce the setting effect with certainty, the proportion of the lower oxide present should not be less than, say, 10 per cent.

The plates may be reduced or peroxidised by ordinary means, but the inventor prefers a method which forms the subject of another Eng. Pat. 11,983, June 17, 1893.

—J. C. R.

*Improvements in Plates or Elements for Secondary Electric Batteries.* The Lithanode and General Electric Company, Limited, and J. T. Niblett, London. Eng. Pat. 13,218, July 6, 1893.

According to this invention the active material which is employed is in the form of plates, slabs, or pellets, is of a coherent nature, being preferably that which is known as "lithanode," and of any desired shape, as square, triangular, hexagonal, cylindrical, around the edges of which grooves, which may be V-shaped, recesses, or holes are formed, by which a number of them are firmly locked together to form a large plate by a conductor cast therearound, itself exposing little surface at the face of the plate. Another plan is to groove the slabs on both faces, with holes extending from the grooves on one face to the grooves on the other face, so that the metal when run in fills these grooves and connecting holes.—J. C. R.

*Improvements in the Manufacture of Plates for Electric Accumulators.* C. Rousseau, Paris, France. Eng. Pat. 13,274, July 7, 1893.

According to this invention, lead plates are moulded with ribs and then, by mechanical means, transverse or oblique cuts or slits are made in the ribs so as to form teeth or projections, more or less sharp, which are designed to be beaten down over the paste or active material to retain it in contact with the lead plate or core.—J. C. R.

*An Improved Electrolytic Process for the Production of an Alloy of Lead and Sodium or Potassium.* C. T. J. Vautin, London. Eng. Pat. 13,568, July 12, 1893.

This invention consists essentially in the employment of lead in a molten condition as a cathode in a fused salt of sodium or potassium as electrolyte, by which, on electrolysis, an alloy of lead and sodium or potassium is obtained, the products given off at the anode being collected, if of value (this Journal, 1894, 449).—J. C. R.

*Improvements in Plates or Electrodes for Accumulators.* R. J. Gülicher, Charlottenburg, Germany. Eng. Pat. 6947, April 7, 1894.

These improvements consist in introducing the active substance on and between the fibres of woven or knitted fabrics, such as felt, the fibres being of either natural or artificial origin, so that the effective layer thus formed is porous, firm, and flexible, and the electrolyte easily penetrates into the interior portions, producing an even action on all the parts. It is preferred to produce on and between the fibres—sheep's wool, slightly or not at all felted—of the form and size of the plates to be produced, a spongy lead by chemical means, as an oxide reduced by an electric current in an electrolyte of weak sulphuric acid, or impregnated with sulphate of lead produced in the fibre by its first being saturated with a solution of lead acetate, and then placed in a bath of diluted sulphuric acid, or pressing a paste of sulphate of lead prepared with distilled water thoroughly into the fabric and covering its surface. The sulphate so applied to the fabric is reduced to spongy lead by zinc in a solution of common salt or weak sulphuric acid, and to prevent adherence of the reduced lead to the zinc plates, sheets of filter paper are introduced between them and the impregnated fabric.—J. C. R.

*An Improved Method, Means, or Process of Producing Caustic Soda and Chlorine Gas by the Electrolysis of Common Salt in Solution.* T. Drake, Ruddersfield. Eng. Pat. 7985, April 23, 1894.

This invention relates to the method of decomposing common salt solution to obtain chlorine and soda, employing a mercury cathode and an anode of carbon. The former is shown contained in an iron vessel, the inner walls or sides of which are "carbursed," with water circulating above it to dissolve out the sodium, and the anode, in a solution of common salt, contained in a porous pot in the middle. Arrangements are described for circulating both water and brine.—J. C. R.

*Improvements in the Electrolytical Production of Nickel and other Metals.* C. Hoepfner, Giessen, Germany. Eng. Pat. 13,336, July 8, 1894.

These improvements relate to methods of obtaining cobalt and nickel from their solutions, neutral or slightly acidulated with bad conducting acids, as citric or phosphoric, electrolytically, by placing them next cathodes, which may be movable, as rotating or oscillatory, and provided with brushes or rubbing cushions; and next the anode, which may be soluble or insoluble, a solution of a metal which is electro-positive to them, as chlorides of sodium, potassium, or calcium. An insoluble anode may be replaced by a soluble one, as zinc, more electro-positive than cobalt or nickel. Between the electrodes there may be placed membranes of nitro-cellulose, "nitro-cloth, nitro-linen," sometimes mixed with asbestos, backed on the cathode side by an open grating or sieve. It is proposed to obtain zinc, lead, tin, and copper from their solutions in a similar manner. In the case of copper, cuprous chloride being preferred, as yielding double the quantity of the cupric salts, and of better quality.—J. C. R.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*Carnaüba Wax.* A. M. Villon. Bull. Soc. Chim. 1893, 9, 1046—1048.

Each leaf of the *Copernicia cerifera* yields 7 grms. of carnaüba wax: the leaves are beaten and the dust from them is melted.

Carnaüba wax is yellowish, hard, brittle, and lustrous; its specific gravity is 0.999; it melts at 84°—85°, and dissolves in alcohol and in boiling ether. It must not be confounded with the palm-wax (*Lerxylon andicola*) from New Grenada, which melts at 72°.

The wax is bleached for candle-making by filtration through animal charcoal, or by hydrogen dioxide, or by potassium bichromate. For the second process the wax is granulated and immersed in hydrogen dioxide (5—10 vols.), saturated with ammonia or sodium carbonate, and kept at 50°; the addition of 0.5 per cent. of borax aids the bleaching. Candles are seldom made of carnaüba wax alone; stearine and ozokerite are generally added to the extent of 20—30 per cent.

"Brilliant paraffin" contains paraffin, 75 per cent.; carnaüba wax, 25 per cent.

"Brilliant gelatin" is prepared by adding a liquid containing water (10 litres), potassium carbonate (1 kilo.), and carnaüba wax (4 kilos.) to a solution of gelatin. The product is used in finishing leather.

Carnaüba wax varnishes are made by saponifying the wax with potassium carbonate containing a little caustic potash, precipitating the solution by the salt of a heavy metal, and dissolving the precipitate in light petroleum or spirit of turpentine.

Carnaüba wax is employed for making phonograph cylinders.—A. G. B.

*Notes on the Examination of Beeswax.* Lyman F. Kebler, J. Franklin Inst. **137**, 1894, 58-59.

Beeswax in American markets is adulterated, according to the author, to the extent of 50 per cent., whilst in the English markets the amount of adulteration rises to 66 $\frac{2}{3}$  per cent.

The melting point of beeswax varies from 62 to 64 °C. It is raised by adding carnauba wax, stearic acid, certain mineral waxes, and paraffins.

China wax, Japan wax, cacao butter, resin, tallow, spermaceti, vegetable wax, certain stearic acids, and paraffins lower it, whilst it is apparently unaltered when adulterated with suint wax, certain mineral waxes, paraffins, and stearic acid.

The specific gravity of beeswax varies from 0.960 to 0.973, and appears to be greatly influenced only by resin, carnauba wax, and certain mineral waxes, which increase it, and by paraffins, which lowers it.

The "acid number" ranges from 19 to 21 mgrms. of potash per gram. of beeswax. Stearic acid, resin, and suint wax increase, whilst carnauba wax, mineral wax, cacao butter, paraffin, and spermaceti decrease the acid number. China wax, Japan wax, and vegetable wax do not vitiate the number seriously. The following is a table embodying the properties of the various substances employed in adulterating beeswax. Some of the data are due to the author, the rest to Dietrich, Dammer, A. and P. Buisine (this Journal, 1891, 52 and 53, also 728-729), and Benedikt:—

Substance.	Melting Point.	Specific Gravity 15° C.	Acids Sol. in Water.	Acid Number.	Ether Number.	Total.	Iodine fixed by 1 gram. of Wax.	Vol. of H at 0° C. and 760 mm. yielded per gram.	Hydrocarbons from 1 gram. of Wax.
Yellow beeswax...	62-64	0.961-0.964	0-1	19-21	73-76	91-97	8-11	53-57.5	12.5-14.5
Beeswax bleached by various agents.	63-64	0.960-0.973	0-2	19-23	74-84.20	94-107.7	1.8-11.30	51-57	11-14.3
Cacao butter.....	30-33	0.945-0.952	0	0-3	132-200	132-204	34	..	..
Carnauba wax....	83-84	0.969	0	4-6	75-76	70-82	7-9	73-76	1.6
China wax.....	53.5	0.970	2	22	196	218	0.85	72.0	0
Japan wax.....	47-54	0.975	2	18-28	194-198	216-222	0.755	60-71	0
Mineral wax.....	60-80	0.918-0.952	0	0	0	0	0-0.6	0	100
Paraffin.....	38-74	0.913-0.914	0	0	0	0	17-31	0	100
Resin.....	53.5	1.104-1.108	0	168	10	178	135.6	35	0
Spermaceti.....	40-50	0.945-0.96	0	0-2	136-142	136-144	..	..	..
Stearic acid.....	53.5-59.2	0.900-1.00	0	204	5	209	2-4	0	0
Suint wax.....	62-66	..	0	95-115	4-7	102-119	13-18.5	0	14-18
Tallow.....	42-50.5	0.952-0.96	0	2.75-5	193-208	196-213	27-40	52-60	0
Vegetable wax....	47-55.6	0.947	2	17-19	203-210	218-220	6.6-8.2	73-74	0

The "ether numbers," varying from 73 to 76 mgrms. of potash per gram. of beeswax, are unaffected by adding carnauba wax; but China wax, Japan wax, cacao butter, tallow, vegetable wax increase it. Mineral wax, paraffin, rosin, stearic acid, and suint wax decrease it. It must be noted that wax bleached by certain chemical agents may have an ether number as high as 84 and yet be pure.

The percentage of iodine varies from 8 to 11, yet wax bleached by certain agents, as chlorine, may vary far from these percentages. Paraffin, mineral waxes, and stearic acid lower the percentage, but cacao butter, rosin, suint wax, and tallow increase it. China wax, carnauba wax, Japan wax, and vegetable wax pass the prescribed limits but very little.

The volume (53 to 57.5 c.c.) of hydrogen evolved from 1 gram. of beeswax and the percentage (12.5 to 14.5) of hydrocarbons evidently are the most reliable data to be secured; the former being vitiated by all adulterants except tallow, and the latter by all except suint wax.—W. S.

*Continuous Purification of Oils.* A. M. Villon. Bull. Soc. Chim. 1893, **9**, 1037-1038.

THE oil and purifying liquid, generally sulphuric acid, are caused to flow in proper proportions into a centrifugal emulsifier, provided with revolving plates. The intimate mixture thus obtained is received in a lead tank, where the reaction is allowed to proceed. The oil is then washed with water in a second centrifugal emulsifier. The mixture of oil and water is then put through a separator similar to a cream-separator. Filtration of the oil through sawdust and wood-charcoal completes the process.—A. G. B.

*Manufacture of Pressure Lubricants.* A. M. Villon. Bull. Soc. Chim. 1893, **9**, 1044-1046.

THESE lubricants consist of solutions of a lime soap in a mineral oil. Several methods may be adopted for preparing them; thus, the soap may be previously prepared and then dissolved under pressure in a mineral lubricant, or the mineral and vegetable oil may be mixed and heated under pressure with lime. Magnesia is sometimes advantageously added. Good proportions are (1) mineral oil, 100 kilos.; linseed oil, 25 kilos.; earth-nut oil, 25 kilos.; lime, 6 kilos.; (2) mineral oil, 100 kilos.; linseed oil, 30 kilos.; earth-nut oil, 20 kilos.; lime, 3 kilos.; magnesia, 3 kilos.—A. G. B.

*Lard.* Samelson. Zeits. anal. Chem. 1894, **33**, 189-192.

See under XXIII., page 766.

*The Disinfecting Power of Soap Solutions in respect of Cholera Germs.* Maximilian Jolles. Zeits. f. Hygiene, 1893, 460. Proc. Inst. Civil. Eng. **116**, 1894 (ii.) 48.

See under XVIII. C., page 754.

#### PATENTS.

*Improvements in or in connection with the Extraction of Oil by Volatile Solvents, and Apparatus therefor.* A. W. Macellwaine, Hull, and G. G. M. Hardingham, London. Eng. Pat. 13,478, July 11, 1893.

IN the extraction of oils from rape and other seeds by volatile solvents, a certain quantity of air, highly charged with the vapour of the solvent, escapes. The mixture of

air and vapour is passed in a finely-divided state through the seed or oil in course of extraction, which absorbs the solvent vapour. On distillation, the solvent is recovered by condensation.—J. J. K.

*An Improved Manufacture of Compounds of Iron with Fatty Acids.* A. K. Huntington, London. Eng. Pat. 14,407, July 26, 1893.

SEVEN parts of oleic acid or linoleic acid with one part of soda or its equivalent in potash, are dissolved in about 80 parts of alcohol and 20 parts of water. Also 4 parts of ferric sulphate are dissolved in 56 parts of alcohol and 28 parts of water. The two solutions are mixed, when liquid oleate or linoleate of iron containing alcohol, and an alcoholic solution of sodium sulphate, are produced. The oleate is separated by decantation, washed with alcohol, and the alcohol retained by the oleate is recovered by distillation.—J. J. K.

*An Improvement in what is commonly called Dry Soap or Soap Powder.* W. H. Horton, Old Trafford, Manchester. Eng. Pat. 8676, May 2, 1894.

THIS relates to the incorporation of disinfectants, such as carbolic acid, with the materials used in the manufacture of dry soap.—J. J. K.

*A New or Improved Dubbing or Dressing for Leather.* T. W. L. Emden, London. Eng. Pat. 8695, May 2, 1894.

See under XIV., page 747.

### XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

#### (A).—PAINTS, PIGMENTS.

##### PATENT.

*Improvements in and connected with the Treatment of Lead Sulphate for use as a Pigment.* W. V. Wilson and J. J. Lundy, London. Eng. Pat. 9521, May 12, 1893.

LEAD hydroxide is incorporated with lead sulphate to give the latter greater covering power, the proportion preferred being two equivalents of the sulphate to one of the hydroxide. The lead hydroxide may be obtained in any convenient way, but the process recommended consists in continuously heating lead carbonate in contact with water or steam. Or the lead hydroxide may be made from a portion of the lead sulphate itself; as, for instance, by treating the lead sulphate with a suitable proportion of solution of an alkaline carbonate, washing away the alkali sulphate formed, and then heating the mixture of lead sulphate and carbonate in the presence of water or steam, the process being accelerated by operating in a closed vessel under pressure.—E. S.

#### (B).—VARNISHES, &c.

##### PATENT.

*An Improved Dressing for Boots and Shoes.* T. James, Derby. Eng. Pat. 15,528, August 16, 1893.

See under XIV., page 747.

#### (C).—INDIA-RUBBER, &c

*Note on Gutta-percha.* — Lagarde, Annales Télégraphiques, 1893, 421. Proc. Inst. Civil Eng. 116 [ii.], 1894, 102—103.

THE gutta-percha examined by the author had been extracted from the leaves of *Isonandra* by Serullas' process. It formed the coating of a copper wire, and appeared to possess excellent insulating properties. On analysis it was found to contain 83.20 per cent. of pure gutta-percha and 16 per cent. of resins. These latter consisted of 1.72 per cent. of fluaville and 14.28 of albane, to which its excellence as a dielectric may be attributed. There was a complete absence of water, which would no doubt add to the durability of the material, while increasing its insulating power. The resins, especially albane, increase the insulation, but facilitate the ultimate deterioration of gutta-percha. Two submarine or subterranean cables made with different kinds of gutta-percha might at first possess the same properties as regards insulation; but the one containing least water and resins would remain the longest in good condition. Chemical analysis therefore affords us the only means of ascertaining whether the gutta-percha of a cable is of good or bad quality, and until recently gutta-percha of bad quality would have been accepted if it complied with the electrical conditions contained in the specification. Cases are known where the insulation of submarine cables has so deteriorated within a few months that they have become useless, and the same thing has occurred with land cables. Gutta-percha containing a large quantity of albane sometimes cracks, especially in cold weather, at places near joints. The author considers that a fairly good gutta-percha should contain at least 55 per cent. of pure gutta, not more than 5 per cent. of water, and 1 per cent. of impurities, and about the same proportion of albane as fluaville. A good material contains 60 per cent. of pure gutta, not more than 5 per cent. of water, or 0.8 per cent. of impurities, the quantity of albane not exceeding that of fluaville. A material containing 65 per cent. of pure gutta, not more than 3 per cent. of water, 0.5 per cent. of impurities, and 12 per cent. of albane, must be regarded as of very good quality.

### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

*Tanning with Extracts.* A. M. Villon. Bull. Soc. Chim. 1893, 9—10, 1038—1044.

THE author holds that "pure" tannin will not convert hide into leather, the extractive matter always associated with the tannin in tanstuffs being essential. He distinguishes between those extractive matters which are "assimilable" in the same way as is the tannin, and those which are merely "adhesive" to the hide fibre, serving to consolidate the leather and render it waterproof (compare Procter, following abstract). If these premises be conceded, the proportion of extractive matter to tannin in a tanstuff is obviously of importance.

"Pure" tannin and an extract free from tannin, but containing the extractive matters, were prepared from oak-bark. Portions of the same hide were tanned in solutions of the pure tannin of varying strength, and in a solution of the tannin to which varying proportions of the extractive matters had been added. The conclusions drawn from the tabulated results are that—(1) pure oak tannin is absorbed in constant proportion by the hide whatever the strength of the liquor; the resulting leather is unfit for use, for it is brittle, stiff, and dry; (2) when the extractive matter is added to the liquor the yield of leather immediately increases by from 5 to 10 per cent.; (3) when the assimilable extractive matters are present to the same or to a greater extent than is the tannin, the yield is very high, but the leather is spongy,

soft, and perishable. When the proportion of assimilable extractive to tannin is 15—20:100 the leather is perfect; in this case the proportion of extractive absorbed to tannin absorbed is about 100:18, namely, the same proportion as that existing between these constituents in the oak-bark itself.

In attempting to explain the influence of the composition of a tan-stuff on its quality for producing leather, the author distinguishes between tannin, assimilable extractives, resinoid extractives, and inert extractives. A tan-stuff is the better the smaller the percentage of resinoid and inert extractives in the soluble matter of the tan-stuff, and the more nearly the proportion of assimilable matters approaches 15—18 per cent.

Extract of oak-bark is not so good as is the bark itself for tanning, and is the less so the higher the temperature at which it has been prepared; for, as will be seen in the

following table, the proportion of assimilable extractive is lowered by reason of the conversion of some of the substances contained in it into resinoid and inert extractives. Still worse is the commercial extract which is prepared with hot water and concentrated to 25° B.

The table, showing the composition of the soluble matter in the tan-stuffs, will serve to indicate which of the ordinary tan-stuffs will give a soft leather or a dry leather, when it is remembered that these qualities are dependent on the presence of a certain proportion of assimilable extractive as indicated above.

It will be noted that Valonia, divi-divi, and Kuoppenn galls approach oak-bark in that their soluble matters contain more than 12—13 per cent. of assimilable extractives, and very little resinoid extractives. Spruce, fir, pine, hemlock, alder, and mimosa barks contain sufficient assimilable extractive, but too much resinoid extractive.

—A. G. B.

*Does Tannin Tan Hides?* H. R. Procter. Chem. Zeit. 1894, 370. (Compare preceding abstract.)

In reply to the above question, the author states that there is no foundation for the assertion that pure tannin will not make leather. The only difference between a piece of a skiver tanned with pure tannin and of one tanned with sumach is in colour, that of the piece tanned with pure tannin being preferable. There is no doubt that the tannin of galls is identical with that of many other tanning materials; it is not a good tannage for sole-leather, which demands the use of tannins giving solid deposits, such as ellagic acid and phlobaphens, which impart firmness.

—A. G. B.

#### PATENTS.

*An Improved Dressing for Boots and Shoes.* T. James, Derby. Eng. Pat. 15,528, August 16, 1893.

This is a mixture of a varnish, known as body, carriage or oak varnish, with emery or other finely-divided substance. It is said to make boots waterproof, and increase their wearing qualities.—J. J. K.

*A New or Improved Material Capable of being used in Substitution for Leather and for Ordinary Paper.* N. Kumagaya, Osaka, Japan. Eng. Pat. 19,181, October 17, 1893.

See under XIX., page 751.

*A New or Improved Dubbing or Dressing for Leather.* T. W. L. Emden, London. Eng. Pat. 8695, May 2, 1891.

The essential feature of this invention consists in the use of a large quantity of mineral oil, varying from 60 to 80 per cent., in combination with about 3 per cent. of "monohydrated calcium oxide." One formula is as follows:—Mineral oil, 60 parts; vegetable oil, 20 parts; fish oil, 10 parts; water, 12 parts; monohydrated calcium oxide, 3 parts. The water and lime are put into a boiler, and the vegetable oil and 20 parts of mineral oil are added. The mixture is gradually heated to 90° C. until saponification takes place; the remainder of the mineral oil and the fish oil are then added, and the mixture allowed to cool.

—J. J. K.

APPENDIX.—This Journal, 1891, June number, page 652, col. 1, in the reference where page omitted, add the figure 648.

	Pure Tannin.	Assimilable Extractive.	Resinoid Extractive.	Inert Extractive.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Oak-bark .....	80	17	1	2
" extract, made at 30°.	80	15	2	2
" extract, made at 100°.	78	12	7	3
" extract, made at 25° B. Coml.	75	10	10	5
Oak-wood .....	85	10	2	3
" extract, cold.	83	9	4	4
" " tepid.	82	8	4	5
" " 20° B. Coml.	80	6	5	9
Chestnut-wood .....	87	9	0.5	3.5
" " extract, cold.	85	8	1	0
" " extract, tepid.	85	8	1	6
" " extract, 25° B. Coml.	83	5	3	8
Quebrache-wood .....	80	13	5	2
" " extract, 20° B.	78	10	10	2
Spruce-fir bark .....	70	15	10	5
" " extract..	70	10	15	5
Pine-bark .....	75	15	12	3
" extract .....	75	12	15	3
Valonia .....	80	18	0.5	1.5
" extract .....	80	16	2	2
Hemlock .....	70	12	16	2
" extract .....	70	10	18	2
Kuoppenn galls .....	78	18	2	2
" " extract...	78	15	5	2
Alder-bark .....	70	12	4	6
" extract .....	78	10	18	4
Divi-divi .....	80	16	2	2
" extract .....	80	14	4	2
Mimosa .....	75	12	12	1
" extract .....	72	10	15	3
Sumach .....	88	8	2	2
" extract .....	86	7	3	4
Myrabolans .....	75	9	9	6
" extract .....	72	7	15	6
Galls .....	90	5	2	3
" extract .....	90	3	4	3

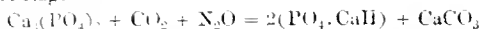
## XV.—MANURES, Etc.

## PATENTS.

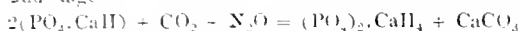
*Improvements in the Manufacture of Manures, and New Processes for obtaining the same.* H. E. Newton, London. From C. Seybold and F. Heeder, Wirlfrath, Elberfeld. Eng. Pat. 11,804, June 15, 1893.

Phosphatic raw materials heated at 50–120° C. in an atmosphere of moist carbonic acid are decomposed according to the equation—

1st stage—



2nd stage—



By the addition of alkaline carbonates the double salts  $(\text{PO}_4)_2, \text{Ca}_2\text{HK}$  and  $(\text{PO}_4)_2, \text{CaH}_2\text{K}_2$  may be similarly prepared.

The conversion takes from 12 to 24 hours, according to the nature of the raw material and the conditions of treatment, which are suitably varied.—C. F. C.

*New Process for the Preparation of Bi-calcic Phosphate soluble in Ammonium Citrate.* T. Chandelon, Liège. Eng. Pat. 13,842, July 17, 1893.

Raw calcium phosphate is treated with dilute sulphuric acid (15–25 per cent.  $\text{H}_2\text{SO}_4$ ), and the resulting solution, containing 7–12 per cent.  $\text{P}_2\text{O}_5$ , is cooled and neutralised with a cooled lime water. Reaction at 0° C. results in the precipitation of the hydrate  $\text{Ca}_2\text{H}_2\text{P}_2\text{O}_7 \cdot 2\text{N}_2\text{O}$ , entirely soluble in ammonium citrate.

A simple form of apparatus is figured for carrying out the reaction under the essential condition of low temperature specified.

This condition constitutes the patentee's claim.—C. F. C.

## XVI.—SUGAR, STARCH, GUM, Etc.

*Loss of Heat in the Steam Engines of a Sugar Factory.* Classen. Zeits. Ver. Rübenzuck.-ind. 44, 80.

To elucidate this question, the author has carried out a series of practical experiments in a beetroot factory, the normal working capacity of which was equal to 7,000–7,500 metre-centners. This factory possessed 17 old non-expansive steam engines, which required 35 kilos. of steam per horse-power per hour. As a mean deduced from a large number of determinations, he finds that the loss of heat corresponds to 16.4 per cent. of the steam required, a value which agrees with that calculated from exact data, thus:—By condensing in the pipes, 5.83 per cent.; by condensing in the cylinders, 1.38 per cent.; by friction of the machinery and by expansion of steam, 4.58 per cent.; giving a total loss of 14.58 per cent.

On economical grounds the author denounces the complete centralisation of the steam engines, the substitution of non-expansive for expansive engines, and the erection of evaporators having a higher than quintuple effect. It is urged that the steam pipes should be simplified as much as possible and well covered, the latter especially in the case of recent plant. Moderate centralisation of the machinery is to be recommended in such a way that the waste steam can be utilised as far as possible.—A. R. L.

*The Multirotation of Sugars.* P. Th. Muller. Comptes rend. 1894, 118, 425–428.

Most sugars exhibit a peculiar phenomenon in aqueous solution: the rotatory power of the substance when dissolved in water varies with the time, and to this phenomenon the name multirotation is given. If the temperature be kept constant, the rotatory power has a certain value immediately after the sugar is dissolved; and this value generally diminishes, more or less quickly, but as a rule attains a constant value in about twenty-four hours. Although the cause of this multirotation remains unknown, we can investigate the variation of the rotation, and find the law which it follows by assuming that during the whole duration of the phenomenon we have present two modifications of the particular sugar, each of which modification possesses a definite specific rotation. This assumption does not premise anything respecting the nature of the modifications—whether the substance retains for a time in solution a certain crystalline structure, whether a gradual process of hydration of the molecules goes on, &c. As the unstable modification A becomes transformed into the stable modification B, the rotation itself will vary. Let  $\rho$  be the specific rotatory power of B,  $\alpha$   $\rho$  the specific rotatory power of A, each referred to the anhydrous substance; suppose that we have  $p$  grms. of substance dissolved in  $V$  cubic centimetres; that we observe the rotation in a tube  $l$  decimetres long; and that the angle of rotation at the time  $\theta$  (counting from the time of solution) is  $\alpha$ . If  $x$  is the mass of A, and  $y$  the mass of B at the time  $\theta$ , we have—

$$\alpha = \frac{a \rho x l}{V} + \frac{\rho y l}{V},$$

$$\text{and } x + y = p.$$

If the final angle of rotation is  $\alpha_n$ , we have—

$$\alpha_n = \frac{\rho p l}{V},$$

$$\text{and } \therefore \frac{(\alpha - 1)x}{p} = \frac{\alpha}{\alpha_n} - 1,$$

an equation which gives the value of  $x$  (the amount of the unstable modification) at the time  $\theta$ . Now, it is natural to assume that the quantity  $dx$  which is transformed during the infinitely short time  $d\theta$  is proportional to the mass  $x$  still remaining at the time  $\theta$ .

$$\text{Thus } dx = -Cx d\theta$$

From this, by integrating, and replacing Napierian by common logarithms, we get—

$$C = \frac{1}{\theta - \theta_0} \log \frac{\alpha - \alpha_n}{\alpha - \alpha_n}$$

where  $\alpha$  is the rotation at the time  $\theta_0$ .

Experiment shows that such a constant ( $C$ ) really exists; thus confirming the above hypotheses and deductions. Pareus and Tollens have studied the rotatory power of sugars during the first few hours after solution, and the author has made use of their curves and tables in testing his theory. From one set of experiments on arabinose (at 20°) he finds 0.0301 as the value of the constant  $C$ ; another set of experiments gives the value 0.0293. The value of the constant for xylose is 0.0201; for rhamnose, 0.0393; fructose, 0.0202; galactose, 0.00927; milk-sugar, 0.00405; maltose, 0.00524; and for glucose about 0.00365. Thus the reaction which takes place during the period of multirotation is of the first order; it follows the law of active masses. The constant  $C$ , at any given temperature, measures the rate of progress of the phenomenon. This rate is much more rapid in the case of the pentoses than in the case of the other sugars (hexoses and polysaccharides).

—D. E. J.

*Diffusion.* Gaz. cukr. 1, 386.

At the meeting representative of the Polish Sugar Industry, a committee was appointed to investigate the influence of the form of the diffusors and the number of vessels in the diffusion battery on the entire diffusion process and more especially on the purity of the juice.

The following is a summary of the report of B. Broniewski, whose experiments were conducted in two factories; one at Krasiniec and the other at Sanniki. The diffusers in the former factory are 1.5 metres in diameter and 2 metres in height, whilst those in the latter factory are barely 1 metre in diameter and are 2.1 metres in height. The experiments confirm Herzfeld's observation that the purity of the juice is the higher the thinner the slices of roots employed. If, however, thin slices of roots are used, the pressure falls in a battery consisting of several high and narrow diffusers, because of the considerable velocity with which the juice runs through, so that the number of vessels in the battery must be diminished. This may, however, not deleteriously, for it causes an increase in volume of the juice, or less perfect edulcoration. In working therefore with thin slices, it is advisable to employ lower and wider vessels. Regarding the number of vessels, the experiments show that a battery consisting of few members and a short diffusion period is to be recommended; in this way the purity of the juice is enhanced. The number of diffusers is so regulated that with 100 per cent. juice extracted sufficient lixiviation is attained.

No special experiments were made to ascertain the speed at which the juice should run through the diffusers. In Sanniki the highest purity is 30 per cent. with a speed of 7 mm. per second.—A. R. L.

#### *Combustible Gases arising from the Diffusion Battery.* Dewald. Deutsch. Zuckerind. 19, 52.

THE evolution of combustible gases from the diffusion batteries is not due, as Stohmann believes, to the action of the acid juice on the iron of which the apparatus is constructed, but is to be ascribed (as already observed by Scheibler) to a lactic or butyric fermentation. The prescribed addition of lime tends therefore to increase rather than eradicate the evil, because these fermentations proceed more vigorously in alkaline solutions. The ferments are derived from the damp soil adhering to the roots, a fact which has been proved experimentally by the author. Portions of this soil were added to a solution of sugar, made alkaline with lime at 35°–40° C., when brisk butyric fermentation set in, much hydrogen and carbonic acid being evolved. The combustible gases arising from the diffusion batteries contained 30–70 per cent. of carbonic acid and 20–35 per cent. of hydrogen. The addition of phenol or of ammonium fluoride was of no avail, but sterilisation was accomplished by raising the temperature to 78° C. or above.—A. R. L.

#### *Filtration of Diffusion Juice after Heating.* Gaz. cukr. 1, 190.

THIS process having been carried out in a certain sugar factory, it is found that the difference in the purity-quotient of the juice before and after filtration never exceeds 0.26. It is probable, however, that the filtration exerts a favourable influence on the final product, inasmuch as a certain amount of melassigenic proteid substance is removed by it. Double filtration, before and after the heating, is still more serviceable; in this way the incrustation of the walls and steam pipes of the heating vessels by impurities is prevented and the heating effect increased.—A. R. L.

#### *Purification of Diffusion Juice.* C. Liedtke. Gaz. cukr. 1, 237.

WHEN diffusion juice is not previously warmed, it filters with extreme slowness, so that a great filtering surface is necessary, and being thus exposed to the air may become acid and ferment. The author proposes to add defecation mud to the diffusion juice to impart a certain alkalinity to it. Various quantities from 0.5 to 3.5 per cent. of defecation mud were added to the juice, which was filtered direct at a temperature of 44° C. and also at 75° C. In the former case filtration proceeded slowly, in the latter fairly quickly; in both cases the filtered juice was sparkling and

did not undergo change when exposed to the air. The purity quotient rose 2–3 per cent. Diffusion juice treated in this way contains little proteid matter and pulp, and saturates more quickly. After the first saturation it yields as pure a liquor as can be obtained by filtering thin liquor over animal charcoal.—A. R. L.

#### *Rate of Filtration of Solutions of Sugar.* Brendel. Zeit. Ver. Rubenzuck.ind. 43, 1088.

THE author's experiments were made with filter paper, using a 60 per cent. solution of pure saccharose. He finds that the rate of filtration increases with the temperature, the increase being the greatest between 0 and 30° C., a fact of considerable importance in carrying out Soxhlet's process of refining.—A. R. L.

#### *Precipitates formed during the Heating of Beet Juice.* A. Herzfeld. Zeit. Ver. Rubenzuck.ind. 43, 1065.

THE author has previously observed (this Journal 1893, 1065) that the precipitates which form on heating crude diffusion juice contain from 10 to 20 per cent. (on the dry substance) of proteids. He now recognises the presence of large quantities of pectous substances, fatty acids, oxalic acid, lime, and magnesia. Phosphoric acid is perhaps also an occasional constituent. No optically active compounds could be detected in the precipitates; it is pointed out, however, that such substances may have been originally present.—A. R. L.

#### *Recovery of Nitrates in Sugar Factories.* Gaz. cukr. 1, 125.

IX Tschernigoff the soil is very rich in salts, especially nitrates, and the beetroots grown there assimilate the latter, which in the course of manufacture passes into the juice. A rainy season tends to increase the quantity of nitrates in the roots. The nitrates remain unaltered during the process of manufacture and occur in the massecuite. Such a massecuite, containing nitrates, does not differ in appearance from an ordinary massecuite. In order to free the sugar from nitrate, the massecuite is covered cold in the centrifugal; that is, very little steam is employed, so that the nitrates, being the more readily soluble, pass into the syrup, and the sugar remains pure enough to be sold to refiners. The nitrates are similarly removed from the after-products. In most factories nitrates occur in the molasses, which latter are sold very cheaply. In order to obtain nitrates from the third product, which is sometimes done, this is dissolved in lukewarm water to a density of 4° Baumé, the solution heated to 60°–70° C., rapidly transferred into flat receptacles, where it is kept for 8–10 days at a temperature of 2–3° C. or –2 to –3° C. A mixture of sodium and potassium nitrates deposits, whilst the supernatant liquor which still contains nitrates is again subjected to the same process.—A. R. L.

#### *Mucilaginous Beet Juice.* Anderlik. Zeit. Zuckerind. Bohm. 13, 190.

THE cause of the juice becoming mucilaginous is a kind of fermentation, the products of which are dextrose, mannitol, non-volatile acids, and carbonic acid. There are also formed organic substances not precipitable by basic lead acetate; these latter may be identical with those occurring in decomposed beetroots.—A. R. L.

#### *Continuous Saturation.* Roboux. Sucri. indigène, 43, 179.

FOR successfully conducting this process, it is necessary to charge the apparatus with a certain maximum of juice at a temperature of 80° C., and to employ gas containing 30 per cent. of CO<sub>2</sub> or preferably a higher percentage. For simplicity, speed, and cleanliness the process is said to surpass all others.—A. R. L.

*Precipitates obtained in the Evaporating Apparatus used for Cane Sugar Juice.* Shorey. *Sugar Cane*, 26, 67.

The author finds that the above-mentioned precipitates often contain considerable quantities of citric acid, which fact is noteworthy inasmuch as Prinzen-Geerlings has not detected this acid among the products of the manufacture of sugar in Java.—A. R. L.

*Control of Beetroot-Sugar Manufacture.* Battüt. *Bull. l'Ass. des Chim.* 11, 478.

The control of the working of a beetroot-sugar factory as it is at present conducted is, according to the author, insufficient. The following important facts are left unexplained and completely ignored:—The amounts of sugar found in the diffusion juice, exhausted beetroot slices, and waste waters are generally far below that found in the roots themselves; whilst the amount of sugar found in the massecuite and defecation mud is below that found in the diffusion juice. The lime and pectous substances may, at all events, play a decided role here.—A. R. L.

*The Manufacture of Potato Starch by the Continual-Flow System.* Boudonneau. *Bull. Soc. d'Encouragement*, 1893, 849—851.

Most potato starch manufactories produce, apart from first-quality starch, considerable quantities of an inferior (coloured) grade, the necessity for so doing arising in great measure from the faulty process employed. Coloration of the starch is due to various causes, first and foremost being the ready decomposition of the pectous bodies existing in the potato-juice into gelatinous products in which the starch and pulp become entangled, forming greasy masses that are very liable to lactic and butyric fermentation. Another factor is the nature of the water used. The lime salts in the latter react with the soluble phosphates of the juice, forming a deeply-coloured and very light precipitate of calcium phosphate. It is well, therefore, to employ the softest water obtainable.

In the majority of cases the process of purification adopted consists in allowing a dilute mixture of the crude starch with water to settle in tanks, the operation being repeated several times. The separation thus effected is, however, by no means complete. Only the lower portion of the deposit consists of pure starch; the middle portion is contaminated with coarse fibrous matters, removable, however, by means of sieves; whilst the upper layer consists of small starch granules and all the finer impurities, and when worked up, only yields an inferior grade of starch. The production of "seconds" may be entirely avoided, however, by a process employed at Chalons-sur-Saône. Instead of settling tanks, a trough is used, 35—40 m. long, 1.5 to 2 m. wide, and 35—60 cm. deep, constructed of masonry, or preferably of wood lined with zinc. Along this trough, the cream of starch is caused to flow continuously in a layer 4—5 cm. deep, and at a rate not less than 5 m. per minute. Under these circumstances, the starch settles with great compactness, and so rapidly, that by the time the stream has reached the end of the trough, very little is left in suspension. On the other hand, the impurities, being lighter, have not time to settle and are swept on by the current. The deposit obtained, when submitted to a second similar treatment, yields perfectly pure starch. The advantages of the process are—

1. Rapid separation of the starch without prolonged contact with water.
2. Nearly the whole of the starch is obtained in the form of "extra superior" quality.
3. The necessity of working up inferior grades disappears.

—H. T. P.

*Compounds of the Sugars with Polyhydric Phenols.* E. Fischer and W. L. Jennings. *Ber.* 27, 1355.

See under XXIII., page 765.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*The Formation of Cane Sugar during the Germination of Barley and during the Ripening of the Apple.* L. Lindet. *Bull. Soc. Chim.* 1894, 11, 18—21.

DURING the germination of barley, the cane sugar it contains gradually increases in amount and the starch simultaneously decreases. Apples, during the ripening process, become richer in cane sugar and invert sugar, also with a simultaneous decrease in the amount of starch. The same occurs during the artificial ripening produced by storing. When the starch is reduced to less than about 2 per cent. the percentage of cane sugar diminishes, while that of the invert sugar increases.—J. G. W.

*The Formation of Mannite in Wines.* H. and A. Malbot. *Bull. Soc. Chim.* 1894, 11, 87—89, 176—179, and 413—415.

The presence of mannite in wines—at first taken as a proof of adulteration and afterwards as only occurring in sour or diseased wines—has for some time been recognised as occurring in thoroughly sound wine, and can in no wise be looked upon as a sign of disease or non-keeping power in a wine. Various hypotheses as to its formation, have been put forward, but none of them cover all the cases in which it has been found. The author has obtained mannite in a wine made by very regular fermentation at 24°, the mannite appearing some time after fermentation had ceased. All the glucose had previously disappeared, but a not directly fermentable carbohydrate was still found, and the mannite was apparently derived from this. In another wine which contained the same carbohydrate, but which was highly sulphited, no mannite was formed. The author believes that strong fumigation or sulphiting will prevent the formation of mannite, if such formation is considered undesirable.—L. T. T.

*Diseases of Wine.* Kayser. *Bull. Soc. d'Encouragement*, 1894, 97—99.

THE ferments causing disease in wine may be classed under two heads, aerobic and anaerobic organisms.

*Aerobic ferments.*—*Mycoderma vini*.—This ferment chiefly attacks new wines, and is readily combated by excluding air from the casks. *M. aceti* favours thin, dry wines, converting their alcohol into acetic acid. Here also exclusion of air is the best preventive.

*Anaerobic ferments.*—The *viscous ferment* generally attacks new white wines containing but little alcohol. It forms chaplets of spherical cells imbedded in a gelatinous matrix. Tannin appears to check its growth, but wine when once affected by it never regains its original flavour and bouquet. The ferment of *bitter* ("amers") wines is found usually in old Burgundian wines. The wine becomes insipid, bitter, and somewhat sharp, and deposits a voluminous coloured sediment consisting of extremely thin immobile rods. According to Pasteur and Duclaux it converts the glycerin into acid.

The ferment producing effervescence (*vins poussés*) may occur in all species of wines, and is favoured by high temperatures. The wine becomes turbid and develops considerable pressure owing to the production of carbonic acid. The ferment occurs in tangled masses of thin filaments, and appears to grow at the expense of the calcium tartrate, which it converts in volatile acids.

The turned ("tourné") wine ferment resembles the preceding, except that it does not produce carbonic acid. It attacks the tannin and tartaric acid of the wine, the products formed being acetic, tartronic, and lactic acids.

The *mannite* ferment occurs only in imperfectly fermented wines when the temperature rises too high in the tuns (40°—55° C.). The consequent enfeeblement of the yeast allows the mannite ferment to act, which it does at the expense of the levulose present, the principal product being mannite, and to a smaller degree fixed and volatile acids.

As regards the general precautions to be adopted in the manufacture of wines, it is of importance to perform the operations of racking and priming at the right time. An infallible preventive, however, consists in heating the wine to 55° C. (pasteurisation).—H. T. P.

*The Chemical Properties of the Alcoholic Extract of Beer-yeast.* J. de Rey-Pailhade. *Comptes rend.* **113** (1894), 201—203.

The chemical phenomena produced in living organisms are caused by proximate principles (*principes immédiats*), generally unstable, which are often destroyed by the action of the solvents used to extract them. It is nevertheless possible, by the aid of low temperatures and certain feeble antiseptics, to obtain solutions which, although deprived of every organised element, produce chemical phenomena characteristic of life. Such a solution may be prepared by mixing 100 grms. of fresh pressed beer-yeast with 55 grms. of water containing a little glucose, and then adding gradually 45 grms. of 90 per cent. alcohol. The mixture is kept corked up in a flask at a temperature of 0 for three days and then filtered. The filtrate is sterilised according to d'Arsonval's method, and is placed in an exhausted receiver to remove excess of dissolved carbonic acid. It is slightly acid, and although it is deprived of all living organisms, it possesses the following chemical properties, which also belong to living yeast:—It produces sulphuretted hydrogen on contact with sulphur in the cold; in the absence of oxygen it spontaneously sets free carbonic acid; and it absorbs oxygen from the air.

Spallanzani showed that tissues recently detached from the living organism absorb oxygen and produce carbonic acid. Some years ago the author showed that tissues when pounded with sulphur produce sulphuretted hydrogen. His present researches show that the proximate principles themselves produce these phenomena when they have been extracted by suitable solvents, and that these effects appear to be independent of any organised element.—D. E. J.

*The Multirotation of Sugars.* P. Th. Müller. *Comptes rend.* 1894, **118**, 125.

See under XVI., page 748.

*New Apparatus for Fractional Distillation.* E. Varenne. *Bull. Soc. Chim.* **11**—**12**, 1894, 289—292.

See under XXIII., page 761.

*The Ebullioscope and its Use for Estimating Alcohol in Beers.* H. Tornøe. *Bull. Soc. Chim.* **11**—**12**, 1894, 116.

See under XXIII., page 762.

*Optical Titration of Mannitol.* J. A. Müller. *Bull. Soc. Chim.* **11**—**12**, 1894, 326—329.

See under XXIII., page 767.

*Spontaneous Formation of Pyruvic Acid from an Aqueous Solution of Tartaric Acid.* R. Otto. *Ber.* **27**, 838.

See under VII., page 733.

#### PATENTS.

*An Improved Distilling Apparatus.* A. Lavy, Paris, France. Eng. Pat. 12,528, June 26, 1893.

THE apparatus comprises an ordinary boiler and a dome-shaped rectifier firmly attached to the top of the latter. The rectifier is divided by horizontal partitions into a series of chambers, each one of which is again separated into two

compartments by an intermediate corrugated partition. The wine, &c., to be distilled circulates through the outer pipe of a double coil arranged round the outside of the dome, and is discharged into the upper half of the first chamber, from whence it flows by an overflow pipe to the upper division of the second, and so on, finding its way at length into the boiler. The alcoholic vapour rises through a central pipe, which connects together the lower portions of all the chambers and finally passes into the inner tube of the coil mentioned above, where it is condensed. It will be seen that the use of cooling water may be entirely dispensed with, the wine itself being made to do the work of condensation. The apparatus is adapted for continuous distillation, and is claimed to yield rectified brandy at one operation.—H. T. P.

*Improvements in and Apparatus for Sterilising or Preventing Fermentation or Acidification of Beers, Beverages, or the like.* P. W. May and R. E. R. Newlands, London. Eng. Pat. 12,609, June 27, 1893.

THE beer or other liquid, previously aerated or charged with carbonic acid, and contained in stoppered bottles or other vessels, is gradually heated to the requisite temperature in a strong water-bath provided with an air-tight cover, and again slowly cooled. Heating may be effected by any of the usual means, and during the operation a considerable pressure is maintained in the water-bath to counteract the internal pressure in the bottles, &c., and prevent their rupture.—H. T. P.

*New or Improved Process and Apparatus for Purifying Liquids.* L. Wagner and J. Marr, Baltimore, Maryland, U.S.A. Eng. Pat. 7027, April 9, 1894.

THE object of this invention is to destroy and remove the organisms from liquids and beverages, such as beer, wine, water, &c. For this purpose a process of electrolysis and filtration is resorted to. The apparatus employed consists of a covered cylindrical outer vessel, inside which a drum constructed of perforated sheet metal covered with wire gauze, and capable of being rotated on its axis, is supported on a vertical shaft. In operation, the liquid to be purified is discharged into the drum, the latter being rotated at a high speed (1,000 revolutions), and submitted to the action of an electric current supplied through carbon electrodes suspended near the bottom of the drum by means of rods attached to the cover of the apparatus. The liquid, passing through the walls of the drum, filters through a filter-bed composed of fragments of sponge clamped between metal plates, situated at the lower part of the outer vessel. During the process, air, sterilised by passage through a furnace, and subsequently cooled and filtered through cotton wool, &c., is supplied to the apparatus.—H. T. P.

*Improvements in the Manufacture of Spirits, and in Apparatus for that Purpose.* J. J. Murphy, Cork. Eng. Pat. 7858, April 20, 1894.

IN order to prevent the excessive production of fusel oil and other by-products during fermentation, the temperature of the liquid in the vats is not allowed to rise above 72° F., attenuating coils being used to regulate the heat if necessary. An aerating device is also provided in the shape of a pipe perforated with holes, placed near the bottom of the vat, by means of which air may be pumped through the liquid.—H. T. P.



# XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY AND WATER PURIFICATION, DISINFECTANTS.

## (A.)—CHEMISTRY OF FOODS.

*Estimation of Fat in Cheese.* S. Bondzynski. *Zeits. anal. Chem.* **33**, 186.

See under XXIII., page 766.

*Drinking-Water obtained from Artesian Wells at Mantua.* P. Bonato. *Annali della Soc. degli Ing. e degli Arch. Ital.* 1893, 399. *Proc. Inst. Civil Eng.* **116**, 1894, 34—35.

MANTUA, a town of 30,000 inhabitants, formerly derived its entire water-supply from surface-wells, vitiated by the contents of the innumerable cesspools necessitated by the absence of any systematic collection and disposal of sewage. To amend this, after consideration of various sources of supply, it was decided to sink a number of artesian wells. The first instalment of the work comprised four such wells, which were sunk to the respective depths of 398 ft. 6 in., 398 ft., 351 ft., and 382 ft. 6 in. From these an excellent supply, at the rate of 28.6 gallons each per minute, was delivered to a height of 13 ft. 3 in. above the street datum level. Following this, ten other wells were sunk, the average delivery being as follows:—

Height above Datum in Feet.	Delivery in Gallons per Minute.
2' 62	41' 0
6' 24	35' 2
9' 52	26' 4
12' 78	14' 3
16' 06	1' 5
16' 49	"

The total supply is equivalent to an allowance of 14.3 gallons per head. Each well cost 300*l.*, the total work therefore being carried out for 4,200*l.*

The tubes are of mild steel,  $3\frac{1}{2}$  in. external diameter, 0.35 in. thickness of metal, in length varying from 7 ft. 3 in. to 19 ft. 6 in., screwed together, and sunk by an 8-cwt. steam-hammer, with a fall of 2 ft. 6 in. at 39 blows per minute. Each tube was sunk in from 20 to 30 days. The water was submitted to severe chemical and bacteriological analysis. It is perfectly clear, and has an average temperature of 58.7° F. At first there is a slightly perceptible but very transient sulphurous odour. The water contains, however, not the slightest trace of sulphuretted hydrogen, and it is considered probable that the peculiarity is simply the effect of the contact of the water with the steel tube, the pressure at the bottom being equal to 176 lb. per sq. in.

## PATENTS.

*Improvements in the Preparation and Treatment of Animal and Vegetable Substances and Fruit, and Compounding the same with other Ingredients for Use as Human Food.* L. C. Tipper, Yardley, Worcester. Eng. Pat. 13,600, July 13, 1893.

CHOPPED meat, either raw or cooked, is appropriately seasoned, mixed with flour or other farinaceous material, and made into a dough with new milk, to which there has been added gelatin containing borax (3 ounces of gelatin, 1 ounce of borax to 12 gallons of new milk for 500 lb. of meat and

flour). The mass is rolled into sheets, made into biscuits, dried, baked, and coated with gelatin, or gelatin and borax, to preserve them from the air.

Fish, vegetables, cheese, and fruit may be similarly treated.—A. G. B.

*Improvements in and relating to the Manufacture of Food for Horses.* H. H. Lake, London. Eng. Pat. 13,704, July 14, 1893.

The food is composed of blood, extract of meat, and Iceland moss. For the last named, chaff or chopped straw, treated as described for the Iceland moss, may be substituted.

The blood is from healthy oxen; it is strained, mixed with 2 per cent. of pure pepsin, and 0.1 per cent. of pure dilute hydrochloric acid; the mixture is kept at 37° C. for eight hours, being repeatedly stirred during this time.

The meat is freed from fat and bone, and is thoroughly beaten with a heavy hammer on a hard wooden block. One part by weight of the mass of meat is placed in twice its weight of water, together with 3 per cent. of its weight of dried melon leaves, and 0.1 per cent. of its weight of hydrochloric acid. After eight hours, the steeped meat is gradually heated in a boiler to a "boiling heat," the process preferably lasting eight hours, the boiling proper being continued for half an hour. The extract of meat thus produced is filtered.

Three kilos. of the washed Iceland moss are steeped in 70 litres of water, containing 180 grms. of potash, for 24 hours, being repeatedly stirred. The colouring matter and bitter principle of the Iceland moss having been thus extracted, the moss is washed and spread on boards to dry.

Two parts of the blood are mixed with one part of the extract of meat, and one kilo. of the Iceland moss is added to three litres of the mixture. When the moss is completely saturated, it is spread on boards and dried for 12—14 hours in a drying chamber. It is then sufficiently brittle to be ground to a fine powder, which is mixed with moistened oats, bran, or other usual food for horses. The condition of horses is stated to be much improved by this food.

—A. G. B.

*Improved Baking Powder.* W. Brothers, Rawtenstall, Lancaster. Eng. Pat. 16,458, September 1, 1893.

AMMONIUM hydrogen carbonate is reduced to a fine powder and mixed with some inert material, such as rice-flour or similar substance, to increase its bulk.—A. G. B.

*An Improved Composition for Use as Food, and Method of Preparing the same.* E. O. Taffin, Stockholm, Sweden. Eng. Pat. 7543, April 16, 1894.

HEMP-SEED extract is mixed with malt and malt extract, cacao-powder being added if it be desirable that the compound should taste of cacao. The hemp-seed extract is obtained by the use of petroleum ether. The malt for the preparation of this food must be dried at a low temperature (50°—60° C.), and the brining and malting must continue during a longer time than is the case in common malt preparation. Wheat and Indian corn are the most convenient kinds of corn. Only part of the malt is mashed and treated with hot water; the solution is evaporated to a syrup. Another part of the malt is ground to meal. The hemp-seed extract and the malt extract (equal parts, for instance) are mixed, and so much malt-meal is added that the whole obtains the consistency of dough; the mass is pressed into cakes and dried; these cakes are easily ground to meal. The cacao-powder is incorporated together with the malt-meal. Cacao-powder may entirely replace the malt-meal. The proportions are varied, according to the taste required; if the bitterness of the hemp-seed extract is to be entirely masked, the proportion of this extract to the malt extract should be 1:4. The food is a suitable substitute for tea and coffee, and is prepared in the same manner as ordinary cacao is prepared.—A. G. B.

## (B.)—SANITARY CHEMISTRY AND WATER PURIFICATION.

*Hydrogen Dioxide in Atmospheric Air and in Aqueous Deposits therefrom.* I. v. N. Hlosva. Ber. 1894, 27, 920—925.

In this paper the author answers the criticism which Schöne has recently passed (Ber. 1893, 26, 3011) upon his (the author's) work which was performed in 1889 (Bull. Soc. Chim. 2, 347, &c.). This work showed that an attitude of suspended judgment is necessary with respect to the assertion that air, rain, and dew contain hydrogen dioxide and ozone.

As reagents for ozone the author used thallous hydroxide and  $\alpha$ -azomethylamine benzene-sulphonie acid, both of which are perfectly trustworthy under the prescribed conditions. Reliance must not be placed on a mixture of potassium iodide, starch, and ferrous sulphate as a test for hydrogen dioxide. Titanic acid in strong sulphuric acid is the most characteristic reagent for hydrogen dioxide, and chromic acid with ether holds the second place; if these reagents will not detect hydrogen dioxide in rain water it must be concluded that the quantity present is below the limit of sensitiveness. There is an oxidising agent present in rain which, whatever its precise nature may be, behaves ultimately as nitrous acid, and liberates iodine from potassium iodide even in the presence of starch.

Schöne himself draws attention to the unreliability of the guaiacum-diastase reaction in the presence of ammonia—another reason for rejecting the evidence of this reagent, since the role of the ammonium hydrogen carbonate in rain water with respect to the reagent, is not known.

There is no doubt but that air contains nitric peroxide as a constant constituent, probably formed during combustion. If a 1 per cent. solution of pure potassium iodide be exposed to the air, even in the dark, iodine will be detected therein by starch after a time; and by warming the liquid until the blue has disappeared, and applying Griess' reagent, nitrous acid will be found to be present. (The starch gave no reaction with Griess' reagent, nor could any sulphur dioxide be detected in the air.) Does not this prove the presence of nitrous acid in the air? Even supposing that the air contains its nitrous acid only as ammonium nitrite, as Schöne contends, there is no reason to suppose that this will not eventually liberate iodine from potassium iodide. When, to a solution of potassium iodide and potassium nitrite, carbon dioxide solution is added, iodine will be liberated, and can be detected by starch; in this way <sup>TABLEAU</sup> part of nitrous acid will be detected in the course of 60—80 minutes. It is hardly surprising, then, that Schöne should obtain such a reaction in rain water, which avowedly contains ammonium nitrite, in the course of 6—8 hours.

That there is an oxidising agent in the air, apart from the oxygen, no one denies, but heretofore the action of nitric peroxide has been overlooked, and, unless it be first removed, the presence of ozone and hydrogen dioxide cannot be affirmed. The author used a 4 per cent. solution of sodium hydroxide for removing the nitrous acid: in such a solution, if free from nitrite, hydrogen dioxide in small quantity does not decompose, even after days, and may be recovered by distillation. Therefore, the contention that if the hydrogen dioxide in the air be in excess of the nitric peroxide it should be possible to detect it in the caustic soda solution, is valid. Obviously, to look for hydrogen dioxide in rain water in which nitrites can be detected is absurd.

It is not necessary to suppose that because nitrate as well as nitrite is found in the dilute caustic soda through which air is passed, the nitrite has been oxidised by ozone or hydrogen dioxide in the air. It is well known that nitric peroxide in presence of oxygen and water is largely converted into nitric acid. Moreover, a dilute potassium nitrite solution containing nitric peroxide will certainly no nitrite after a month.

To render his 1 per cent. solution of potassium iodide more sensitive to ozone, the author provided for the potassium hydroxide which would be liberated, by the addition of sufficient phosphoric acid to change litmus

paper feebly red. When air, freed from nitrogen oxides, is passed through such a dilute solution of hydriodic acid, iodine is not liberated even after weeks.

The statement that atmospheric ozone is more abundant in summer is curiously in accord with the fact that nitric peroxide is more abundant during the summer months.

—A. G. B.

*On the Question of Atmospheric Hydrogen Dioxide.*

E. Schöne. Ber. 1889, 27, 1233—1235.

This paper is a reply to Hlosva (see preceding abstract). The real basis of the dispute is the question whether a mixture of potassium iodide, ferrous sulphate, and starch, and a mixture of guaiacum and diastase are characteristic reagents for hydrogen dioxide or not. Hlosva is in error when he states that oxides of nitrogen or neutral nitrites give the same reactions as are given by hydrogen dioxide. It is by no means easy to refute Hlosva's statements, but the author claims this has been done in a paper published at the beginning of the year (Zeits. Anal. Chem. 1894, 33, 137). In the same paper it is also shown that thallous hydroxide is not a perfectly trustworthy reagent for ozone, and that carbon dioxide in the presence of ammonium nitrite cannot give a reaction with potassium iodide, in presence of ferrous sulphate and of starch, which can be mistaken for that of hydrogen dioxide.—A. G. B.

*The Electrical Purification of Sewage on the "Hermite" System.* Georges Vitoux. La Revue Techn. 1894, 106. Proc. Inst. Civil Eng. 113, 1894, 39—40.

An account is given of a small plant erected at the Figaro offices in Paris for the trial of this process, which is already in successful operation at Brest and other towns in France, and at Worthing in this country. A current of electricity is employed to decompose an aqueous solution of certain soluble chlorides, preferably magnesium chloride, or as mixture of this salt with sodium chloride in the proportion found in sea-water or in salt-lye. By this means, at the positive extremity a very unstable compound of oxygen and chlorine is produced, which possesses powerful oxidising properties, while at the other pole an oxide is formed which is capable of precipitating the various organic substances present in sewage-water. It thus becomes possible to destroy entirely the products of putrefaction, together with the foul gases, such as the sulphuretted and carburetted hydrogen, as also the germs and microbes which may be present, while, owing to the precipitation which takes place of the albumenoid matters, the foul liquid is immediately clarified. The disinfecting liquid, as fast as it is prepared, is pumped up from the central producing-station to the closet basins or other places when it has to be used, having therefore the freshly-formed chemical compounds in the nascent state, and capable at once of attacking the various impurities. This latter apparatus consists of a vessel constructed of galvanised east-iron, having in the lower part of it a tube perforated with a number of holes and furnished with a zinc tap. The sea-water or other solution of chlorides enters through this tube. In the upper part of the vessel, also formed of galvanised east-iron, and widened out to serve as a trough or channel for the decomposed liquid, which rises to this point, is a pipe through which this liquid is drawn off for use, and thus a constant circulation of the contents is effected. The negative electrodes consist of a number of zinc discs, mounted on two horizontal shafts passing through the lower part of the vessel, which shafts are caused to revolve slowly. Between each pair of zinc discs are placed the positive electrodes, the active surfaces of which consist of sheets of platinum-gauze, held in position by a framework of ebonite, which ensures the necessary rigidity. The upper part of the gauze is soldered to a plate of lead, which is perfectly isolated. Every positive electrode communicates by means of the lead with a copper rod traversing the electrolyser, and contact is made by a screw nut. The copper bar which carries the positive electrodes communicates with the positive pole of the dynamo. The current is conveyed to each of the platinum

electrodes and passes thence, through the liquid to the zinc discs, serving as the negative electrodes: these are in contact, through the iron containing-vessel, with the negative pole of the dynamo. Arrangements are made for the mechanical cleaning of the zinc discs. Currents are employed of from 1,000 to 1,200 amperes. The liquid, as it issues from the electrolyser, has a faint odour of chlorine and is ready for immediate use. For this purpose it may be pumped from the central station to the points where it is required.

#### PATENT.

*New or Improved Process and Apparatus for Purifying Liquids.* L. Wagner and J. Marr, Baltimore, Maryland, U.S.A. Eng. Pat. 7027, April 9, 1894.

See under XVII., page 751.

#### (C).—DISINFECTANTS.

*The Disinfecting Power of Soap Solutions in respect of Cholera Germs.* Maximilian Jolles. Zeits. für Hygiene, 1893, 460. Proc. Inst. Civil Eng. 116, 1894 [ii.], 48—49.

To determine the value of soap-solutions in the disinfection of materials containing the germs of cholera, five different sets of experiments were undertaken with each of the following kinds of soap, viz., potash washing-soap, potash lysol-soap, glycerin-soap, Leda toilet-soap, and shaving-soap. Detailed analyses are given of each of these substances, the total alkalis in which varied from 10.4 to 7.3, while they contained from 0.009 to 0.065 per cent. of free alkali. In the original series, each solution was tested at 10 different degrees of strength, from 1 up to 10 per cent. In carrying out the tests 100 cub. cm. of soap solutions were introduced into sterilised Erlenmeyer flasks provided with cotton-wool stoppers. These flasks, with contents, were exposed on three separate days for half an hour each day to the action of the Koch steam-bath and cooled down to 15° C. A 10 per cent. meat-juice bouillon, infected with cholera bacteria, and kept for five days at a temperature of 25° C. was also prepared. Twenty cub. cm. of this culture was introduced into each of the flasks, and, after agitation of the contents, three specimens from each flask were taken by means of a pipette at intervals of 10, 30, and 60 minutes: also in 6, and 24 hours, and cast into sterile plates of gelatin for cultivation. Control tests were also made from similar flasks filled with 100 cub. cm. of sterilised distilled-water, infected in a similar way with a cultivation of cholera-bouillon. The cultures took place at a uniform temperature of 25° C. The experiments indicated that, after the lapse of a given time, even the 1 per cent. solution proved fatal to the cholera germs. In respect to their energy as disinfectants against cholera bacteria, these soaps differed but little. They are all equally capable of being used for this purpose in each case where soap solutions can be employed for disinfection. The advantages they possess over other disinfectants consist in their ready applicability and in their complete freedom from danger.

#### PATENTS.

*Manufacture of Oleaginous or Saponaceous Compounds suitable for producing Medicinal Emulsions for Absorption by the Skin, or for Disinfectants.* F. W. Klever, Brandenburgerstrasse, Cologne. Eng. Pat. 12,446, June 24, 1893.

THE object of this invention is to impart to certain medicaments, such as creosote, camphor, chloroform, eucalyptus oil, and to disinfectants, a permanent or increased capacity for forming emulsions, in order to facilitate their absorption by the skin. Mineral oils (sp. gr. 0.900) are treated with oxygen, preferably under a pressure, varying from 7 to 20 atmospheres, according to the nature of the oil, at a temperature of 150°—200° C., until the oil is oxygenated. Then alkalis (preferably soda or ammoniac) are added in quantity not exceeding 5 to 7 per cent. of the oil, and to prevent the oil becoming thick, a small quantity of alcohol, not exceeding 1½ per cent., may

be added to it. The product is capable of forming, with water an emulsion, from which the oil will not separate; in this state it will dissolve certain medicaments and disinfectants which have hitherto been considered to be insoluble, thus rendering them capable of being readily absorbed by the skin.—J. J. K.

*A New and Improved Hydrostatic Apparatus for Automatically Mixing with Water, or other Liquid, Disinfectants, Softening or Purifying Substances, or Saturated Solutions of any Soluble Substances in Definite Proportions.* F. Spence, Newcastle. Eng. Pat. 13,146, July 5, 1893.

See under I., page 713.

*An Improvement in what is commonly called Dry Soap or Soap Powder.* W. H. Horton, Manchester. Eng. Pat. 8676, May 2, 1894.

See under XII., page 746.

## XIX.—PAPER, PASTEBOARD, Etc.

#### PATENTS.

*An Improved Machine for Straining Pulp and the like.* P. Reinicke, Cöthen, Germany. Eng. Pat. 11,531, June 12, 1893.

See under I., page 713.

*Improvements in Apparatus for Mixing and Regulating Paper Stuff.* W. Borland, Currie, Midlothian. Eng. Pat. 12,420, June 24, 1893.

IN the apparatus for regulating the density of the paper pulp an agitator is placed in a vessel separated from the hydrometer-vessel, the hydrometer regulating the required quantity of size-water.—H. S.

*Improvements in or connected with Boilers for Boiling or Chemically Treating, and for Washing Esparto and other Grasses, Straw, Wood, Rags, and other Fibrous Materials for Paper-making.* G. Sinclair, Leith. Eng. Pat. 13,175, July 6, 1893.

AN addition to the now well-known "Sinclair" boiler (Eng. Pat. 2035, 1880; 6441, 1893) of an inlet pipe (steam), whereby a circulation of the boiling liquor in the reverse direction is determined, i.e., upwards through the fibrous material and returning *via* the vomiting pipe. An injector is also added in connection with this pipe for the admission of air or water. The patentee "claims" the additional inlet pipe and injector as described and figured (*q.v.*).

—C. F. C.

*A New or Improved Material capable of being used in Substitution for Leather and for Ordinary Paper.* N. Kinnagaya, Osaka, Japan. Eng. Pat. 19,481, October 17, 1893.

STRONG paper, such as made from Manilla hemp or from fibres of the kozo tree of Japan, is mixed or impregnated with gelatinous matters during, or subsequent to, its manufacture. The paper sized in this manner with gelatin is brushed or otherwise wetted with a solution containing tannic acid and dried. This operation is repeated two or three times, the tannin liquid employed being a solution made from persimmon, or an oak-bark solution of a strength of from 25 to 33 Tw. The material, after having been finally dried, may be employed as a substitute for leather or ordinary paper for various purposes, e.g., for wrappers and linings, for covering the rollers used in spinning machinery, &c.—H. S.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Mercurous Iodide obtained Crystallised by the Wet Process.* François. Jour. Pharm. et de Chim. [5], 29, 67.

50 GRMS. mercuric iodide, 100 aniline, and 200 of alcohol of 90 are heated until solution is effected; the liquid is filtered and allowed to stand some days. Crystals of diphenylmercuriodiammonium iodide separate; to the clear filtrate from these, 350 grms. of commercial ether of 65 are added, and the whole allowed to stand for a week. Owing to the presence of aldehyde in the ether the mercuric iodide present becomes reduced to mercurous iodide, which separates in yellow scales. After washing successively with cold alcohol, boiling alcohol, and ether, the salt is obtained pure; it sublimes in orange-yellow crystals. Treated with potassium iodide under the microscope it dissolves with separation of shining globules of mercury.—C. R. A. W.

*Chloralose.* M. Hanriot and C. Riche. Bull. Soc. Chim. 1894, 11, 37—44.

CHLORAL unites with glucose, forming well-crystallised compounds. These were first obtained by Heffter in 1889.

The method of preparation consists in heating on the water-bath a mixture of equal parts of glucose and chloral, both in the anhydrous condition. A vivid reaction sets in and a readily soluble, vitreous mass is produced. From this the uncombined chloral is removed by boiling with water. The concentrated aqueous solution deposits a crystalline mass, from which, after drying and repeated washing with hot ether, boiling alcohol extracts a substance crystallising in fine iridescent scales, which by the authors is called para-chloralose. By exhausting the aqueous solution, from which the para-chloralose has been separated, with the ether used for washing the latter, and evaporating the ethereal solution, beautiful needles of chloralose are obtained. The composition of both these compounds corresponds to the formula  $C_6H_{11}Cl_3O_6$ .

*Chloralose* melts at  $187^\circ$ , is fairly soluble in water, alcohol, and ether, slightly soluble in chloroform, and almost insoluble in petroleum. It does not reduce Fehling's solution or silver nitrate. Dilute acids split it up only after long continued boiling. By alkalis it is rapidly decomposed on heating. It combines neither with phenylhydrazine nor with hydroxylamine. By acetyl and benzoyl chlorides it is readily converted into the corresponding tetra-derivatives, and with sulphuric acid it forms the unstable compound  $C_6H_9Cl_3O_4(SO_4H)_2$ .

*Chloralic Acid*,  $C_6H_5Cl_3O_6$ .—Obtained by adding 50 grms. of nitric acid, and subsequently a solution of 20 grms. potassium permanganate to 30 grms. of chloralose dissolved on the water-bath in one litre of water. From the residue resulting by the evaporation of the ethereal extract of the solution concentrated *in vacuo* the sodium salt is prepared. This, after washing with ether, is decomposed with dilute sulphuric acid, the free chloralic acid being subsequently extracted by means of ether, from which it crystallises in fine needles melting at  $212^\circ$ .

Chloralose is a powerful hypnotic. Its ethers, as well as chloralic acid, are physiologically inactive.

*Para-chloralose* crystallises in shining prisms, melting at  $227^\circ$ . It is almost insoluble in water, chloroform, and ether, but dissolves pretty freely in boiling alcohol and in caustic potash. With Fehling's solution, silver nitrate, dilute acids, phenylhydrazine, and hydroxylamine it behaves in the same manner as chloralose. By alcoholic potash it is rapidly decomposed. Like chloralose it combines with acetyl and benzoyl chlorides and with sulphuric acid. The acetyl and benzoyl derivatives are less stable than the corresponding chloralose compounds, while the sulphuric ether shows a somewhat higher degree of stability than that of chloralose.

*Para-chloralic Acid.*—Prepared in much the same manner as chloralic acid. Large crystals containing two molecules of water. Melting point  $202^\circ$ . Extremely soluble in alcohol. The sodium salt crystallises in lustrous scales.

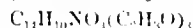
Para-chloralose and its derivatives possess no hypnotic properties.—E. M.

*Chloralose.* M. Hanriot and C. Riche. Bull. Soc. Chim. 11—12, 1894, 303—305.

CHLORALOSE is soluble in alkalis, but does not combine with them to form compounds analogous to saccharates; thus ether dissolves it completely from potash solution, whilst the ammoniacal solution loses all ammonia by exposure to air, leaving only chloralose behind. No carbon dioxide is evolved from alkaline carbonates by chloralose. Neither chloralose nor para-chloralose (when pure) reduce Fehling's solution until decomposition has been more or less effected by the potash present; nor do dilute acids act on chloralose even after several hours heating; although, as shown by Petit and Polonowsky, more concentrated acids hydrolyse it into chloral and glucose. Saturated hydrochloric acid at  $120^\circ$  does not act thus upon it.—C. R. A. W.

*Researches on the Homologues of Gallanilide: Galliparalulide.* P. Cazeau. Bull. Soc. Chim. 11, 1894, 82—85.

WHEN equal weights of paratoluidine and gallic acid are heated together at  $150^\circ$  C., *galliparalulide*,  $C_{17}H_{15}NO_6$ , is formed. It crystallises with 2 aq. in white flakes, melts at  $211^\circ$  C., is soluble in alcohol, ether, and boiling water, very sparingly so in cold water. It loses its water of crystallisation at  $100^\circ$ . It is decomposed by hydrochloric acid at  $150^\circ$ ; alkalis appear to have no action on it even at  $100^\circ$ . It oxidises and becomes brown on exposure to air. With acetic anhydride it yields a *triacetyl derivative*—



crystallising in needles which do not melt without decomposition, and are soluble in alcohol and insoluble in water. The acetyl compound has no action on ferric chloride, whilst the base gives a blue colour therewith. A boiling solution of the toluidide yields with zinc acetate a *zinc salt*,  $C_{14}H_{11}NO_4Zn$ , which is insoluble in water. Corresponding *lead, barium, and calcium* salts were obtained.

Attempts to produce similar derivatives from orthotoluidine, methylalanine, dimethylalanine, and xylyline, have so far proved unsuccessful.—L. T. T.

*Germicidal Action of Gallanilide or Gallanol.* P. Cazeau. Bull. Soc. Chim. 11, 1894, 81—82.

IN conjunction with Nicolas, the author has investigated the germicidal effect of gallanilide (or gallanol) on the carbuncle bacillus, the bacillus staphylococcus, bacillus pyocyaneus, Eberth's bacillus, and the intestinal bacilli. With large doses of the germicide all the bacilli were killed; with small doses (1 in 1,000) the Eberth's bacilli were killed, the vigour and reproduction of the intestinal bacilli and the staphylococci was reduced, but no result could be observed on the other two species; with very small doses (1 in 5,000) no result was observed on the growth of the bacilli, but their pathogenic action was almost completely destroyed. G. Roux finds a similar destructive effect of this reagent on the cholera bacillus, but has not yet completed his researches.

Gallanilide acts like salicylic acid in preventing the putrid fermentation of eggs and blood, or the turning of milk, and has the advantage over salicylic acid of being non-toxic (unless injected directly into the blood) in doses of as much as 5 grms. The non-toxic effect is due to its insolubility in water.—L. T. T.

Pure *d*-Coniine. A. Ladenburg. Ber. 27, 858—859.

SPECIMENS of natural coniine yielded to the author specific rotatory powers not exceeding  $\alpha_D = 13.8^\circ$ , the value found for synthetic coniine; latterly a sample was obtained of higher value. By conversion into bitartrate and frequent crystallisation of the salt, the alkaloid ultimately regenerated was found to boil at  $166^\circ$ — $167^\circ$ , and to have the rotatory power,  $\alpha_D = 15.6^\circ$ , the value ascribed by Schiff to *d*-coniine. Pure *d*-coniine platino-chloride dissolves completely in alcohol-ether, and separates in a crystalline form on evaporation; whereas if impure, an oil first separates, only becoming crystalline on standing for some time.

—C. R. A. W.

Isoborneol. J. Bertram and H. Walbaum. J. Prakt. Chem. 1894, 49, 1—15.

WHEN camphene is warmed with a mixture of glacial acetic acid and a small quantity of a mineral acid (sulphuric, nitric, &c.), it is rapidly converted into the acetic ether of isoborneol,  $C_{10}H_{17}.COOC_{10}H_{17}$ , from which the solid alcohol, isoborneol,  $C_{10}H_{17}.OH$ , is readily formed by the action of alcoholic potash. The reaction occurs equally well whether the camphene be of natural origin or formed from bornyl chloride or from pinene hydrochloride.

Isoborneol differs from borneol in several marked ways, more particularly in readily losing the elements of water, reproducing the parent hydrocarbon; thus heating with zinc chloride or diluted sulphuric acid, readily forms camphene, under conditions where pure borneol is not all acted upon. Borneol melts at  $203^\circ$ — $204^\circ$ , isoborneol at  $212^\circ$ ; owing to extreme volatility, the melting point must be determined in a sealed capillary tube; for the same reason the boiling point cannot be determined; that of borneol is  $212^\circ$ . The solubilities in benzene and petroleum ether are different; and various differences in melting point, &c., are observed in the case of corresponding pairs of derivatives, such as the phenylurethanes, chloral compounds, bromal compounds, formic and acetic esters, and methylene ethers. On the other hand, by oxidation, each gives ordinary camphor, melting at  $177^\circ$ , and boiling at  $207^\circ$ — $208^\circ$ , and yielding an oxime, melting at  $118^\circ$ — $119^\circ$ . When isoborneol is dissolved in methylic or ethylic alcohol and warmed with sulphuric acid, the methylic and ethylic ethers are respectively formed, boiling at  $192^\circ$ , and  $203^\circ$ — $204^\circ$ ; borneol is unacted upon under similar conditions. Isoborneol is present to some extent in the crude borneol obtained by the reduction of camphor.—C. R. A. W.

The Occurrence of Camphene in Ethereal Oils. J. Bertram and H. Walbaum. J. prakt. Chem. 1894, 49, 15—19.

CAMPHENE is distinguished from all the other terpene isomerides by its solid form, and by being converted into isoborneol acetic ether by treatment with glacial acetic acid and a little sulphuric acid. By means of this latter reaction, its presence has been detected in various essential oils, the fraction passing over between  $155^\circ$  and  $165^\circ$ , or thereabouts, being treated with these acids, and the undissolved hydrocarbons, &c. separated. On adding water to the acid solution, an oily fluid separates, largely consisting of isoborneol acetic ether, and furnishing isoborneol on saponification with alcoholic potash. In this way citronella oil was found to contain notable quantities of camphene, not so easily separated as solid hydrochloride by dissolving in ether and saturating with gaseous hydrochloric acid. Oil of ginger (*Zingiber Officinale* Roscoe), Kessö oil (from Japanese valerian roots), and camphor oil were similarly found by this mode of treatment to contain, *inter alia*, small quantities of camphene.—C. R. A. W.

Presence of Camphene in Essence of Lavender.

G. Bouehardat. Bull. Soc. Chim. 11, 1894, 147—150.

By the distillation of large quantities of essence of lavender, the author obtained a small quantity of liquid boiling at  $165^\circ$ — $170^\circ$  C. from which, by means of the hydrochloride,

he succeeded in isolating a dextro-rotatory camphene, boiling at  $158^\circ$ — $160^\circ$  C., and showing all the characteristics of ordinary camphene. The hydrochloride has an opposite rotation to the hydrocarbon, and the latter, when treated with formic acid at  $100^\circ$ , yields a borneol; thus proving it to be a camphene. The camphene appeared to be associated with a terebenthene.

In opposition to Barbier, the author maintains that the linalol obtained by him from essence of lavender is identical, and not isomeric, with the licareol found by Morin in essence of *licari kanali*, and that the geraniol which the author obtained from linalol is also identical with the licarhodol obtained by Barbier from licareol.—L. T. T.

Composition of Essences of Fir Needles. J. Bertram and H. Walbaum. Arch. der Pharm. 231, 299.

THE fragrant principles of these essences is the acetic ether of levo-borneol, identical with the synthesized product. Each species contains a certain number of terpenes. When the essence is distilled, the borneol acetate is decomposed, and the acetic acid liberated causes the polymerisation of a part of the terpenes. The following table gives the composition and physical properties of the principal essences:—

Origin.	Density at $15^\circ$ .	Rotation ( $l = 100$ ).	Composition.
Needles of <i>abies pectinata</i> .	0.875	$-20^\circ 40'$	Levo-pinene, levo-limonene, borneol acetate (1.5 per cent.), sesquiterpene.
Cones of <i>abies pectinata</i> .	0.854	$-72^\circ$	Levo-pinene and levo-dimanicene.
<i>Abies canadensis</i> .	0.907	$-29^\circ 54'$	Levo-pinene, borneol acetate (3.6 per cent.), and sesquiterpene.
<i>Picea vulgaris</i> ...	0.888	$-21^\circ 40'$	L. pinene, L. phellandrene, dipentene, borneol acetate (8.3 cent.), and sesquiterpene.
<i>Pinus pumilis</i> Haenke.	0.845	$-9^\circ$	L. pinene, L. phellandrene, silvestrene, borneol acetate, sesquiterpene.
<i>Pinus sylvestris</i> (Swedish).	0.872	$+10^\circ 40'$	Dextro-pinene, d. silvestrene, borneol acetate.
<i>Pinus sylvestris</i> (German).	0.886	$+10^\circ$	D. pinene, d. silvestrene, dipentene, sesquiterpene, borneol acetate.

The authors have also prepared the following ethers of dextro-borneol:—

Ether.	Boiling Point under 10 mm.	Rotary Power ( $l = 100$ mm.)	Density at $15^\circ$ C.	Refractive Index $n_D^{20}$ at $115^\circ$
Formate.....	$90^\circ$ C.	$+31^\circ$	1.013	1.47078
Acetate (levo).....	$98^\circ$	$-35-26'$	0.991	1.46635
Propionate.....	$109^\circ$ — $110^\circ$	$+21^\circ$	0.979	1.46435
Butyrate.....	$120^\circ$ — $121^\circ$	$+22^\circ$	0.966	1.46380
Valerate.....	$128^\circ$ — $130^\circ$	$+26^\circ$	0.956	1.46280

—L. T. T.

A New Derivative from Coto-bark. G. Ciamician and P. Silber. Ber. 27, 841—850.

IN the course of manufacture of cotoine by E. Merck, of Darmstadt, a substance was isolated apparently novel. After crystallisation from petroleum ether it melted at  $68^\circ$ , and gave numbers on analysis leading to the formula  $C_{11}H_{16}O_2$ ; further investigation showed that its relationship to paracotoine is indicated by the occurrence in the latter of the group  $C_6H_5(CH_2O_2)$ , represented in the former by

$C_6H_5$ ; i.e., that the new body contains a phenyl group, which in paracotone is converted into a dioxymethylene-phenyl group.

The new body (phenyleumaline) is readily soluble in ether, alcohol, glacial acetic acid, and chloroform. Methyl alcohol dissolves it freely at the ordinary temperature, but a good deal crystallises out again at 0°. Warm petroleum ether dissolves it to some considerable extent. It melts under boiling water, and crystallises from solutions therein in thin plates. Acetic anhydride forms no acetyl derivative; nor is any methoxyl, &c., indicated by Zeisel's test; so that no hydroxyl, ethoxyl, or methoxyl is present (as with paracotone). Various substitution derivatives are described, e.g., a bromo-derivative,  $C_{11}H_7BrO_2$ , and a nitro-derivative,  $C_{11}H_7(NO_2)O_2$ ; the actions of phenylhydrazine, caustic potash, hydrochloric acid, and methyl iodide (in presence of alkalis) have also been studied.

—C. R. A. W.

**Occurrence of Trigonellin in the Seeds of *Pisum Sativum* and *Cannabis Sativa*.** E. Schulze and S. Frankfurt. *Ber.* 27, 769—770.

TRIGONELLIN,  $C_8H_7NO_2$ , has been found by Jahns to be identical with Hantzsch's synthetic methylbetaine of nicotinic acid, and to occur in the seeds of *Trigonella fauon grecum*. Schulze found it in ordinary peas (*Pisum Sativum*), and the authors have obtained it from hempseeds (*Cannabis Sativa*). It forms two aurochlorides; one melting without decomposition at 198°, and indicated by the normal formula,  $C_8H_7NO_2 \cdot HCl \cdot AuCl_3$ ; the other crystallising in fine needles melting at 186°, and indicated by the formula 4 ( $C_8H_7NO_2$ ), 3 HCl, 3  $AuCl_3$ . The platinochloride can crystallise either anhydrous, or with 4 molecules of water. The quantity present in hempseed is very small.—C. R. A. W.

**The Alkaloids of the *Papaveraceæ*: Codeine.** W. Gochlich. *Arch. der Pharm.* 231, 235.

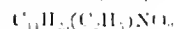
THE author has repeated and amplified some of the experiments of various chemists who have examined codeine salts and derivatives. He describes the hydrobromide, hydriodide, sulphate, chromate, acetate, salicylate, chloraurate, chloroplatinate, and chloromercureate. By the action of phosphorus pentachloride in chloroform solution, the *chlorocodide* of Matthiessen and Alder Wright is obtainable in purer condition than by the action of hydrochloric acid; it crystallises from petroleum spirit in sparingly soluble scales, melting at 118°, and forms a chloroaurate, fusible at 171°—172°. Alcoholic potash in sealed vessels at 120°—130° transforms chlorocodide into *apocodine*; but neither sodium and alcohol, nor tin and hydrochloric acid avail to remove the chlorine. Concentrated sulphuric acid converts codeine into a sulphonated derivative, the *sulphocodide* of Anderson; diluted acid forms the "amorphous isometide of codeine" described by Armstrong; this the author has obtained in crystalline crusts, fusible at 180°; it gives the same colour reactions as codeine, but is precipitated by sodium carbonate. The author regards it as identical with the "pseudocodeine" of Merck, and describes the hydrochloride, sulphate, chloraurate, and chloroplatinate. Pseudocodeine contains a methyl group, but differs from codeine in not containing a hydroxyl group, so that it neither reacts with acetyl chloride nor with acetic anhydride.

—C. R. A. W.

**Carpaïne.** J. J. L. van Ryn. *Arch. Pharm.* 231, 184.

THE best mode of extraction of this alkaloid is to digest the finely powdered dry leaves of *carica papaya* with alcoholic hydrochloric acid, and subsequently with alcohol; after evaporating the extract to a syrup it is treated with watery acid, whereby chlorophyll remains undissolved. The watery solution is treated with ether to remove resin, and then with soda and ether so as to dissolve out the alkaloid in the usual way. The yield is about 0.08 per cent. of the weight of the leaves. Carpaïne is very bitter, perceptible even in a dilution of 1 in 100,000. It is insoluble in water, but readily soluble in various organic solvents, e.g., in 5.5 times its weight of benzene; in 9 times of absolute alcohol, and of

amyl alcohol; in 103 of petroleum ether; and in 574 times its weight of alcohol of density 0.95; in carbon disulphide and chloroform it is soluble in all proportions. It melts at 121° and can be sublimed with partial decomposition. Its rotatory power does not vary with the concentration;  $[\alpha]_D^{20} = +21.9$ . Carpaïne gives no colour reaction with concentrated acids, nor with sulphuric acid and potassium bichromate; its composition is indicated by the formula,  $C_{14}H_{21}NO_2$ . It precipitates with gold chloride, platonic chloride, potassium iodomercureate, phosphomolybdic acid, phosphotungstic acid, picric acid, and iodised potassium iodide solution. Various of its salts are described; the chloroplatinate, chloraurate, hydrochloride, hydrobromide, hydriodide, sulphate, and nitrate. Carpaïne appears to be a secondary base, as its compound with ethyl iodide is decomposed by potash, forming *ethyl carpaïne*—



This base is crystallisable from alcohol; it melts at 91°, and combines with ethyl iodide forming a crystallisable ethyl carpaïne ethioidide, not decomposed by soda, but acted upon by silver chloride, and silver oxide, forming further tertiary ammonia derivatives. Carpaïne is not acted upon by benzoyl chloride, nor by acetyl chloride; with nitrous acid it forms a nitroso derivative,  $C_{14}H_{21}(NO)NO_2$ , crystallisable in colorless prisms, soluble in alcohol.—C. R. A. W.

**Alkaloids from the Seeds of *Lupinus Albus*.** A. Soldani. *Arch. der Pharm.* 231, 321.

THE author has extracted two isomeric alkaloids from these seeds. The first is a solid melting at 99° C., very soluble in water, alcohol, and chloroform, less so in benzene, very sparingly so in light petroleum. Its aqueous solution is strongly alkaline and becomes turbid at the boiling point. The alkaloid has a strong odour of sperm, and a bitter taste. The hydrochloride gives a yellow precipitate (soluble to a blue solution in ammonia) with phosphomolybdic acid, a brown precipitate with a potassium iodide solution of iodine, white precipitates with Meyer's reagent and with phosphotungstic acid, and yellowish-orange precipitates with bromine water and Dragendorff's reagent. It is inactive towards polarised light. The *hydrochloride*,  $C_{15}H_{21}N_2O \cdot HCl + 2H_2O$  forms deliquescent prisms soluble in alcohol and melting at 105°—107° C. The *hydriodide* forms yellow anhydrous crystals melting at 179—180° C., the *platinochloride* orange needles melting with decomposition about 200°; the *aurochloride*, orange needles melting at 182°—183°; the *thiocyanate*, + aq., large crystals melting at 123°—124°; the *methiodide* crystals melting at 237°—238°; the *tribromide*,  $C_{15}H_{21}N_2O \cdot Br_3$ , orange crystals melting at 123—124°; and the *picrate*, *oxalate*, and *tartrate* well formed crystals.

The second alkaloid is a liquid which, when kept over sulphuric acid, deposits large prisms which are, however, so deliquescent that they could not be isolated. Its solutions are dextrorotatory. The *hydrochloride*, + 2 aq. forms colourless prisms soluble in alcohol and in water, insoluble in ether, and melts at 132—133°, or in anhydrous prisms melting above 135°. Alcoholic potash decomposes it, yielding a substance differing from the original alkaloid. The *hydriodide* forms crystals melting at 184—185°; the *platinochloride* ( $C_{15}H_{21}N_2O \cdot HCl$ )<sub>2</sub>PbCl<sub>4</sub> + 4 aq. orange needles or prisms melting with decomposition at 120—125°; the *aurochloride*, yellowish-red crystals melting about 199°—200°; the *thiocyanate* + aq. yellow crystals melting at 183—184°, and the *methiodide* melts at 231—232°. This alkaloid is probably identical with Hagen's lupanine. Both alkaloids are tertiary monatomic bases and both toxic, though in different degrees.—L. T. T.

**Active Principle of *Cientia Virosa* Grains.** J. Frapp. Petersburg. *Arch. der Pharm.* 231, 212.

ON treating the seeds with a weak alkaline solution and distilling with water, cymene and cumenic aldehyde were obtained; but the nature of the toxic principle could not be determined.—C. R. A. W.

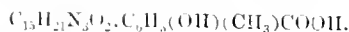
*A Study of Eserine.* A. Petit and M. Polonovsky. Bull. Soc. Chim. 1893, 9, 1008—1015.

Eserine melts at 105—106°, not at 69° as is very generally stated. Its rotatory power is as follows:—In chloroform,  $\alpha_D = -82$ ; in 98 per cent. alcohol,  $\alpha_D = -89$ ; in benzene,  $\alpha_D = -120$ ; in toluene,  $\alpha_D = -120$ .

Since the salicylate is the only salt of those heretofore known that is not very deliquescent, and since eserine has considerable therapeutic value, the authors have prepared the following salts:—

*Eserine benzoate.*—This is prepared by mixing ethereal solutions of eserine and of benzoic acid, and evaporating the ether, when the new salt separates in short white prisms; it is not deliquescent, and it melts at 115—116°. It is very soluble in water (1:4 at 18°) and alcohol yielding neutral solutions.

*Eserine metacresolate.*—

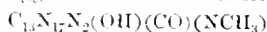


This is made by mixing ethereal solutions of eserine and of metacresotic acid, when the new salt is precipitated in the form of small white prisms. It melts at 156°—157°, and is sparingly soluble in water (1.7 grms. in 100 cc. at 15°); the solution is neutral. This salt does not deliquesce.

*Eserine hydrogen citrate*, prepared as a precipitate by mixing ethereal solutions of eserine and of citric acid, and *normal eserine tartrate*, made by dissolving eserine in tartaric acid and crystallising over sulphuric acid, are both very deliquescent.

An investigation into the chemical nature of eserine has led to the following conclusions:—(1.) Eserine is a mono-acid, tertiary base. (2.) Only one of the three nitrogen atoms imparts a basic character to eserine. (3.) One of the nitrogen atoms is attached to a  $CH_3$  group. (4.) The nitrogen atom of this substituted group does not appear to be the same as that which fixes alkylidide in such compounds as eserine methiodide. (5.) The grouping  $X-CO-C$  is probably present. (6.) The second oxygen atom appears to be of a phenolic character.

The authors suggest the constitutional formula—



as representing the above conclusions.—A. G. B.

#### *The Volatile Hydrocarbons of Essence of Valerian.*

Oliviero. Bull. Soc. Chim. 11, 1894, 150—152.

The author has examined the more volatile portions (boiling below 180° C.) of essence of valerian. In these he found a levo-rotatory camphene boiling at 157°, melting above 0°, giving a rotation  $[\alpha]_D = -21^\circ$  and yielding a hydrochloride having a rotation  $[\alpha]_D = +10^\circ$ ; and also a levo-rotatory terebenthene yielding a levo-rotatory hydrochloride of rotation  $[\alpha]_D = -25^\circ$ . A small quantity of a citrene is also present. As considerable quantities of valeric acid are obtained during the steam distillation of the valerian roots, the author suggests that the camphene may very probably be derived from a borncol valerate present in the root.—L. T. T.

*Essence of Ylang-Ylang.* A. Reyhler. Bull. Soc. Chim. 11, 1894, 407—412.

GAL, (Comptes rend. 76, 1482) states that this substance distils between 160° and 300° C., without leaving any residue, contains no aldehydes and yields on saponification benzoic acid and a mixture of alcohols insoluble in water and boiling between 170° and 200° C., but the nature of which he did not determine. Flückiger and Convent in 1881 (Arch. d. Pharm. [3], 13, 24), state that benzoic acid is only present in this essence in very small quantities, and that it is accompanied by a phenol and an aldehyde or ketone. The author believes these investigators to have been working on different materials, as two preparations, *oleum Annonae odoratissima* made in the Philippine Isles, and *oleum Canangae*, a less pure substance obtained from Java, the Indies, or Réunion, go under the same name. He is therefore examining the former or true Ylang-Ylang again. He obtains two chief fractions boiling under 23—30 mm. pressure at 100—112°

C. and 130°—145° C. respectively. The fraction of lower boiling point yields on saponification large quantities of benzoic acid and an alcohol of the formula  $C_{10}H_{18}O$ , resembling and either identical or isomeric with geraniol, to which the author gives the provisional name of ylangol. It boils at 103—107° under 23—30 mm. pressure, has a density of 0.886 of 15 and has at 15° the refractive coefficient 1.47165. The rotatory power is  $[\alpha]_D = -20.7$ . He has not yet determined what other alcohols are present in this fraction. The fraction of higher boiling point appears to contain a terpene, but is still under investigation. He has not been able to obtain any aldehyde from this essence.—L. T. T.

*Bulgarian Essence of Roses.* W. Markownikoff and A. Reformatsky. Soc. Ph. Ch. de St. Petersburg, 1892, 9.

WITHOUT knowing of Eckart's work on this subject (this Journal 1892, 265) the authors have investigated the composition of Bulgarian rose oil. The substances examined were derived from three different sources, and were viscous solids containing paraffin-like crystals and melting at about 23—24° C. They were all levorotatory. The stearoptene was separated by freezing out. Of the residual eleoptene the greatest part boils about 222°—222.5° C., and is an alcohol of the formula  $C_{10}H_{20}O$ . It takes up two atoms of bromine, indicating the presence of one double linking and appears therefore to belong to the allyl series. The acetate  $C_{10}H_{18}O \cdot COCH_3$  is a very mobile colourless liquid having an odour quite different from that of the alcohol; the alcohol (which the authors term *roseol*) has no action on ammoniacal silver nitrate. It is oxidised by potassium permanganate to a trihydric alcohol  $C_{10}H_{19}(OH)_3$  which is soluble in water, sparingly so in alcohol and in ether. The triacetate is a yellow liquid of aromatic odour which is not viscous at -10°, and is insoluble in water, soluble in ether and in alcohol. With  $H_2$  at 180°—200° a hydrocarbon  $C_{10}H_{20}$  is formed.

The stearoptene  $C_{16}H_{34}$  is a crystalline solid, becomes viscous at 34° and melts at 35.5—36.8°. It is a hydrocarbon of the paraffin series.—L. T. T.

*Extract of the Root of Aspidium Filix.* A. Ehrenberg. Arch. der Pharm. 231, 345.

This root contains an acid (already described under various names); and an oil to which it probably owes its vermifugal properties. This oil only appears in the root in the autumn. It is a yellow liquid of penetrating odour and acid reaction; has a specific gravity of 0.85—0.86, boils with partial decomposition between 140° and 350° C., and distils with steam. 100 kilos. of root gathered in the autumn yielded 40 grms. of the essence. Robert finds that as a medicine a small dose of this essence kills the principal species of worms. Subjected to fractional distillation it yielded propionic, butyric, and caproic acids, leaving a residue consisting of the hexylic and octylic ethers of butyric, caprylic, and pelargonic acids. The essence also contains small quantities of aromatic alcohols and of cineol, to which its properties as a vermifuge are probably due.—L. T. T.

*Pyrazolone Reactions.* R. v. Rothenburg. Ber. 27, 782.

See under XXIII., page 765.

*Conicine and Nicotine.* G. Heut. Arch. der Pharm. 231, 376.

See under XXIII., page 767.

#### PATENTS.

*Manufacture of Geraniol from Citronella-oil.* J. Bertram, Leipzig, Germany. Eng. Pat. 13,401, July 10, 1893.

THE investigations conducted by the patentee have shown that citronella-oil contains pinene, camphene, and dipentene, as well as *citronell-aldehyde* (*citronellone*)  $C_{10}H_{18}O$ . The oil contains also a considerable amount of a substance



identical with geraniol, the aromatic constituent of palmarosa-oil and of geranium-oil, accompanied by small quantities of fatty acid esters of geraniol and of sesquiterpene.

The first claim is embodied in the following paragraph:—

The citronellon above referred to is removed by shaking citronella-oil (100 kilos.) with concentrated sodium bisulphite solution (20—40 kilos.). After cooling, the oil is separated by pressure or suction from the bisulphite compound, and it is boiled with potassium hydrate (6 kilos.) dissolved in alcohol (25 kilos.). After distilling off the alcohol, and driving over the oil with a current of steam, the geraniol is purified by fractional distillation *in vacuo* or with steam. If it be not desired to recover the citronellon, the citronella-oil (100 kilos.) is heated without previous treatment with potassium hydrate (10—15 kilos.) dissolved in alcohol (50—70 kilos.) for about three hours, and the residual oil treated as already described.

Jacobsen has shown (*Annalen*, 157, 232) that geraniol forms crystalline compounds with the chlorides and nitrates of calcium and magnesium. The patentee claims also the use of these for the purification of crude geraniol; *e.g.*, equal parts of powdered calcium chloride and of crude geraniol are mixed; the oil is separated from the resulting solid mass by pressure, suction, or centrifugal action. The mass is then washed with ether, light petroleum spirit, chloroform, &c., and the geraniol, regenerated by treatment with water, is purified by distillation *in vacuo*.—A. R. L.

**Improvements in Bottles for Holding or Containing Poisons or Poisonous Liquids.** R. H. Quine, Pendleton, Lancashire. Eng. Pat. 14,553, July 28, 1893.

See under I., page 713.

**Improvements in the Manufacture of Compounds for Alimentary and Medicinal Purposes.** J. Y. Johnson, London. From C. F. Boehringer und Soehne, Waldhoff, Germany. Eng. Pat. 15,714, August 18, 1893.

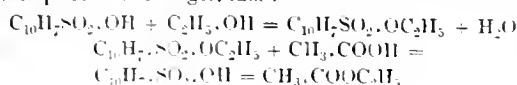
A FURTHER improvement in the process described in Eng. Pat. 818 of 1893 (this Journal, 1893, 370). The improvement is as follows:—After the albumen solution has been heated on a water-bath for about 5 hours with soda-lye, tartrate of iron, and tartrate of sodium (*loc. cit.*), a current of steam is passed into the liquid until it has attained a temperature of 96° C. Subsequently, without stopping the current of steam, small quantities of tartaric acid or of hydrochloric acid are introduced until the solution has a perfectly neutral reaction; additional quantities of acid are added after the lapse of a few minutes until the solution is distinctly acid. When the solution becomes clear it is allowed to cool, filtered, and the filtrate heated at 92° C. with ammonia (1 part of 30 per cent. ammonia for every 10 parts of albumin); the further treatment has already been described. The claim is for the introduction of acid into the hot solution and the separation thereby of unchanged albumin and other extraneous matter.

—A. R. L.

**Manufacture of Esters, Phenol, and other Ethers by Means of Aromatic Sulphonic Acids.** F. Krafft and A. Roos, Heidelberg, Germany. Eng. Pat. 6190, March 27, 1894.

This specification relates to an extension of Eng. Pat. 21,938 of 1892 (this Journal, 1893, 464) to the manufacture of esters of acids and phenols.

By heating sulphonic esters (*loc. cit.*) with carboxylic acids the esters of the latter and free sulphonic acids are obtained. If, for example, a continuous stream of a mixture of acetic acid and ethyl alcohol be added to  $\beta$ -naphthol sulphonic acid heated to 125°—135° C., the distillate will contain, besides acetic acid and alcohol, ethyl acetate and water, the sulphonic acid being regenerated. The reaction takes place in two stages, thus:—



For the manufacture of esters having a high boiling-point the treatment may be effected in a partial vacuum so that the process becomes continuous; liquid acids are added mixed with the alcohols, whilst solid acids are preferably introduced separately.

The sulphonic acids are available for the ethification of those compounds belonging principally to the aromatic series with which sulphuric acid cannot be used because it acts as a sulphonating agent. The numerous carboxylic acids of the aromatic series, as well as phenols, naphthol, aromatic alcohols, hydroxy acids, &c., may be converted into esters by the process.—A. R. L.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**A New Photographic Printing Paper.** Artigues, Bull. Soc. d'Encouragement, 1894, 93—94.

PREPARED by coating paper, first, with a substratum of gelatin, and then with a very thin film of lamp-black (or other suitable pigment.) For use it is sensitised by soaking for a short time in a 5 per cent. solution of potassium bichromate, and subsequently dried. The sensitised paper will remain in good condition for several days, and the whole of the colouring matter may be removed from its surface by simply washing with warm water. But after exposure behind a negative in the usual way, more or less of the pigment adheres to the film, according to the degree of illumination received. After exposure, the print is soaked in cold water, suspended vertically by its upper edge, and developed by gently pouring over it (both back and front) a thin cream of fine white sawdust (previously boiled and washed with water) and water, at a temperature of 27° C. The gentle friction thus applied to the print suffices to remove the non-adherent portion of the colouring matter from its surface. When development is deemed to be complete, the print is washed two to three times in cold water, passed through an alum bath, again washed, and finally dried. The prints thus obtained are permanent, and are stated to possess a depth and delicacy of half-tone unattainable by any other process.—H. T. P.

### PATENT.

**Improvements in or relating to Colours for and Colouring Photographs and other Articles.** F. C. D. Beacham, Bristol. Eng. Pat. 11,924, July 20, 1893.

COLORS, such as will stand wet processes, as "enamelling," "opalines," &c., after use of mordant, or which will serve for ordinary processes without a mordant, are prepared from alizarine colours dissolved in water and ether, with addition of acetic acid, fused potassa, borax, a bichromate, "kreosote," and tartar. The following ingredients and proportions are recommended: 1 dram of colour; 6 oz. water,  $\frac{1}{2}$  oz. ether, 40 drops acetic acid,  $\frac{1}{2}$  scruple fused potassa, "10 drops boric acid," 20 drops kreosote,  $\frac{1}{2}$  scruple borax, and  $1\frac{1}{2}$  grains tartar. When the photograph is to be treated by wet processes, a mordant bath is used, containing solution in 6 oz. water of 1 dram potash and 1 scruple resin, for stock solution; of which for use 1 oz. is taken to 8 oz. water. A second bath is used containing 2½ parts potassium bichromate and 3 parts tartar to 20 to 30 parts water. Details are given of the manipulation required, and in respect to spotting or re-touching, reference is made to Eng. Pat. 2390 of 1891 (this Journal, 1891, 793). Iron salts may be used to darken the colours, either applied by a brush or as a bath. Tin salts may be used in the same manner, or added to the colour employed. In colouring lantern slides and transparencies, solution of gum arabic, sugar, starch, or dextrin may be applied to any part required to be left white. The film is hardened by a bath of chrome alum. The gum varnish may be applied to photographs, back and front, the gum or like substance being mixed to the consistence of cream with equal parts water and alcohol, a few drops of carbolic acid being added.—E. S.



## XXII.—EXPLOSIVES, MATCHES, Etc.

## PATENTS.

*Improvements in or Connected with Blasting and Blasting Cartridges.* J. Macnab, London, and J. Dickson, St. Albans. Eng. Pat. 9688, May 15, 1893.

THE object of this invention is to prevent blown-out-shots and the dangerous tongue of flame which is forced out and often causes an explosion of the fire-damp and coal-dust of the mine.

The improvements consist essentially in the employment of a metallic device composed of two strong discs of mild steel which are the full diameter of the cartridge case and are connected rigidly together by a strong metal rod. A hole in one of the discs serves for the introduction of the powder into the cartridge, between the discs, and the subsequent insertion of the fuse.

Hermetically sealed tubes containing solutions of ammonia or ammonium nitrate, for the purpose of cooling the flame, may be attached to the connecting-rod of the device prior to charging the cartridge with the explosive. The device acts beneficially by offering longitudinal resistance to the force of explosion, thus relieving the strain on the tamping and preventing blown-out shots.—W. M.

*Improvements in Explosive Compounds.* A. V. Newton, London. From A. Nobel, Paris. Eng. Pat., 11,645, June 13, 1893.

THE inventor makes a smokeless explosive by incorporating nitromannite with nitrocellulose, alone or in combination with other substances. When dry nitrocellulose is used, the nitromannite is dissolved in acetone and then the nitrocellulose is added and thorough incorporation effected by malaxation or kneading, and then the plastic mass can be formed into suitable grains or cords by known means.

When wet nitrocellulose is employed, then the nitromannite is mixed with a volatile solvent, not miscible with water, such as methyl nitrate, methyl, ethyl, or amyl acetates in such quantity as to permit of easy incorporation by what is known as the water method of Lundholm and Sayers (this Journal, 764, 1890), or the nitromannite may first be mixed with dinitrobenzene, mono- or di-nitronaphthalene, &c., and when such mixture is heated on a water bath it easily incorporates nitrocellulose and a cellulosidal explosive is obtained.—W. M.

*An Improved Still or Retort, chiefly to be Used for a Distillation Process.* V. Edwards, Richmond Hill, Surrey. Eng. Pat., 13,567, July 12, 1893.

THE inventor claims the use of a flat bottomed copper flask, tapering towards the top in place of the usual glass flask employed in Kjeldahl's process.—W. M.

*Improvements relating to Electric Fuzes.* F. W. T. Brain, Cinderford. Eng. Pat. 14,091, July 21, 1893.

THE chief feature in this invention is the substitution of silver or platinum for copper phosphide in the firing mixture, which usually consisted of potassium chlorate, antimony sulphide, and copper phosphide.—W. M.

*Improvements in Miners' Squibs.* J. R. Powell, Plymouth, Pa., U.S.A. Eng. Pat. 8097, April 24, 1894.

THIS invention relates to mining squibs, and it has for its object to provide an improved squib of this character, which, while very simple in construction, at the same time provides a perfectly safe one, which can be easily inspected so as to avoid the danger of premature explosion. To this end the main and primary object of the present invention is to form a mining squib having a perfect roll-closure at one end to prevent the powder from working into the match portion, and at the same time to provide a squib wherein the match portion thereof can be easily untwisted its entire

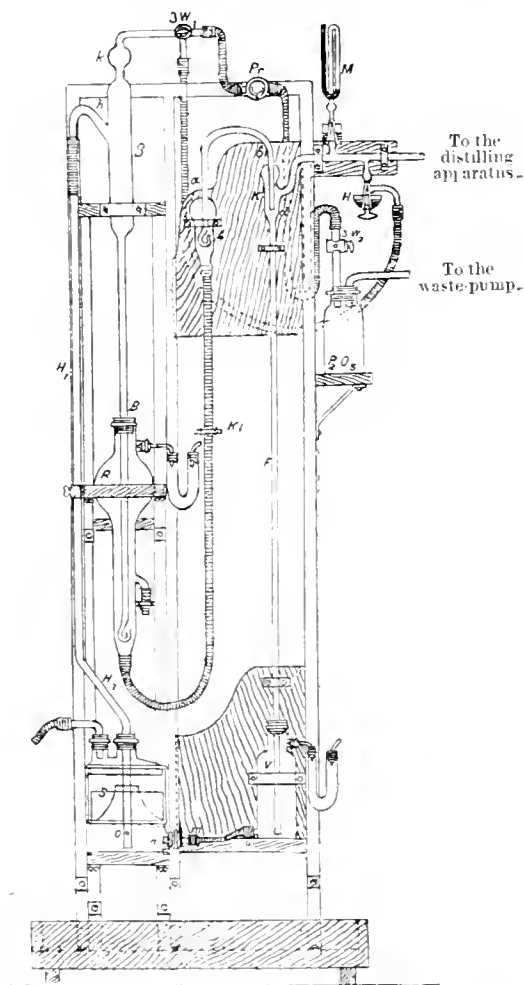
length by the miner before using, in order to ascertain if by any accident any particles of powder had escaped into the same. These objects are attained by the special manner in which the paper wrapper is cut and rolled. The drawings accompanying the specification show the manner of carrying out this invention.—W. M.

## XXIII.—ANALYTICAL CHEMISTRY.

## APPARATUS, ETC.

*New Form of Mercury Air Pump.* G. W. A. Kahlbaum. Ber. 27, 1386—1394.

THIS modification of the mercury air-pump is more especially intended for use in distillation experiments under diminished pressure. The mercury passes down the fall-tube F into a reservoir V, from which it is again elevated



by means of a water pump, which causes it to ascend through the pipe H, H, so that the supply reservoir R is kept refilled as required. To avoid introduction of water vapour, it is desirable to place phosphoric anhydride in a receptacle between the water-pump and the mercury. By means of the pressure-gauge M, the pressure in the distillation apparatus is readily measured.—C. R. A. W.

Condensers for Laboratories. J. Walter. J. Prakt. Chem. 1894, 49, 41-48.

INSTEAD of a Liebig's condenser, an arrangement is in many cases more convenient where the cooling water is contained in a coil, &c. placed in the interior of the tube or chamber into which the vapour is led. For many purposes it is not necessary that the vapour should be condensed in contact with glass, and in such cases a metal condensing arrangement is convenient. Fig. 1 indicates the section of a

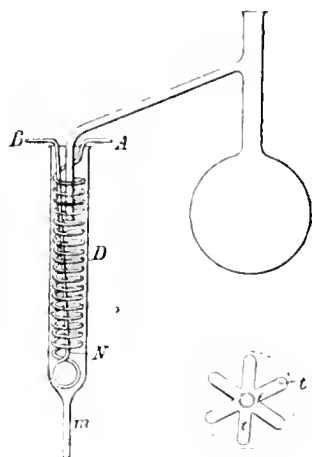


Fig. 2.

Fig. 1.

stellate form, where the cooling water enters and leaves at the top by the connections *t, t*: the cooling surfaces being mostly lateral tubes uniting the ends of the rays, and also the central tube *f*. Fig. 2 represents a worm form, which for a small condensing arrangement may be constructed by coiling up about 120 cm. of metal tubing; copper answers well, especially if tinned or coated with nickel, the outer diameter of the pipe being about 3, and the inner diameter about 2 mm. The cooling water enters at A and leaves at B, or *vice versa*, the condensed liquid dropping out through *m*. Arrangements of this description may be conveniently adapted to extraction apparatus, such as Soxhlet's, and the various modifications thereof subsequently introduced by others, or they may be used as dephlegmators, or as condensers for fractional distillation by passing water, &c. at a given temperature through the coils. For special purposes, where the cost is not prohibitive, silver or platinum tubing may be employed, or copper plated with gold, &c.

—C. R. A. W.

New Apparatus for Fractional Distillation. E. Varenne. Bull. Soc. Chim. 11, 1894, 289-292.

THE condensing column is formed of elements entirely separated from one another, but communicating by means of two tubes connecting each consecutive pair; one traversed by the vapour, the other bringing back the liquid condensed (Fig. 1). A number of comparative trials of the efficiency of this arrangement (six bulbs) as compared with that of the apparatus of Savalle, Le Bel-Henninger (15 plates), and of Claudon-Morin respectively, gave the values,

Apparatus of Savalle .....	1.58
„ Le Bel-Henninger.....	1.56
„ Claudon-Morin.....	1.53
„ E. Varenne.....	1.683
Theoretical coefficient.....	1.025

thus indicating a high degree of superiority of Varenne's arrangement.

Similarly the following figures were obtained as regards the quality of the rectified spirits distilled from the same "low wines" in each case.

	Le Bel-Henninger.	Varenne Small sized Laboratory Model.	Varenne Large Working Factory Model.
Requiring more than 10 minutes to decolorise potassium permanganate.			
More than 15 minutes.	0	30	50
More than 20 „	0	25	35

Fig. 1.

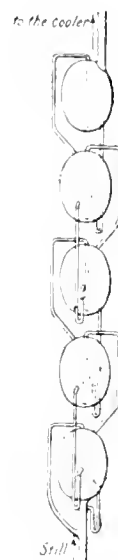
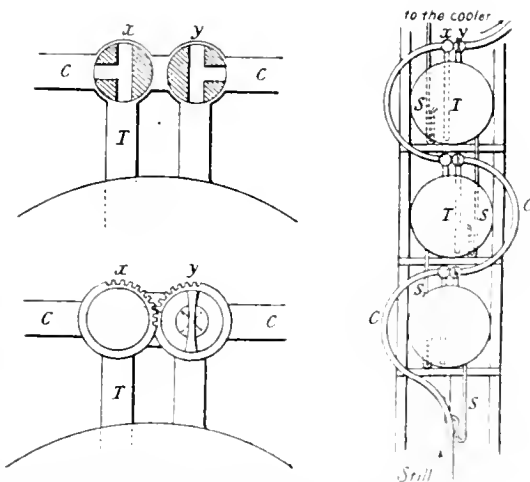


Fig. 2.



The larger size arrangement is thus more effective than the smaller one: Fig. 2 represents this industrial form of the apparatus.—C. R. A. W.

*The Ebullioscope and its Use for estimating Alcohol in Beers.* H. Tornøe. *Bull. Soc. Chim.* **11**, 1894, 115—120.

THE author employs a slight modification of the ebullioscope of Vidal and Mahigand, in which the movable scale is replaced by graduations on the thermometer stem itself. The author has proved that the amount by which the boiling point of water is lowered by the presence of a given quantity of alcohol is independent of atmospheric pressure, but varies sensibly according to the form and size of the apparatus. Employing ebullioscopes of like shape and size these variations may be reduced to negligible quantities. The tables prepared for use with the ebullioscope being based upon the results of experiments with mixtures of alcohol and pure water, it was found that when this apparatus was used with beers, the percentage of alcohol found was always slightly higher than that found by the distillation method. This difference was clearly due to the extract present in beer, but Raoult's work, showing that the lowering influence on the boiling-point of non-volatile substances in solution is inversely as the molecular weights of such substances, seemed to point to any such direct action of the extract (with its high average molecular weight) being far too slight to account for the differences observed. The difference was always found to be least when the proportion of alcohol was largest, and greatest when the extract matter was large. The author points out that in the ordinary distillation method the distillate is made up to the original weight of the beer, and the gravity then taken. Thus, the percentage of alcohol found is not the percentage on the water in the beer, but on the water plus the solid extract, whilst the ebullioscope result is the percentage on the water in the beer, and should be corrected for the solid extract. Based on these considerations, and on the fact that the solution of a given quantity of solid extract in water or in a mixture of water and alcohol, causes the same increase in specific gravity in each case, the author has devised a means by which both a correct estimation of alcohol and also an estimation of the solid extract, may be easily and rapidly made by help of the ebullioscope. If  $S_b$  represents the specific gravity of the beer,  $S_p$  that of the mixture of water and spirit in the beer (which serves as a solvent for the extract) and  $S$  the specific gravity of a hypothetical solution of the solid extract in a weight of water equal to the combined weight of the water and alcohol in the beer, we get the formula  $S_b - S_p = S - 1$  or  $S = 1 + S_b \cdot S_p$ .

The ebullioscope reading gives the percentage of alcohol to water, and from this number, by reference to a specific gravity table of aqueous alcohol,  $S_p$  can readily be obtained.  $S_b$  is readily determined and  $S$  is therefore obtainable. An approximate gravity table of aqueous solutions of solid extract may be easily constructed, and by its help  $S$  will give the solid extract. This number may then be used to correct the alcohol estimation. By this means both the alcohol and the solid extract in a beer may be estimated in about 20 minutes.—L. T. T.

*The Determination of High Temperature by Fusion Tests.* C. Schneider. *Uhländ's tech. Rundsch.* 1894, 54; *Proc. Inst. Civil Eng.* 1894, **116** [ii], 103—104.

THE use of vitrifiable test pieces for the determination of high temperatures, introduced by the late Prof. H. Seger, has been found to be of great value in ceramic manufactures, where the heat to be measured is higher than the melting-points of the metals in ordinary use. The substances used for this purpose are analogous in composition to porcelain glazes containing silica, alumina, ferric oxide, lime, and potash, in varying proportions, the mixtures becoming more refractory as the silica is increased. The different ingredients being thoroughly incorporated into a paste, which is moulded into taper-pointed pieces, either cones or three-faced pyramid, having a number corresponding to the composition impressed upon one side. When required for use, a series of the air-dried cones (ten or a dozen of different numbers, in the case of a preliminary experiment) are placed upon a plate of burnt fireclay, being made to adhere by slightly moistening their bases and rubbing upon the plate, which is then exposed in the

furnace to the heat to be measured. The test-piece is considered to be melted when it is so much softened that the point bends over and touches the surface of the plate, which, with a little practice, can be readily determined.

The standard mixture, or No. 1 at the lower end of the scale, has the following composition.—Silica, 65; alumina, 8.3; ferric oxide, 8.7; lime, 10.6; potash, 7.6 per cent.

Its melting-point is 1,150° C., while that of No. 20, the least fusible number of the series, is 1,700° (near that of platinum), giving a range of 550°, which, divided over the intermediate ones, gives a difference of 28.9 between consecutive numbers, so that collectively they represent the higher portion or gold-platinum series of the Prinscp pyrometer. Until lately, the extension of the scale downwards had not been satisfactorily effected; the most ready method of lowering the fusibility of the standard mixture, namely, the partial replacement of lime by oxide of lead being objectionable, on account of its volatility as well as its liability to reduction when exposed to furnace gases. E. Cramer has, however, found that when half an equivalent of silica, corresponding to 8 per cent. in the above mixture, is replaced by the boric acid, the melting-point is reduced by 190° to 960° C., or that of silver, so that the silver-gold series of alloys can be equally replaced.

The new material is a glass made of the following mixture:—Crystallised borax 193 parts, marble 50 parts, china clay 52 parts, sand 96 parts, melting at 960°. When mixed with No. 1 in such proportions as to vary the contents of boric acid by one-twentieth of an equivalent, a scale of numbers differing in melting-points from each other by 19° is formed, which, to maintain the uniformity with the original series, are numbered from 01 to 010, the latter being the most fusible. The author of the note points out that these tests are likely to be of value in studying the distribution and utilisation of heat in boiler flues, as they can be more easily used than calorimeters or pyrometers. The test-pieces are made by the Royal Porcelain Works, Berlin, in two sizes, about 2½ and 4 inches high.

#### INORGANIC CHEMISTRY.—QUALITATIVE.

*Hydrogen Dioxide in Atmospheric Air and in Aqueous Deposits therefrom.* J. v. N. Hosva. *Ber.* 1894, **27**, 920.

See under XVIII. B., page 753.

#### INORGANIC CHEMISTRY.—QUANTITATIVE.

*Corrections to be applied in Titrating Liquids containing Suspended Matter.* E. Lenoble. *Bull. Soc. Chem.*, **11**—**12**, 1894, 336—345.

WHEN a liquid containing matter in suspension is filtered and titrated, the result is too high in reference to the actual fluid present, unless the bulk of the suspended matter is taken into account. If  $V$  be the volume of liquid + solid matter, and  $P$  that of the suspended solids, the result of the analysis is calculated on  $V - P$  instead of on  $V$ . By filtering off a known quantity and titrating, and adding this volume of water to the residue and repeating the operation two figures are obtained, respectively  $A$  and  $B$ , from which the true value may be deduced. Thus the percentage richness of a sugar-beet juice, reckoned on clear liquid + pulp, is—

$$\frac{A^2L}{V(A-B)}$$

when  $V$  is the volume of liquid + pulp operated on, and  $L$  that of the liquid filtered off for the titration. When a vacuum pump is not at hand, and a filter has consequently to be used of considerable volume, the method may be modified by making three determinations: first, a given quantity of fluid is filtered off and titrated, giving the value  $A$  as before; the filter paper and adherent pulp are then put back, and as much water added as will bring the whole to the original volume, so that the solid matter present is now greater than

# COURSE OF AVERAGE PRICES OF

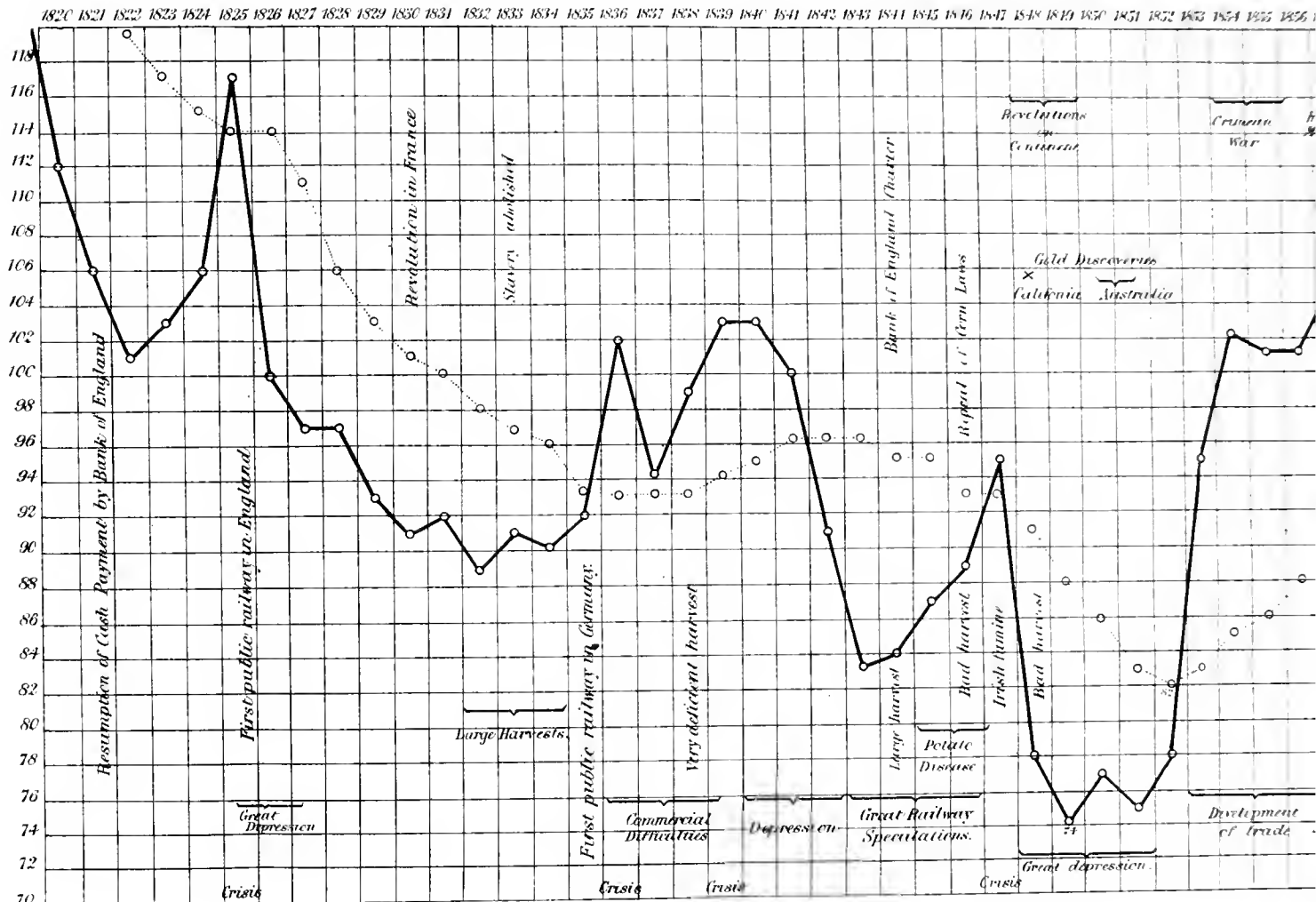
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## PRICES OF COMMODITIES IN ENGLAND.

Number of Article		AVERAGE PRICES				Index Numbers 1893 (1867-77=100.)
		1867-77	1878-87	1884-93	1893	
0	Silver .....	58½	50	44½	35½	58.6*
1	Wheat, English gazette .....	54½	40	32	26¼	48
2	" American .....	56	43½	35	27/6	50
3	Flour, town made white .....	46	34½	29	26	54
4	Barley, English gazette .....	39	31½	27½	25/7	66
5	Oats, " .....	26	21	19	18/9	72
6	Maize, American mixed .....	32½	25	22½	20	61
7	Potatoes, good English .....	117	102	77	65	56
8	Rice, Rangoon cargoes .....	10	8	7	6/2	62
9	Beef, prime .....	59	55½	49	48	81
10	" middling .....	50	46	40	39	78
11	Mutton, prime .....	63	64½	57	53	84
12	" middling .....	55	53	46	42	76
13	Pork, avg. (large & small) .....	52	49	44	50	96
14	Bacon, Waterford .....	74	71	65½	68	92
15	Butter, Friesland, finest .....	125	116	106	106	85
16A	Sugar, West Indian refining .....	23	17	13½	14½	62
16B	" beet, German, 86 p.c. f.o.b. .....	24	18	13½	15	61
17	" Java, floating cargoes .....	23½	21½	16½	17½	61
18A	Coffee, Ceylon plantation, low middl. ..	87	78	86	103	123
18B	" Rio, good channel .....	64	52	66	81	53
19A	Tea, Congou common .....	11½	6½	5½	5½	53
19B	" average import price .....	17½	12½	11	9.81	61
20	Iron, Scotch pig .....	69	46	43½	42¼	51
21	" bars common .....	8½	5½	5½	5	51
22	Copper, Chili bars .....	75	55	51	44	59
23	Tin, Straits .....	105	89	95	85	81
24	Lead, English pig .....	20½	14	12½	9½	48
25	Coals, Wallsend Hetton in London sh. p. ton	22	16½	17½	19½	89
26	" average export price .....	12½	9	9½	9.98	80
27	Cotton, middling Uplands .....	9	6	5½	4½	51
28	" fair Dhollerah .....	6½	4½	3½	3.½	53
29A	Flax, St. Petersburg 12 head best £ p. ton	46	33	30½	34	67
29B	" Russian, average import ..	48	34	30	31½	64
30A	Hemp, Manila fair roping .....	43	35½	35	26	68
30B	" St. Petersburg clean .....	35	26½	27	24	60
31	Jute, good medium .....	19	15	13½	13	60
32A	Wool, merino P. Phillip, avg. fleece d. p. lb.	21½	18½	15½	12½	52
32B	" Adelaide, avg. grease ..	9½	8½	7	6	54
33	" English, Lincoln half hogs ..	19½	11½	10½	10½	65
34	Silk, Tsatlee .....	23	15	13½	12½	81
35A	Hides, River Plate, dry .....	9	8½	6½	5½	87
35B	" salted .....	7	6½	5½	4½	72
36	Leather, crop hides, 30-45 lbs. ..	16	15	14	13	69
37A	Tallow, St. Petersburg, Y.C. .....	45	41	39	48	32
37B	" town .....	45	35½	28½	30½	62
38	Oil, palm .....	39	32½	26½	28½	66
39	" olive .....	50	40	38	36	76
40A	" linseed .....	30	28	20½	20½	71
40B	Seeds, linseed .....	60	46	41½	42	32
41	Petroleum .....	124½	6½	5½	4	62
42	Soda, crystals .....	92	62	57	58	66
43	Nitrate of soda .....	14	12½	9½	9½	76
44	Indigo, Bengal, good consuming ..	7½	6	4½	5½	71
45A	Timber, hewn, average import ..	60	47	42½	38	68
45B	" sawn or split, avg. import ..	54	47	44½	43	68
45 Commodities ... .. Total		3061				68
Average Index No. for 1893 ... ..		68				

\* Silver compared with 60/84d. per ounce, being the parity between gold and silver at 1:15½, and not included in the general average. † Petroleum average from 1873-77 only.  
Sh. = Shillings. d. = Pence. qtr. = Quarter.



### EXPLANATORY NOTES.

The Diagram gives the course of prices in England according to my Index Numbers of 45 commodities (before 1846 only 31 principal commodities). It formed part of my paper on "Prices of Commodities and the Precious Metals," and was first published—with movements up to 1886—in the Journal of the Royal Statistical Society. The commodities comprise the following, some of the more important articles being represented by two descriptions: Wheat, flour, barley, oats, maize, potatoes and rice, beef, mutton, pork, bacon and butter, sugar, coffee and tea, iron, copper, tin, lead and coals, cotton, flax, hemp, jute, wool and silk, hides, leather, tallow, palm-oil, olive-oil, linseed-oil, petroleum, soda, nitrate, indigo and timber.

The Index Numbers have been constructed in the following manner: The average prices of the 11 years 1867-77—which in the aggregate are equivalent to the average of the 25 years 1853-77—were called 100 in the case of each com-

modity and the percentage variation was calculated for each year; for instance English wheat, average 1867-77=54/6d. per qtr. equal to 100, average price in 1855=74/8d. equal to 137 (or 37 per cent. above the average point) and average price 1893=26/4d. equal to 48 (or 52 per cent. below the average point). The percentage figures of all articles in each year were added together and divided by the total number of commodities and the figure thus obtained formed the Index Number for the respective year, for instance 1893, total numbers of 4 commodities=3061, Index Number 66 or 32 per cent. below the average point. The Index Number is therefore the arithmetical mean, all articles being considered as of equal value. By the test of quantities—according to the importance of each article—the result was found to be perfectly reliable.

AUGUSTUS SAUERBECK, F.S.S.

3, MOORGATE STREET BUILDINGS, LONDON.

(AVERAGE OF 1867-1877 BEING 100.)

COMPILED BY AUGUSTUS SAUERBECK, F.S.S.

[illegible]

culated for each year; for instance (1931) equal to 100, average price in 1932 (above the average point) and average price in 1933 (below the average point). The two figures were added together and divided by two. The figure thus obtained formed the base figure for 1932. For instance in 1933, total numbers of 45 (above the average point) and 45 (below the average point) were added together and divided by two. In this way, the arithmetic mean, all articles being considered as of equal importance—according to the importance of the article—was perfectly reliable.

SAUERBECK, F.S.S.,  
DEGATE STREET BUILDINGS, LONDON.

This dotted line gives the best picture of the gradual movement of the average prices of whole periods as the fluctuations from year to year are obliterated.

ENTERED AT STATIONERS' HALL.

at first by the bulk of the filter. After admixture, a portion of liquid is filtered off again and titrated, giving the value B, the filter now used being the duplicate of the former one (cut simultaneously from the same sheet). This second filter and pulp, &c., is put back as before, water added to the original volume, and, after mixing, a third portion filtered off (through any convenient filter paper) and tested, giving the result C'. If L be the volume taken for titration in each case, and V the total volume of fluid and pulp, &c., operated on, the required percentage,  $x$ , is given by the equation—

$$x = \frac{AL \{ B(A + B) - 2AC \}}{V \{ B(A + C) - 2AC \}}$$

Another variation is obtainable by directly measuring the bulk of the filter paper by noting the water its duplicate displaces on introduction into a burette; preferably with a suction pump arrangement to eliminate air bubbles.

—C. R. A. W.

#### Volumetric Method of Estimating Phosphoric Acid.

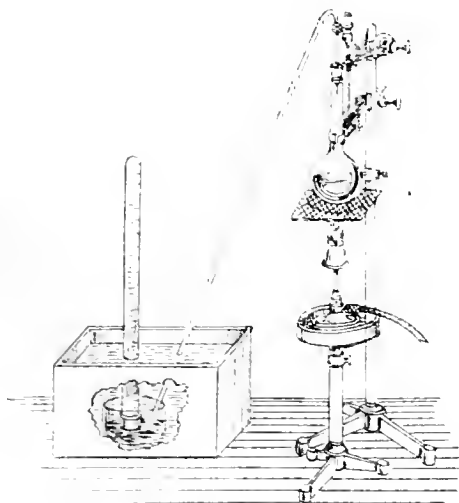
A. F. Holleman. *Zeits. anal. Chem.* **33**, 185—186.

In a paper published by the author in the "*Recueil des travaux chimiques des Pays-Bas*," vol. 12, 1, he describes a volumetric method for the determination of phosphoric acid depending upon the precipitation of the phosphoric acid from a solution of a phosphate,  $M_3HPO_4$ , as silver phosphate,  $Ag_3PO_4$ , by a standard solution of silver nitrate, when sodium acetate is present to fix the nitric acid liberated by the reaction. The excess of silver nitrate is then titrated by Volhard's method with a solution of potassium thiocyanate. The method has been recently tested by van Ryn in the author's laboratory with solutions containing known quantities of calcium phosphate,  $Ca_3(PO_4)_2$ , dissolved in nitric acid, equivalent to from 94.9 to 190.0 mgrm. of  $P_2O_5$  in 50 cc. of the solution. A table is given showing that the results obtained are good, except when a considerable excess of the silver solution has been added (equal to 17.3 and 14.6 cc. respectively of one-tenth normal thiocyanate solution), the results being then too high.—H. S. P.

#### A New Method of Performing Schlösing's Process for Estimating Nitric Acid. L. L. de Koninck. *Zeits. anal. Chem.* **33**, 200—205.

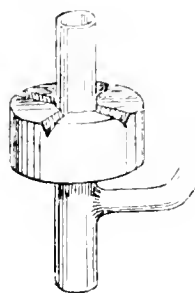
FIGURE 1 shows the apparatus which is designed to simplify the process, and especially to prevent a back

Fig. 1.



rush up the delivery tube into the flask. The decomposition flask is a tractional distillation flask of from 200–300 cc. capacity. The side tube of this flask is bent upwards and attached to a small funnel by a piece of India rubber tubing which can be opened and closed by a clip. The delivery tube is of small bore and passes through a perforated enouchoupe stopper fitting into the neck of the flask. A ring of cork is slipped over this delivery tube, round which a wire is fastened to hold the small funnel firm. The delivery tube is so long that the difference of level between its upper and lower bends is from 75 to 80 cm. The lower end is constructed as shown in Fig. 2; a piece of tubing

Fig. 2.



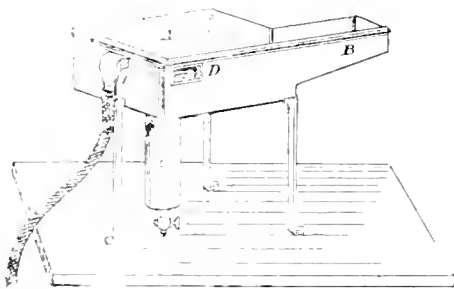
open at both ends, 6–7 cm. in length and 7–9 mm. internal diameter is fused upon the end of the delivery tube. A cork of about 10 mm. thickness, and with four radiating grooves cut in it, is slipped over the upper part of the vertical tube. The delivery tube passes into and rests upon the bottom of a small crystallising dish, which contains enough mercury to cover the opening of the delivery tube where it is fused into the vertical tube, but not enough to reach to the cork. The dish is stood in a large vessel filled with water. The tube in which the nitric oxide is collected and measured is placed over the vertical tube, and rests upon the cork, and so does not dip into the mercury. A free communication between the water inside and outside of the tube is allowed along the grooves cut in the cork. When the gas is generated in the flask it easily breaks through the thin layer of mercury which covers the mouth of the delivery tube and rises up the vertical tube into the collecting tube. If, on the contrary, a back pressure is formed into the flask, only the mercury rises up the tube, but owing to its length it never enters the flask. This can also be provided for by a long delivery tube dipping into mercury, but in that case when the gas is evolved rapidly there is a risk of bubbles escaping at the side of the collecting tube.

Fig. 3 shows a convenient form of trough for cooling the gases to the temperature of the room, and reading off their volumes in the collecting tubes. A is a right-angled square trough made of zinc, lacquered on the inside to prevent the action of any mercury accidentally introduced. B is a lengthening of the trough at one corner to allow of the tubes being easily laid down and filled. C is a cylindrical deepening in the bottom of the trough, into which the tubes are placed to cool and to permit of the gas contained in them being read off at the atmospheric pressure. D is a small glass window for reading the volume of gas through. E is an overflow pipe to allow of a constant stream of water being sent through the trough.

In carrying out an analysis the author recommends Grandean's modification (Grandeau, *Analyse chimique appliquée à l'agriculture*) of comparing the volumes of nitric oxide yielded under the same conditions by the sample under examination and by a known quantity of a pure

nitrate. He conducts the operation in the above described apparatus as follows:—40 cc. of concentrated solution of ferrous chloride (containing 200 grms. of iron per litre) are poured into the flask, followed by 40 cc. of hydrochloric acid (sp. gr. 1.1), and the side tube is also filled with hydrochloric acid up to the funnel. The delivery tube is

Fig. 3.



then attached and the contents of the flask boiled until all the air is expelled from it. A collecting tube of 100 cc. capacity is then placed over the delivery tube, and 10 cc. of a solution of pure potassium nitrate (containing exactly 40 grms. of this salt per litre; 10 cc. of this yields from 95–100 cc. of nitric oxide) are poured into the funnel. The flame is then withdrawn to establish a slight vacuum in the flask, and the solution is then allowed to flow into the flask until the funnel is nearly empty. The funnel is then rinsed twice with 10 cc. of hydrochloric acid (sp. gr. 1.1) which is each time allowed carefully to flow into the flask. The flask is then again boiled and the nitric oxide evolved collected. The boiling is continued until bubbles of gas cease to enter into the collecting tube. The collecting tube is then put on one side and another put in its place. The sample to be analysed is then treated in the same way. As much of it is taken as will yield about the same volume of gas as the pure nitrate; and after this a second control analysis with the solution of pure nitrate is made. All the tubes are then placed in the cooling trough and the volumes of gas they contain read.

As many as eight or nine experiments may be made with the quantity of ferrous chloride here given, and still more if it be regenerated in the flask by adding some concentrated stannous chloride through the funnel. Care must in this case be taken, (1) that an excess of stannous chloride is not added, as this may lead to the formation of ammonia; and (2) that the funnel and tube are afterwards well rinsed with hydrochloric acid so as to remove every trace of stannous chloride from them. The number of tests that can thus be made is only limited by the tendency of the salts to crystallise out.—H. S. P.

*The Estimation of Nitrates by Means of the Nitrometer.*  
E. Henry. Bull. Soc. Chim. 1894, 11—12, 24—27.

THE method of estimating nitrates by the action of sulphuric acid and mercury, making use of the nitrometer, of the form Fig. 1 for liquids or Fig. 2 for solids, for the purpose of measuring the nitric oxide evolved, may give inaccurate results unless certain precautions are observed. Nitric oxide being slightly soluble in sulphuric acid, the least possible quantity of acid must be used for washing out the funnel. When restoring equilibrium at the conclusion of the reaction the level of the mercury in the tube *b* should be arranged at a little less than one-seventh below the level of the sulphuric acid in tube *a* so that there is a slight depression in the latter branch. Equilibrium is then restored by pouring small quantities of acid into the funnel and admitting them into the tube. This operation is also continued until the nitrometer has cooled down to the surrounding temperature.

A comparative experiment with a pure nitrate is recommended in order to estimate the effect of the solubility of nitric oxide in sulphuric acid of the formation of small

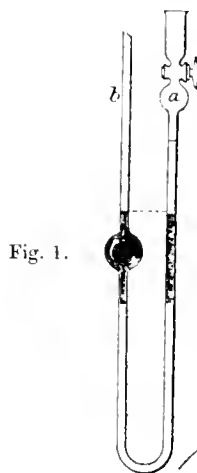


Fig. 1.

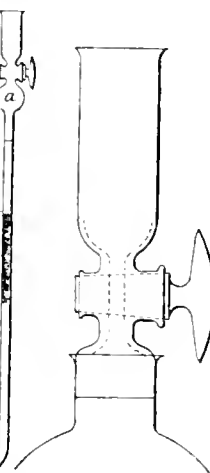
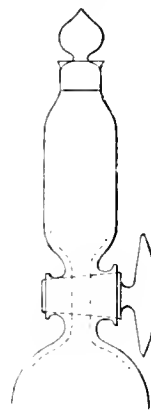


Fig. 2.

quantities of nitrous oxide, and of the sulphuric acid being more viscous than the water with which the tube was graduated.

For solids the form Fig. 3 is recommended as more suitable than form Fig. 2, the body to be analysed being intro-

Fig. 3.



duced into the stoppered funnel, dissolved in sulphuric acid or in a little water, and then sulphuric acid added, and the solution admitted into the tube.

The author considers it preferable to keep the pressure in the tube below that of the atmospheric pressure in order to diminish the error due to the solubility of the gas.

—J. G. W.

*Estimation of Ammonia in Ammoniacal Liquor, and in other Liquids containing Sulphur or Cyanogen, with Litmus as an Indicator.* E. Henry. Bull. Soc. Chim. 1893, 9, 1118.

TO avoid the inconvenience caused by the decolorisation of the litmus by hydrosulphuric and hydrocyanic acids—which, notwithstanding the presence of excess of alkali in the retort, come over with the ammonia and dissolve in the standard acid during the process of estimating ammonia—the author adds basic lead acetate to the ammoniacal liquor to be analysed. Fixed and volatile acids are thus precipitated.—A. G. B.



*Valuation of Zinc Dust.* O. Bach. *Zeits. angew. Chem.* 1894 [10] 291-292.

F. MEYER (*Zeits. angew. Chem.* [8], 1894); this *Journal*, August 1894), has devised a method for the valuation of zinc dust. The method now described by the author is the same in principle as that of Meyer, but does not necessitate the use of special apparatus. (See also this *Journal*, 1893, 65).

A weighed quantity of zinc dust is introduced into a glass jar of 150 cc. capacity and mixed with water; the jar is closed by means of a doubly-bored caoutchouc stopper through which two tubes bent at right angles pass. One tube dips below the surface of the liquid contained in the jar, whilst the other tube terminates just below the stopper. Within the jar there is also a small cylinder containing an excess of acid. The gas-delivery tube is connected with a flask containing 10 per cent. soda, and the evolved hydrogen is collected and measured in a burette of 250 cc. capacity divided into 0.5 cc. The particular form of burette employed is that described by Pinette (*Chem. Zeit.* 1890, 1442). The longer tube is connected with a carbonic anhydride apparatus, and the last named gas passed through the jar until the whole of the air is expelled. A burette filled with soda-lye is now connected to the gas-delivery tube, and the jar tipped so that a portion of the sulphuric acid contained in the small cylinder comes into contact with the zinc dust, and the evolution of hydrogen commences. As one burette is not of sufficient capacity to contain the whole of the evolved hydrogen from 1 gram of zinc dust, a second one must be attached, which can be accomplished without any loss if it be done at a moment when there is a pause in the gas evolution. When all the acid has been mixed with the zinc dust and the evolution of gas has ceased, carbonic anhydride is passed through the apparatus until the volume of the gas contained in the burette no longer increases, or the gas contained in the jar is driven out by filling it with water. The burettes are finally placed in a large cylinder of water, the temperature and pressure noted, and the volume of hydrogen, duly corrected, calculated as metallic zinc.—A. R. L.

*Notes on the Cupellation of Bismuth-Silver Alloys.* E. A. Smith. *Proc. Chem. Soc.* 1894 [139], 117.

THE loss of silver in the cupellation of lead-silver alloys has been the subject of a large number of experiments at various times, but comparatively few results have been published on the loss of silver in the cupellation of bismuth-silver alloys.

The present experiments were made to ascertain whether the loss of silver by cupellation was the same when varying quantities were employed with a constant ratio of bismuth in the place of lead, and also to find the loss of silver when cupelled with a gradually increasing ratio of bismuth.

The bismuth and the silver employed were specially purified.

The temperature was slightly lower than that usually adopted in the cupellation of lead-silver alloys.

Mr. Hambly concluded that, provided a constant ratio of lead and silver be employed, the loss of silver very slightly increases according to the decrease in the weight of the silver cupelled.

The author finds that a very much greater loss takes place under the same conditions when bismuth is present instead of lead, although the ratio of the loss appears to be about the same.

The results confirm the fact, already known, that an increasing ratio of lead or bismuth produces an increasing loss of silver, the loss, however, being very much greater in the case of bismuth.

As the loss of silver by volatilisation during cupellation is altogether inconsiderable, unless the temperature at which the operation has been conducted has been much too high, the loss is evidently due to the fact that silver is absorbed by the cupel.

*Quantitative Analysis of Vanadiferous Ligulite.* Prof. Kyle. U. S. Consular Rep. June 1894, 176-180.

See under N., page 738.

## ORGANIC CHEMISTRY.—QUALITATIVE.

*Pyrazolone Reactions.* R. v. Rothenburg. *Ber.* 27, 782.

BODIES of the pyrazolone class (most substance of antipyrine) containing non-substituted NH groups, give yellowish-brown to chestnut-brown colorations with ferric chloride. Silver nitrate gives precipitates of varying compositions; ammoniacal silver solutions are not reduced. Both alkalis and acids dissolve pyrazolones, but do not form hydrazines. Nitrous acid forms nitroso derivatives of yellow or orange colour, and benzaldehyde furnishes strongly coloured benzal derivatives, when the hydrogen in the CH<sub>2</sub> group is not wholly replaced. Bromine replaces hydrogen in this group. Diazo salts in alkaline solution give more or less unstable compounds. Oxidising agents exert energetic actions on pyrazolones, the complex HN.CO being usually destroyed.—C. R. A. W.

*Compounds of the Sugars with Polyhydric Phenols.* E. Fischer and W. L. Jennings. *Ber.* 27, 1355-1362.

ALTHOUGH the alcohols of the fatty series react with certain sugars, forming glucosides (this *Journal*, 1894, 167), it is found that the monohydric phenols do not. When, however, the polyhydric phenols are submitted to a treatment analogous to that already described in the case of the fatty alcohols (*loc. cit.*), compounds are obtained which differ according to the nature of the phenolic compound employed. The authors describe compounds of arabinose, glucose, and other carbohydrates with resorcinol, which are amorphous colourless compounds. The particular technical interest attaching to these compounds is, however, that they are found to give beautiful colour reactions when treated in alkaline solutions with certain oxidising agents, such as lead peroxide, mercuric oxide, and silver oxide. The most beautiful of these reactions is that with Fehling's solution, which, although not quite so delicate and somewhat less convenient than Molisch's reaction (*Monatsh. chem.* 7, 198), is recommended as a confirmatory method supplementary to the latter. The method of carrying out the test is as follows:—To about 2 cc. of the dilute aqueous solution of the substance to be tested, 0.2 gram of resorcinol is added and the mixture saturated with gaseous hydrochloric acid at 0°. If the quantity of carbohydrate present is small, the test is now allowed to remain at the ordinary temperature for 12 hours, but if, on the other hand, the amount of carbohydrate is considerable, one hour suffices. The liquid is diluted with water, an excess of caustic soda added, and it is warmed with Fehling's solution, of which latter only a few drops are taken when the amount of carbohydrate is small. A reddish-violet very characteristic coloration ensues; it is fugitive after a time when the liquid is highly diluted. Insoluble carbohydrates such as starch are triturated with water, and after the addition of resorcinol the cooled mixture is saturated with gaseous hydrochloric acid. The reaction has been found to answer for the detection of small quantities of saccharose, lactose, maltose, dextrin, gum, glycogen, starch, and cotton wool, besides being available for the simpler sugars (pentoses and hexoses); normal urine also exhibits the reaction.

Compounds with other phenols are described in the paper, which have only a purely scientific interest.—A. R. L.



## ORGANIC CHEMISTRY.—QUANTITATIVE.

*The Employment of "Micro-Chemical" Reagents in Analytical Chemistry. First Communication.* W. Lenz. *Zeits. Anal. Chem.* **33**, 193—204.

*The Estimation of the Ethereal Oils, particularly in Cloves and Mace.*—The author having known for some time that a solution of equal parts of water and crystallised sodium salicylate was an excellent clarifying and preservative reagent for microscopic preparations, and having also noticed the peculiar solvent properties of this solution upon various ethereal oils, especially of oil of cloves, was led thereby to use it in the analytical determination of oil of cloves. He first of all describes his method for extracting ethereal oils by water alone, which is as follows:—From 10—20 grms. of the material to be examined are placed in a tubulated retort of about 250 cc. capacity, the long neck of which is bent to a right angle at about the middle. Enough water is added to the material to make a thin "slurry," the neck of the retort, which ascends to the bend and then dips downwards, is attached to a condenser, and steam is then passed through the mixture in the retort by means of a tube passing through the tubulus. The contents of the retort are at the same time heated by a gas flame so regulated that the volume of liquid is kept constant. The particles of material may be prevented from being carried up the sides of the retort by the addition of about 10 cc. of pure olive oil to the contents of the retort. The distillation with steam is maintained until the distillate is tasteless and odourless, and is without a trace of oily particles passing over. The volume of distillate in the author's experiments was about 500 cc. The whole of this can be collected in a separating funnel. Sodium chloride is added to it nearly to saturation (150 grms. NaCl to 500 cc. of distillate), and it is then shaken repeatedly with ether until the residual liquid, after expelling the ether, has no smell of the ethereal oil sought for. The author found three shakings with ether—first with from 50—100 cc., then with 50 cc., and again with 50 cc.—quite sufficient. To the ethereal solution sufficient fused calcium chloride (about 20 grms.; so much should be added that the particles of calcium chloride are only bound together and not liquefied by the water extracted) and it is then allowed to stand at least three, and better, four days. The clear water-free ethereal solution is transferred to a tared flask, and the flask, containing the calcium chloride, rinsed out with water-free ether. The ether is then expelled at a temperature not exceeding 30° C. by passing a stream of dry air through the flask until the smell of ether is gone and the weight of the flask remains constant. When necessary, the evaporation of the ether may be done in a dark place, or, instead of air, a stream of dry carbon dioxide or hydrogen, &c., may be used.

The author then describes the results he has obtained by using an exactly neutralised solution of equal parts of sodium salicylate and water, instead of water alone, for extracting the ethereal oils from cloves. He used 10 grms. of powdered cloves in each experiment, and extracted the oils from this amount, in one series using water alone and in another the sodium salicylate solution. The neutralisation of the sodium salicylate is necessary because the commercial salt generally contains a trace of free salicylic acid, which is volatile and would pass over with the ethereal oils. The percentage of eugenol (the actually valuable constituent of oil of cloves) in the oils obtained was determined by separation as benzoyl-eugenol according to Thoms' method (*Zeits. anal. Chem.* **30**, 78).

	Oils.		Eugenol.
	Per Cent.	Per Cent.	
10 grms. of cloves distilled with water yielded—	(1) 18.00	14.91	
	(2) 17.49	14.19	
10 grms. of cloves distilled with sodium salicylate yielded—	(1) 20.20	16.17	
	(2) 18.70	16.70	

From this it is seen that the salicylate distillation yields nearly 2 per cent. more oil and more than 2 per cent. more eugenol than the water distillation. The oil from the water distillation contained 79.44 per cent. of eugenol, and the oil from the salicylate distillation contained 84.52 per cent. eugenol. Thoms (*Pharm. Zeitung*, **35**, 609) found from 76.8 to 90.64 per cent. of eugenol in various samples of oil of cloves. Schimmel and Co. have informed the author that they find that a good oil of cloves contains about 80 per cent. of eugenol. They also told him that the yield of oil from 100 kilos. of Amboina cloves was from 19—20 kilos. The perfect drying of the ethereal extract is very important in the above process. Although the amount of total oil varied somewhat considerably, the percentages of eugenol showed more agreement, but the highest percentages of eugenol, both in the case of the water distillation and salicylate distillation, were obtained when the ethereal solution had stood four days instead of three days with calcium chloride.

In order to explain the action of the sodium salicylate, a series of experiments were next made on the extraction of the ethereal oil of mace (which is almost insoluble in sodium salicylate) from powdered mace. 10 grms. of the sample were distilled with water, with sodium salicylate solution, and further, with 50 per cent. solutions of potassium acetate and calcium chloride respectively, in order to show if the larger yield of oil were not obtained on account of the increased temperature, due to the raising of the boiling point above 100° by the salts in solution. The results obtained were as follows:—

	Per Cent. of Oil of Mace.
Water distillation .....	6.70
Sodium salicylate distillation .....	8.44
Potassium acetate " .....	6.71
Calcium chloride " .....	6.77

The oil from the salicylate distillation was found to contain only a very minute trace of salicylic acid. It is seen, therefore, that in this case also a much larger yield of oil is obtained when sodium salicylate is used. This result must therefore be ascribed to the solvent action of this reagent upon the plant tissue and cells containing the ethereal oils, which thereby sets free the oil and allows it to be carried over by the steam. It is this same solvent action upon tissue which makes sodium salicylate useful in microscope work as a clarifying medium.—H. S. P.

*Lard.* Samelson. *Zeits. anal. Chem.* 1894, **33**, 189—192

THE author finds that the Welman reaction is unreliable as an indication of adulteration with cotton-seed oil, as he obtained a faint blue reaction with undoubtedly pure lard. He also found this reaction less sensitive than the Beechi nitrate of silver test. Beechi's test, it is however admitted, does not react with superheated cotton-seed oil. Gautier's sulphuric acid test he also finds unsatisfactory for detecting admixture with vegetable oils. He concludes that no reliable test for vegetable oils in lard, is known, and considers that the iodine absorption number is the thing most to be depended upon as an indication of adulteration in lard.

—H. S. P.

*Estimation of Fat in Cheese.* S. Bondzyski. *Zeits. anal. Chem.* **33**, 186—189.

THIS is an adaptation of the Werner-Schmid method of fat estimation in milk to testing cheese. A quantity of the finely rubbed-up cheese (the weight that should be taken is not stated) is brought into a glass tube such as is used in the Werner-Schmid method of estimating fat in milk, and 20 cc. of hydrochloric acid of sp. gr. 1.1 are added, and the contents of the tube are warmed gently over wire-gauze until the cheese dissolves and the melted fat rises in a layer to the surface. The tube is then cooled and about 30 cc. of ether are added. The fat dissolves at once in the ether. The contents of the tube are then allowed to stand at 40° C. until the acid solution and the ethereal layer have become sharply separated. The volume of the ethereal solution

is now read, 20 cc. of it are then pipetted off into a tared Erlenmeyer flask, and the fat contained in it weighed after expelling the ether. In comparison with the Soxhlet extraction method, this process gave very good results. In two Erlenmeyer flasks the author found 31.5 per cent. and 32.6 per cent. respectively of fat.—H. S. P.

*Optical Titration of Mannitol.* J. A. Muller. Bull. Soc. Chim. **11**—**12**, 1894, 329—336.

In order to obtain an optical method of assaying wines for mannitol, this sugar was isolated from various natural wines containing it, and carefully compared with mannitol from manna; the identity of the two was clearly demonstrated. Solutions of mannitol in water are inactive towards polarised light, but become active on further addition of certain salts, such as arsenates and borates, or the free acids corresponding therewith. A saturated borax solution gives, with a given quantity of mannitol, a rotatory action varying with the temperature; with 2 to 3 grms. of mannitol per 50 cc. an increase of temperature gives an augmentation in rotation of about 0.1 per degree. Having due regard to this correction, the following table may be used to determine the mannitol present in 50 cc. of fluid saturated with borax:—

Polarimetric Deviation.	Mannitol in 50 Cc.	Polarimetric Deviation.	Mannitol in 50 Cc.
0.0	Grms. 0	0.0	Grms. 1.000
1.0	0.048	10	1.080
20	0.007	20	1.160
30	0.146	30	1.240
40	0.195	40	1.324
50	0.244	50	1.413
1.0	0.293	4.0	1.502
10	0.344	10	1.613
20	0.393	20	1.726
30	0.443	30	1.843
40	0.500	40	1.963
50	0.552	50	2.106
2.0	0.625	5.0	2.250
10	0.688	10	2.335
20	0.751	20	2.430
30	0.814	30	2.571
40	0.876	40	2.742
50	0.958	50	2.975

These figures are apt to be modified by the presence of certain kinds of inactive organic matter, more especially glycerol. In applying them to wine, the liquid should be precipitated by basic lead acetate, and the filtrate treated with sulphuretted hydrogen, and concentrated, preferably only to such an extent that the mannitol present does not exceed 2.5 to 3 grms. per 50 cc. Sugars capable of fermentation, if present in the original wine, must be previously fermented away.—C. R. A. W.

*Conicine and Nicotine.* G. Hent. Arch. der Pharm. **231**, 376.

CONICINE is differentiated from nicotine by its odour, its crystalline hydrochloride, and by the greater difficulty of obtaining with it precipitates with the ordinary reagents. Conicine instantly turns an alcoholic solution of phenolphthalein red, whilst nicotine does not do so. The author uses this reaction as a means of estimating these two alkaloids when mixed. The mixture is titrated with normal

sulphuric acid in the presence of phenolphthalein. As soon as the red coloration disappears (showing the neutralisation of the conicine) the titration of the nicotine is effected with litmus as indicator. L. T. F.

*Acetone Its Valuation.* E. Kopp and E. Grandmougin. Bull. Soc. Ind. Mulhouse, 1894, 112—116.

See under IV., page 729.

*Notes on the Examination of Beeswax.* Lyman F. Kehler. J. Franklin Inst. **137**, 1894, 58.

See under XII., page 745.

## ANALYTICAL AND SCIENTIFIC NOTES.

*Compounds of Hydrogen Cyanide with the Anilides of the Sugars.* W. v. Miller, J. Plochl, and R. Strass. Ber. **27**, 1281—1296.

CONDENSATION products of the sugars with aniline and paratoluidine were first prepared by H. Schiff (Annalen, **140**, 123; **154**, 34), but they were only obtained in the crystalline form some years later by Sorokin (J. Prakt. Chem. [2], **37**, 304).

The authors have discovered the fact that the anilides of the sugars, of which glucose anilide—



is the prototype, combine with hydrogen cyanide. *Anilido-glucose cyanide*,  $\text{C}_6\text{H}_5\text{OH} \cdot \text{CH}_2\text{OH} \cdot \text{CH}_2\text{OH} \cdot \text{CH}(\text{CN})\text{NHC}_6\text{H}_5$ , is obtained by adding an excess of aqueous hydrocyanic acid to a solution of glucose anilide at the ordinary temperature, and allowing the mixture to remain in a cool place. Needle-shaped crystals separate out after a while; the compound melts at 166—168°. *Toluidogluco-cyanide* melts at 128°; *anilidogalacto-cyanide* melts at 138°; *toluidogalacto-cyanide* melts at 145—146°; and *anilidoleuco-cyanide* melts at 131°.

The hydrazone and oxime of glucose were found, under none of the conditions tried, to combine with hydrogen cyanide.—A. R. L.

*The Influence of Moisture on Chemical Action.* H. Breerton Baker. Proc. Chem. Soc. 1894 [139], 111—113.

SINCE the publication of a former note (this Journal, 1893, 553), further investigations of the actions described, have been made. Sulphur trioxide does not combine with highly purified lime; but a trace of moisture brings about vivid incandescence. Dry copper oxide is also without action on sulphur trioxide. Purified and dried lime does not decompose dried ammonium chloride, which may be sublimed from the mixture without evolving any ammonia. Purified and dried nitric oxide gives no brown fumes with dried oxygen, but a trace of moist air causes them to appear at once. A mixture of purified and dried hydrogen and chlorine can be exposed to daylight for four days without more than three-fourths of the gases combining. Ammonium chloride, heated in a vacuum in a soft glass tube, leaves a residue of ammonia gas; but when dried and heated in a hard glass tube, no gaseous residue is obtained. Separation of dried ammonia and hydrogen chloride gases takes place when plates with opposite electrical charges are introduced into the mixture though the action cannot be electrolytic, since discharge is, under the circumstances, impossible. Similar separation was noticed with air and with a mixture of dried hydrogen and oxygen. Electric discharge, when not of high tension, will not pass in dried gases; but the high tension discharge does pass, and renders it possible for the low tension discharge to pass afterwards. With regard to the influence of moisture, it appears that what holds for chemical combination holds also for electrical discharge, and that there is great significance in the apparent similarity in the two cases.

In the discussion which followed, Dr. Armstrong spoke of Mr. Baker's results as of great value and as calculated

to convince all of the important influence exercised by moisture in promoting very many chemical changes; he had long been satisfied that the presence of an electrolyte was essential to every chemical change, and that water was of service because it gave rise to an electrolyte. It was now important to ascertain whether other substances besides water could condition the formation of electrolytes and thus co-operate in producing change, and to determine what their nature was; probably water was one of very few substances able to act in this manner, if indeed it were not the only one. In the case of hydrogen and chlorine, there was reason to think, as Mr. Shenstone had indicated, that some substance other than water was also essential to the occurrence of change.

He was glad to hear Mr. Shenstone say that he was not yet satisfied that pure oxygen can be ozonised; his statement that more ozone was obtained from dried oxygen might be regarded as evidence that water influenced its formation; the amount of ozone finally obtained depended on the amount destroyed as well as on the amount originally formed, and if both its formation and destruction were influenced by moisture, it might well be that one action was more directly influenced than the other when but very little moisture was present. If the destruction took place the less rapidly the less water present, the amount finally obtained would be the greater the more carefully the oxygen was dried.

Regarding the decomposition of ammonium chloride by heat, although moisture would promote its decomposition, the absence of moisture would not have the effect of making it a stable substance at all temperatures; it might be expected that, if dry, it would decompose when heated sufficiently, but would not dissociate; in other words there would be no re-formation of ammonium chloride under such conditions.

## New Books.

**HANDWÖRTERBUCH DER PHARMACIE.** Herausgegeben von A. BRESTOWSKI. Wien und Leipzig: Wilhelm Braumüller. K. u. K. Hof- und Universitäts Buchbändler. 1893. London: H. Grevel & Co., 33, King Street, Covent Garden.

VOLUME II. of this Dictionary is now continued with Parts 15 and 16, which have just appeared. Part 15 commences with the unfinished subject "Mineralwasser," and ends with that of "Niederschlag," which is continued into and ended in Part 16. Part 16 concludes with an article on "Pasta," or Pastes, which will be continued into Part 17.

**TABELLÄRISCHE UEBERSICHT ÜBER DIE KÜNSTLICHEN ORGANISCHEN FÄRBESTOFFE UND IHRE ANWENDUNG IN FÄRBEREI UND ZEUGDRUCK.** Von Dr. ADOLF LEHNE. Herausgeber der Färber-Zeitung. Fünfte Lieferung. Berlin: Verlag von Julius Springer. 1894. London: H. Grevel & Co., 33, King Street, Covent Garden.

This work has now reached the issue of Part V., containing, besides all the requisite tests of the dyes described, also the methods of application, solubilities, powers of resistance, &c., with a column containing beautifully dyed and printed illustrative specimens.

Part V. commences with "Malachite green" and ends with Cyanosine B. (This Journal, 1894, 429.)

**WATER ANALYSIS.** A Practical Treatise on the Examination of Potable Water. By J. ALFRED WANKLYN and E. T. CHAPMAN. Ninth Edition. By J. ALFRED WANKLYN. London: Kegan Paul, Trench, Trübner & Co., Limited. 1894. 5s.

A NINTH edition of this work is now issued, the eighth having become exhausted.

**ANALYSES OF TWELVE THOUSAND PRESCRIPTIONS.** Compiled by W. MARTINDALE. Joint Author of the Extra Pharmacopœia. London: H. K. Lewis, 136, Gower Street, W.C. 1894. 2s. 6d.

THIS little work is dedicated to the General Council of Medical Education and Registration, of the United Kingdom. The Index of the seventh edition of the Extra Pharmacopœia was the basis on which the analyses were compiled, and the last column of figures indicates the pages of the work referred to. The figures are statistics of the frequency of use, not only of the official preparations (printed in italics), but also of unofficial medicaments, so that if carefully examined, they indicate not only the preparations which might be deleted should the Medical Council think fit, but also those which might be made official, *ceteris paribus*, on account of their being frequently prescribed.

The analyses are of the last 12,000 medical prescriptions dispensed antedated to the 1st ultimo, in six pharmacies in typical positions in the United Kingdom, 2,000 at each centre. There are 2,000 from the compiler's own pharmacy, and 10,000 from the pharmacies of friends in Aberdeen, Bournemouth, Carlisle, Cork, and Oxford.

**DIE CHEMISCHE INDUSTRIE AUF DER COLUMBIEN WILTAUSSTELLUNG ZU CHICAGO UND IN DEN VEREINIGTEN STAATEN VON NORD-AMERICA IM JAHRE 1893.** Bericht dem Königlich Preussischen Staatsminister und Minister der Geistlichen, Unterrichts- und Medicinalangelegenheiten, Herrn Dr. Bosse, erstattet von Dr. OTTO N. WITT, Professor der Chemischen Technologie, an der Königl. Technischen Hochschule zu Berlin. Berlin: R. Gaertner's Verlagsbuchhandlung. Hermann Heyfelder. 1894. London: H. Grevel & Co., 33, King Street, Covent Garden. Price 5s.

Two volume, bound in cloth and containing 148 pages of subject-matter, preceded by a short preface and table of contents. An introduction describing the occasion, and the various reasons for, and the nature of the great World's Fair at Chicago, more especially with reference to the chemical industries. The following departments and subjects of that portion of the exhibition devoted to the chemical industries, are treated of and generally described. I.—Fuel and Heat-producing Agents at the Columbian Exhibition and in the United States. A. Natural Gas and Petroleum. Refining Petroleum. B. Natural Products more or less related to Petroleum. C. Coals and Coke. D. Wood as Fuel. II.—Chemical Industries involving Inorganic Chemistry. A. The Raw Materials of the Acid and Alkali Industries. B. Acids and Alkalis. C. Artificial Manures. D. Glass. E. Glass and Pottery. F. Chemical Apparatus. G. Chemical Preparations. III.—Chemical Industries involving Organic Chemistry. A. Industries relating to Dry Distillation. B. The Manufacture of Dye-stuffs. C. Dyeing and Calico Printing. D. Textile Fibres. E. Fats and Soaps.

**NATURE'S HYGIENE.** A Systematic Manual of Natural Hygiene, containing a detailed Account of the Chemistry and Hygiene of Eucalyptus, Pine, and Camphor Forests, and Industries connected therewith. By C. T. KINGZETT. Fourth Edition. London: Baillière, Tindall, and Cox, King William Street, Strand. 1894. Price 10s.

THE third edition of 1888 (this Journal, 1888, 459) having been for some time out of print, the author has now brought out a new one. This fourth edition is the result of a very careful revision of the third, and also of the addition of a chapter on some new developments of bacteriological science (in relation to the subjects of Phagocytosis and Immunity). A further chapter on Alimentation and Foods has been added, and, moreover, the results of the author's recent investigations of the Chemistry of the Aerial Oxidation of Turpentine and other Essential Oils. The author acknowledges the help rendered by his assistant, Mr. D. Homfray, B.Sc.

THE LABORATORY GUIDE. A Manual of Practical Chemistry for Colleges and Schools, specially arranged for Agricultural Students. By A. H. CURRIE, M.A., F.R.S. Seventh Edition, revised. London: Gurney and Jackson, 1, Paternoster Row. 1894. Price 6s. 6d.

8vo. volume, bound in cloth, and containing Prefaces, Table of Contents, and 283 pages of subject-matter, illustrated with 22 wood engravings.

An alphabetical index of subjects, and a list of apparatus required by the student in following the course advocated in the work, complete the same.

It is announced that, besides a careful revision of the sixth edition, this, the seventh, contains an additional Lesson on Soils, which is now introduced in Part I., whilst certain improved Quantitative Methods appear in Part III. Three processes, now lapsed and obsolete, have been allowed to disappear from the volume: these are the citric acid method for determining phosphorus pentoxide, the old process of extracting fat from milk residues, and the sulphuric acid method for the isolation of indigestible fibre.

The text is sub-divided as follows:—I. Chemical Manipulation. Preparations and Experiments. II. Qualitative Analysis. III. Quantitative Analysis. Analysis of Manures. Analysis of Soils, Waters, and Foods.

The volume is strongly bound in a good waterproof cover.

## Trade Report.

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

(From the Board of Trade Journal.)

#### CANADIAN TARIFF CHANGES—continued.

(This Journal, June 1894, p. 672.)

Articles.	Rates of Duty.
<i>Sugar, Syrups, and Molasses.</i>	
All sugar above No. 16 Dutch standard in colour, and all refined sugars and molasses, N.O.P., of whatever kinds, grades, or standards.	Per lb. 1½ cent.
Sugars, syrups, and molasses of all kinds, N.O.P., the product of the sugar-cane or beetroot, N.E.S., and all imitations thereof or substitutes thereof.	" 1 cent.
Glucose or grape sugar, glucose syrup and corn syrup, or any syrups containing any admixture thereof, a specific duty of.	" 1 cent.
Sugar candy, brown or white, and confectionery, including sweetened gums, candied peel, and pop-corn.	35 <sup>+</sup> ad val.
Liquorice paste, and liquorice in rolls and sticks.	20 <sup>+</sup> ad val.
Molasses produced in the process of the manufacture of cane-sugar from the juice of the cane, when imported in the original packages from the district where produced in the country where the cane was grown, and which has not been subjected to any process of treating or mixture after leaving the country from which originally shipped. The packages in which imported when of wood to be free.	
(a.) Testing by polariscope, 49° or over, a specific duty of.	Per gall. 12 cents.
(b.) When testing by polariscope, less than 40° and not less than 35°, a specific duty of.	" 1½ cents.
For each degree or fraction of a degree less than 40°.	Per degree additional 1 cent.

#### CANADIAN TARIFF CHANGES—continued.

Articles.	Rates of Duty.
<i>Small Arms.</i>	
Fertilisers, compounded or manufactured.	10 ad val.
Fireworks.	25 ad val.
Gun, rifle, and pistol cartridges, cartridges cases of all kinds and materials, percussion caps and gun wads of all kinds.	30 ad val.
Blasting and mining powder.	Per lb. 2 cents.
Cannon, musket, rifle, gun, and sporting powder and canister powder.	" 3 cents.
Nitro-glycerine, gun powder, and nitre, and other explosives.	" 4 cents.
Photographic dry plates.	30 ad val.
Oil, sperm, whale and other fish-oils, and all other articles the produce of the fisheries, not specially provided for.	20 ad val.

The following are free:—Acids used for medicinal, chemical, or manufacturing purposes not specially provided for in this Act; alum in bulk only; aluminium and its salts; ambergris; ammonia and its salts; aniline salts, and dyes in bulk, and alizarine; aniline oil, annatto; antimony; arsenic; asphalt; bismuth; blood, albumen, and tannic acid; bones, emude.

#### UNITED STATES.

##### Customs Decisions.

Hydrated oxide of iron is dutiable at 25 per cent. *ad valorem* under paragraph 76, N.T.

Magnesite bricks of the kind used in furnaces for the smelting of lead ores are dutiable at 1.25 dols. per ton under paragraph 93.

A compound known as encalyptol camphyline is dutiable at 25 per cent. *ad valorem* under paragraph 76, N.T.

Coffee substitute put up in papers and in tins, and composed of a mixture of chicory-root, beet-root, oil, syrup, and other ingredients, is dutiable at 1½ cents per pound under paragraph 321, as substitutes for coffee.

Crude olein or oleic acid, a fat oil of animal origin, chiefly and commonly used in soap making, and in its condition as imported fit only for such uses, is entitled to free entry under paragraph 599.

### EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

#### IRON MINING IN NORTHERN SPAIN.

Mr. Victor de Larrea, Acting British Consul at Bilbao, in the annual commercial report of that consular district for the year 1893, gives the following particulars of the mining of iron ore in Spain, special reference being made to the Biscay district:—

There is no doubt that in Spain there yet exist abundant deposits of minerals, particularly of iron ore, which being far inland remain unworked, partly from want of easy means of conveyance and partly from want of enterprise.

The total number of the mines registered in Spain is over 15,000, of which only about 2,000 are worked.

The province of Biscay, however, still gains very considerable profits from its rich iron ore mines, as may be deduced from the fact that in 1893 of a total production in Spain of 5,497,540 tons, the consular district of Bilbao was responsible for 4,918,650 tons; whilst in 1892, out of a total for Spain of 5,405,170 tons, the consular district of Bilbao furnished 4,573,560 tons.

The exportation of iron ore from Bilbao to the United Kingdom in 1893 was over 3,000,000 tons, and, as the total foreign import of iron ores into the United Kingdom was

1,065,563 tons (3,624,083 tons from Spain and 441,780 tons from other countries), it appears that shipments of Bilbao ores made up 84 per cent. of the importation from Spain and 75 per cent. of the total foreign importation of iron ores in the United Kingdom in 1893; and, calculating the yearly production of iron ores in the United Kingdom to be 14,000,000 tons, it would appear that Bilbao supplies about the fifth part of the iron ores yearly consumed in the United Kingdom.

With regard to the output of iron ore in Biscay, and the very considerable quantities exported for the last 16 years at the rate of 3,000,000 tons to 4,000,000 tons per annum, the question has often been raised as to how long the mines may be expected to continue this yield. This is certainly a matter very difficult to forecast, but it may be observed that the number of mines yielding good quality ores is becoming smaller, with the inevitable result that in the course of time the exportation will decrease and the quality deteriorate.

The forms of the iron ore deposits in the province of Biscay are simple and practically on the same geological horizon, a circumstance which adds materially to a ready comprehension of them. They are bedlike in form generally, and appear with an exposed surface, or simply covered by shale, or overlaid by heavy beds of limestone.

The deposits are all quarried, and for quarrying operations the explosives used are powder and dynamite.

The number of men employed in the working of the iron ore mines in the province of Biscay is estimated at 12,000. —(No. 1398, *Foreign Office Annual Series*.)

#### OPIMUM ADULTERATION IN CHINA.

The following note on the subject of opium adulteration is taken from the commercial report for 1893 of Mr. R. W. Hurst, Her Majesty's Consul at Tientsin:—

Sesamum seed cake is now quite out of use for opium adulteration, being supplanted by "Tientsin" cake, a repulsive preparation of boiled-down skins and all kinds of refuse of a "gelatinous" nature, to which, in boiling, has been added opium pod, stalk, husk, &c.—in fact, anything that would impart to it the taste or smell of opium.

It is used for the adulteration of opium in increasing proportions; last year two or three parts of the cake to seven or eight of opium; this year three or four parts of the cake to six or seven parts of opium. The price in 1892 averaged from 17 dols. per cwt. to 25 dols. per cwt.; this year 54 dols. per cwt. to 42 dols. per cwt. It is imported under the name of "medicinal cake" mostly by foreign vessels, but to some and to an increasing extent also by junk. The poorer classes buy a mixture of nearly equal parts of opium and "Tientsin cake."

The cake is sold by the dealers with each chest of opium sold, the adulteration being done by the wholesale dealers in prepared opium, and again by the retail dealers.—(No. 1401, *Foreign Office Annual Series*.)

#### GENERAL TRADE NOTES.

##### PETROLEUM DISCOVERIES IN THE CAUCASUS.

The *Bulletin du Musée Commercial*, in its issue for the 2nd June, states that discoveries of petroleum were made some time ago in the Northern Caucasus, and especially in the district of Groznoje; later soundings have given very remarkable results.

During the month of October 1893 soundings were made in this district to a depth of 440 ft.; the results obtained were very good as regards quality and quantity.

According to the *Journal de la Chambre de Commerce de Constantinople* this experiment was an innovation, for hitherto wells of little depth, like those sunk at Bakn, had been made.

The yield of the wells referred to was, for a period of four days, 200,000 pounds, or about 3,280 tons. This yield would probably have been larger if, owing to the manner

in which the pipes were connected, a large quantity of water coming from the upper beds of soil had not mixed with the naphtha.

The product was gathered in a reservoir built in the ground, and representing a capacity of 10,000 tons; its further treatment showed that it contained a greater proportion of lighting oil and a smaller proportion of residues than Bakn petroleum.

More recently two other wells have been opened by another firm, with diameters of 20 and 9 ins., and new soundings are now being suggested. Carriage will be effected by the Vladikavkaz-Petrovsk railroad.

##### THE MATCH MONOPOLY IN FRANCE.

The *Economiste Français* for the 2nd June publishes the following particulars of the results of the match monopoly of 1893, taken from the report of the general direction of State manufactures. There were manufactured 28,422,242,550 matches (27,006,377,050 of wood and 1,415,865,500 of wax) ready to be delivered for sale. The cost of manufacture was 3,394,270 francs; the average cost for manufacture was therefore 119.42 francs per million matches. The general cost price of the matches ready for sale comes to 194.59 francs per million matches, which are sold wholesale for about 900 francs. The regie expended last year 6,349,006 francs. It sold wholesale or into the warehouses 29,340,192,390 matches, corresponding to receipts aggregating 25,874,542 francs. The profit on the manufacture of matches was therefore 19,500,000 francs, to which should be added the increase in the capital of the regie, which gives a net profit of 20,072,456 francs.

##### THE MINERAL STATISTICS, 1893.

The official mineral statistics for the year 1893 have just been completed and issued by the Home Office. Taking the returns in alphabetical order, we find the quantity of alum clay or bauxite was 8,740 tons, as against 7,322 tons in 1892—entirely raised in Antrim, Ireland. Of aluminium there are only two makers in the United Kingdom, and no details are procurable of their production. Alum shale is raised in Yorkshire only, from a bed lying between two seams of the Stanley Main bed of coal, and the quantity was 2,115 tons, against 2,922 tons in 1892. Antimony ore is mined in Cornwall, but none was raised last year, and only very small quantities in previous years. Arsenic is produced in Cornwall and Devonshire, the quantity last year being 5,976 tons, as against 5,114 tons in 1892. Of arsenical pyrites the quantity was 3,036 tons, as compared with 4,497 tons. The quantity of barytes (mainly sulphate) was 22,343 tons, against 24,247 tons in 1892, the principal districts being Northumberland, Shropshire, and Ireland. Of bismuth there is only one producer, and no returns are given. Bog ore, chiefly used for gas-purifying, is exclusively obtained in Ireland, the quantity last year being 10,747 tons, against 15,363 tons in 1892. Of clays, not including ordinary clay, the quantity was 3,065,208 tons, against 3,103,852 tons in the previous year. Of coal, the total quantity mined last year was 164,325,795 tons, as compared with 181,786,871 tons—the decrease being attributable, no doubt, to the coal strike. There is only one mine raising cobalt and nickel ore, and it is closed at present. Copper-mining is an industry which the report says is "rapidly decreasing in Britain," the output having fallen from 210,000 tons in 1863 to a little over 5,000 tons in 1893, and 6,265 tons in 1892. Our imports of copper come from various parts of the world, but, as for many years past, principally from Spain and the United States, the latter country sending 37,761 tons, and Spain 48,709 tons last year. The total quantity of copper obtained by smelting in the United Kingdom last year was 69,810 tons, against 78,346 tons in 1892. Of fluorspar the quantity was 215 tons, compared with 171 tons. Of gold ore there was raised 4,489 tons, yielding gold valued at 7,657*l.*, as against 9,990 tons and 9,168*l.* in 1892. All the mines are at Dolgelly. Of gypsum the output was 143,486 tons, against 147,540 tons in 1892. The aggregate quantity of iron ore raised last year was 11,203,476 tons, against 11,312,675 tons in the previous year. The quantity of iron ore imported was

4,065,864 tons, compared with 3,780,503 tons in 1892. It is of some interest to note that there were 327 furnaces in blast in 1893, making 6,976,940 tons of pig iron from 16,620,653 tons of ore, and using 13,806,728 tons of coal. Of iron pyrites the quantity raised was 15,837 tons, against 13,967 tons the year before. The quantity of jet produced was 888 lbs., as against 929 lbs. in 1892, the output of this mineral having decreased greatly since 1886. Our production of lead ore was 10,808 tons, yielding 29,698 tons of lead, as compared with 10,024 tons of ore and 29,510 tons of lead in 1892. The principal districts are Durham, the Isle of Man, Flintshire, and Derbyshire. Our imports of lead and lead ore were 2,857 tons, against 18,217 tons in 1892, and our exports of British pig lead 29,735 tons, against 39,178 tons, and of lead sheets, piping, &c., 19,366 tons, against 18,984 tons in 1892. These were in addition to 18,836 tons of foreign pig and sheet lead last year, against 15,613 tons in 1892. Only one British mine yields lignite, the quantity being 3,264 tons, against 4,217 tons in 1892. Manganese ore figures for 1,336 tons, compared with 6,078 tons in 1892, whereas our imports of this mineral last year were 121,773 tons. Of ochre, amber, &c., our production was 10,534 tons, against 12,131 tons in 1892. Oil-shale was raised, chiefly in Scotland, to the total of 1,956,520 tons, against 2,089,937 tons in the previous year. Lindlithgowshire and Edinburghshire are the principal districts. Of petroleum (found only in North Staffordshire on a scale worthy of being recorded) we produced 26 tons, against 18 tons in 1892. The total of phosphate of lime was 3,300 tons, as against 12,200 tons in 1892, the increasing imports of cheaper foreign materials having serious effects upon this Bedfordshire and Cambridgeshire industry. Our output of salt last year was 1,924,029 tons, as compared with 1,956,524 tons in the previous 12 months. Of silver, all produced from argentiferous lead ores, the quantity was 274,160 oz. Of slates the quantity was 138,993 tons, against 118,241 tons in 1892. Sodium is made by four firms, but no returns are given. Strontia sulphate is dug from shallow pits in Gloucestershire and Somersetshire, the quantity being 5,812 tons, as against 5,066 tons in 1892. Of tin ore we produced 13,688 tons, against 11,355 tons, the yield of tin being 8,837 tons, as compared with 9,270 tons in 1892. Our imports of tin were 671,162 cwt., compared with 589,365 cwt. the previous year. One mine only, in Cornwall, yields uranium, the quantity being 25 tons, against 37 tons in 1892; and one mine only, also in Cornwall, raises wolfram, last year's output having been 22 tons, against 125 tons in 1892. Of zinc ore our output was 23,754 tons, yielding 9,284 tons of metal, against 23,880 tons and 9,349 tons respectively in 1892.—*Ironmonger.*

#### THE ENGLISH AND GERMAN CHEMICAL INDUSTRIES.

The report on technical education presented in July, 1891, to the Manchester Whitworth Institute by the deputation which was appointed by the council of the Manchester Technical School to visit the technical schools and institutions in England and on the Continent, was remarkable for the statements made therein respecting the lack of efficient technical instructors in certain of our industries. It added:—"The forethought of Continental Governments in this respect (viz., the supply of properly qualified teachers), may be compared, in several very interesting and striking directions, with our own want of provision. For instance, when this council has required the services of a competent instructor for the chemical, dyeing, and calico-printing classes, it has been found indispensable to engage one who has been trained in a Foreign Technological Institution for that important section of its work; and it is well known that many of our leading firms experience almost insuperable difficulty in finding, amongst our own countrymen, that combination of scientific with practical knowledge, by which alone they can hope to compete with their Continental rivals."

This is surely very severe condemnation of our want of system in training competent teachers and assistants for the chemical trade of this country. Indeed, it is partly due to this negligence that Germany has succeeded in building up a very extensive trade in chemicals and chemical compounds of all descriptions.

If we take the export trade of this country in alkali, bleaching materials or preparations, chemical manure, mineral preparations, &c., we shall find that it has not made that progress which we might have expected under proper conditions. The appended table shows the values of our exports in the years named—

	1888.	1891.	1892.
Alkali .....	£ 1,638,770	£ 1,512,000	£ 1,087,206
Bleaching materials or preparations .....	615,081	564,113	506,976
Manure (chemical) .....	1,849,035	2,000,170	2,070,216
—	—	—	—
	1891.	1892.	1893.
Alkali .....	£ 2,035,112	£ 2,119,791	£ 1,858,157
Bleaching materials or preparations .....	522,445	508,619	551,167
Manure (chemical) .....	2,111,370	2,137,811	2,312,967

The figures relating to chemical manure indicate a small expansion, but those dealing with the export of alkali and bleaching materials indicate a contraction of trade. There was a small spurt in the years 1890-92, but last year's trade was only fractionally better than that of 1888, as regards alkali.

On the other hand, if we refer to statistics dealing with Germany's exports, we find that she has increased her shipments to this country of the following goods, comparing 1889 with 1891, the latest year for which figures are available:—

	from	Metric Centners.*
Caustic soda	228 to 3258	
Alum	26987 to 33,137	
Soda calcined	3373 to 21,164	
" crude	621 to 19,947	
Ammonia and coal-tar	13,867 to 18,194	
Sulphuric	1,379 to 19,087	

\* Metric Centner: About 35 lbs. avoirdupois.

It is significant that our exports to Germany have decreased in the case of bleaching powder from 34,555 metric centners to 28,451 centners; from 278,396 to 198,840 metric centners in the case of sulphate of ammonia; from 34,001 to 12,264 metric centners for catechu; from 27,706 to 19,526 metric centners for cinchona bark; from 22,678 to 14,100 metric centners for carboic acid. The list could be further extended, but it is sufficient to show that in many lines of chemical industry we have been losing ground with Germany, while that country has gained the trade. The comparisons quoted above are 1889 with 1891.

In some lines Germany has an absolute monopoly, as, for instance, in manufacture of kieserite, which is made exclusively in that country. The chemical trade of Germany in many articles is sufficiently strong to disregard the Customs barriers raised by other countries in the shape of protective tariffs. When a foreign country imposes duties on articles of her chemical industry, these do not lose their sphere of trade; the foreign country simply pays the duties because cannot do without German chemicals.

It would be very interesting to inquire how it is that Germany has been able to occupy this strong position. The answer is partly to be found in the superior system of technical training, as applied to this industry, pursued in Germany, and partly to some natural advantages. The fact that her system of training is superior to our own must be admitted when we have to go to a foreign country for a competent person to teach the art of dyeing and calico printing to our own operatives. Up to the present the cost of production, so far as labour is concerned, has been rather favourable to German manufacturers. In England, according to Mr. Charles Booth (*Chem. Tr. Jour.*, 341, p. 351), 5 per cent. of the persons engaged in our chemical industry receive under 20s. per week; 25 per cent. earn from 20s., but under 25s.; 20 per cent. receive from 25s., but less

than 30s.; 25 per cent. earn from 30s., but under 35s.; 15 per cent. from 35s., and less than 45s.; while the remaining 10 per cent. receive 45s. and upwards. In America we know that most of the employes engaged in making chemical preparations (compounded) receive from 36s. to 40s. a week, while those engaged in the manufacture of dyestuffs earn from 32s. to 3*l*. and even more (this Journal, 1894, 557). In Germany it is undoubtedly less, as the cost of living is much cheaper than in England or the United States, although it is known that the tendency of late years has been to increase the cost of living in Germany—more especially in the principal towns.

Although Germany has been extending her foreign trade in this respect, yet it does not follow that her manufacturers have been proportionately successful in obtaining good pecuniary results. Indeed, so far as statistics are available, the reverse appears to be the case. Out of 85 factories, no fewer than 24, or 28·23 per cent. of the whole, have yielded no dividend for some years; four have paid a maximum dividend of 2½ per cent., or 4·71 of all the companies; 7, or 8·25 per cent. of all the companies, have paid a maximum dividend ranging from 2½ to 5 per cent.; 8, or 9·41 per cent., have paid a maximum dividend of from 5 to 7½ per cent.; 22, or 25·88 per cent., have paid a maximum dividend of from 7½ to 10 per cent.; while 13, or 15·29 per cent., have declared dividends of from 10 to 15 per cent., 7, or 8·24 per cent., have paid a dividend of more than 15 per cent.—(*Chemical Trade Journal*).

#### MANGANESE ORE IN VIRGINIA.

Some very fine manganese ore has recently been found on the Glenmore property, about two miles from White Sulphur Springs, Va., and the deposit or series of deposits has been traced on the surface outcrop for nearly two miles. The first carload of the ore taken out was shipped to the Carnegie Steel Company, Pittsburgh, and was settled for on the following analysis: Iron, 0·200 per cent.; manganese, 49·964 per cent. As no deduction was made for phosphorus, it is safe to assume it to have been below the Bessemer limit. Other analyses of samples of this ore give an average of about 0·1 per cent. phosphorus, and from 1 per cent. to 3 per cent. silica; the manganese going up to 55 per cent., and iron sometimes down to a trace. This ore is pure enough for chemical manufactures, and as the amount of it seems to be very great, it promises to be a very important find. Some 200 or 300 tons of the ore are now being shipped to the Carnegie Steel Company, which pays from 28 to 30 cents per unit for the manganese and 0·06 cent. for the iron. Such ore would, however, be worth much more to the chemical manufacturers than to the steelmakers on account of its remarkable freedom from iron and its high contents in manganese. For the information concerning this important find we are indebted to Mr. Wm. N. Page, manager of the Ganley Mountain Coal Company, at Ansted, W. Va.—(*Engineering and Mining Journal*).

#### ARTIFICIAL CITRIC ACID.

For some considerable time there have been rumours of the artificial production of citric acid from a sugar solution, by growing in it a fungus. The new method has been the subject of prolonged experiment in a chemical factory in Alsace well known for the many synthetic products that have been brought into commerce by it. The news of the discovery has created much uneasiness in the West Indies—notably in Dominica—where the preparation of concentrated lime-juice for citric acid manufacture is an industry of some importance. The uneasiness has been allayed somewhat, however, by a letter read by Dr. Alfred Nicholls, of Dominica, from a London drug firm, in which the opinion is expressed that the experiments are not likely, within the near future, to reach a stage at which they would become dangerous to the lime industry. This letter appears in the current issue of the *Kew Bulletin*, in which is also printed a communication, dated 13th June, from the British Ambassador at Berlin on the same subject. Sir E. B. Malet states that, according to information which he has received from the Imperial German Foreign Office, the director of the chemical works at Thann, in Alsace, states that the result

of the experiments in the production of citric acid from sugar solution increases the prospect of ultimate success, but that certain difficulties have been encountered which must be overcome before there can be any question of the mercantile utilisation of the process.—(*Chemist and Druggist*).

#### THE EXPORT OF TINCTURES AND ESSENCES.

##### *Concessions by the Board of Inland Revenue.*

The following general order, No. 15, 1894, dated June 18th, 1894, containing further regulations on the subject of the exportation, on drawback, of tinctures and flavouring essences, has recently been issued by the Board of Inland Revenue:—

The attention of the service is directed to the following changes in the existing regulations affecting the exportation of tinctures and flavouring essences:—

1. Tinctures and flavouring essences may be exported in bottles or metal canisters, holding any quantity, packed in cases each containing not less than two liquid gallons. Essences, however, in bottles not exceeding 4 oz. each, may be packed in 1-gallon cases, but no consignment less than 2 gallons can be exported. The bottles or canisters may be of different sizes, but the number of such sizes that may be packed in a case is not to exceed three. Tinctures and essences may be packed in the same case.

2. Jars, covered with wicker-work, containing not less than two and not more than five gallons, and iron drums holding not less than two and not more than ten gallons, may be exported without being cased, but a wooden tally showing the distinguishing particulars required to be branded, or inscribed on a case, must be affixed securely by wire to the neck of each jar or drum.

3. The exporter must set apart the bottles or canisters intended for each case, grouping each size, so that the officer may conveniently count them and test their contents. When metal canisters are used they must not be finally corked, or otherwise closed, until the officer has fully satisfied himself that the contents correspond with the specification in the notice to pack.

4. The quantity of tincture or of flavouring essence in any bottle or canister, whether to be invoiced by weight or otherwise, is to be ascertained by imperial measure.

The exporter, in his notice to pack, must specify the following particulars against each different size of bottle or canister:—

(a.) The quantity in bulk to the nearest thousandth part of a gallon which each bottle or canister contains. And as to bottles not exceeding four ounces each, the total contents of six such bottles may be declared.

(b.) The total number of bottles of each size.

(c.) The total quantity in bulk gallons and parts of a gallon.

(d.) The alcoholic strength at proof.

(e.) The proof gallons and parts of a gallon.

5. Whenever practicable the officer should check the bulk quantity by the ordinary standard imperial measures provided by the exporter. When the quantity in any bottle or canister does not correspond to the recognised imperial measure, as a gallon, quart (0·250 gallon), pint (0·125 gallon), or half-pint (0·062 gallon), graduated glass measures, which will be supplied to supervisors on application to the principal of the laboratory, are to be used, and the quantity checked to the thousandth part of a gallon. The calculations for drawback are to be regulated by the order of February 4, 1893, whether the bottles or canisters be measured singly or in groups of six.

6. When the number of bottles in any consignment does not exceed 100, the officer is to measure 1 in every 25. When it exceeds that limit, then 1 in every 50 after the first 100. When canisters are used the measurement of not less than 1 in 10, and, if jars or drums, not less than 1 in 5, should be checked. A larger proportion of bottles may be measured should the officer, from any exceptional circumstances, consider it necessary.

7. Spirits of nitre need no longer be sampled in each consignment. This preparation however, should be frequently sampled, but when of the same strength one



sample from any number of consignments, on the same day, and belonging to the same exporter, will be sufficient.

8. The exporter should, as a general rule, enter on separate forms preparations packed in any consignment which carry different rates of allowances for waste, or which are not scheduled as entitled to any such allowance. If more convenient, however, they may, in the case of small consignments, be shown in separate groups on the same form, and in either case the allowance claimed must be stated—*Chemist and Druggist*.

#### COAL IN IRELAND.

During the past 18 months boring operations have been carried on at Dungannon, under the direction of Mr. Donald Munro, to prove the existence of coal. The work was brought to a successful issue this month, when a seam of bituminous coal, 5 ft. 11 in. in thickness was struck. The depth at which the seam was discovered is 188 yards from the surface. Arrangements will be made by the Dungannon Collieries Company, Limited, for working the coal at an early date. The company own 1,800 acres of land in the neighbourhood, and the engineer is of opinion that coal lies under the whole of this area. A stratum of red sandstone intervenes between the surface and the coal measures.—*Chemical Trade Journal*.

#### THE NITRATE OF SODA MARKET.

The market opened in January at 9s. per cwt. as the value of arrived cargoes, and 9s. 1½d. to 9s. 3d. for spring arrival, holders being in a very confident mood.

The actual consumption in Europe for the half-year amounts to 588,000 tons, of which the Continent took 503,000 tons and the United Kingdom 85,000 tons. This represents an increase of 11 per cent. on the Continent and

20 per cent. in the United Kingdom as compared with the same period last year. Such an increase we fully anticipated in our circular of 31st December last.

There is no material change to note in the consumption in the United States, which remains very stationary at the rate of about 100,000 tons per annum.

The consumption in Europe for the first six months of the past three years has been as follows:—

	1892.	1893.	1894.
	Tons.	Tons.	Tons.
Continent .....	493,000	451,000	503,000
United Kingdom .....	84,000	71,000	85,000
	577,000	522,000	588,000

Whilst the consumption for the twelve months ending 30th June 1892, 1893, and 1894 has been:—

	1892.	1893.	1894.
	Tons.	Tons.	Tons.
Continent .....	613,000	633,000	733,000
United Kingdom .....	114,000	102,000	114,000
United States .....	100,000	100,000	100,000
	827,000	835,000	947,000
The shipments for the same period have been.....	907,000	765,000	1,004,000

#### NITRATE OF SODA.

*Shipments, Consumption, Stocks, and Prices, from 1887 to 1894.*

		1888.	1890.	1892.	1894.
Shipments from South American ports to all parts for the six months ending 30th June.	Tons	268,000	335,000	402,000	450,000
Do. for the 12 months ending 30th June.....	"	715,000	909,000	907,000	1,004,000
Afloat for Europe on 30th June.....	"	148,000	138,000	180,000	225,000
Stocks in United Kingdom ports:—					
		1888.	1890.	1892.	1894.
Liverpool.....	Tons	5,500	8,000	8,000	2,000
London.....	"	2,500	2,300	1,700	2,700
Out ports.....	"	4,000	7,700	7,300	2,300
		12,000			
		18,000			
		17,000			
		7,000			
Stocks in Continental ports on 30th June.....	Tons	34,000	98,000	107,000	27,000
Consumption in United Kingdom for the six months ending 30th June.	"	75,000	91,000	84,000	82,000
Do. in Continent do. do.....	"	310,000	495,000	493,000	503,000
Do. in United Kingdom for the 12 months do.....	"	105,000	123,000	114,000	114,000
Do. in Continent do. do.....	"	185,000	700,000	645,000	734,000
Do. in United States do. do.....	"	55,000	75,000	100,000	100,000
Do. in the World do. do.....	"	645,000	898,000	860,000	948,000
Visible supply on 30th June (including the quantity afloat, for Europe and Stocks in United Kingdom and Continent) .....	"	180,000	254,000	304,000	258,000
Price on 30th June .....	per Cwt.	9s.	8s.	8s. 3d.	9s. 1½d.



## THE BENGAL SHELLAC INDUSTRY.

THE French Consul at Calcutta, in a report upon the present condition of the Indian lac industry, states that the finest descriptions of stick lac are found in Assam and Bengal, that coming from the former country being a very important article of trade. Lac from Burmah, which is chiefly produced in the upper districts and the Shan States, is sent to Calcutta to be worked up. Burmah, it is stated, is in a position to supply endless quantities of lac, as the vast forests there contain many descriptions of trees adapted to its development. The districts of the Punjab and Mysore are large producers of lac, which is chiefly used at the place of production, its inferior quality rendering it unfit for exportation. Then come Bengal, Oudh, Seinde, and the Central Provinces. According to quality, it is either sent to Calcutta to be melted, or to certain towns of the interior, such as Hyderabad and Mirzapore, for working up into bangles and other articles. There are large numbers of lac factories in India, but most of them are of little importance, and only turn out products of very second-rate quality. The methods of manufacture vary according to district. In hat-making, a mixture of lac, mastic pounce, and other resins, dissolved in alcohol, is used to stiffen silk hats, and in lithography lac is used in connection with the preparation of the ink. Mixed with resin and certain colouring material it makes sealing-wax. Lac also enters into the composition of numerous varnishes. In adding to a solution of lac in alcohol a yellow clear substance such as gum gutta, saffron, &c. a liquid is obtained which gives to copper and other metals the appearance of gold, while still preserving their brightness. The quantity of lac exported in 1892-93 amounted to 125,246 cwts. valued at 7,787,583 rupees. In the preceding year the value amounted to 7,444,460 rupees.

— *Ibid.*

## BOARD OF TRADE RETURNS.

## SUMMARY OF IMPORTS.

Articles.	Month ending 30th June.	
	1893.	1894.
	£	£
Metals.....	1,719,400	1,727,878
Chemicals and dyestuffs.....	401,262	404,124
Oils.....	554,143	633,748
Raw materials for non-textile industries.....	3,462,845	4,562,931
Total value of all imports....	31,869,592	34,250,031

## SUMMARY OF EXPORTS.

Articles.	Month ending 30th June.	
	1893.	1894.
	£	£
Metals (other than machinery) ....	2,934,051	2,620,433
Chemicals and medicines .....	749,734	735,366
Miscellaneous articles.....	2,670,898	2,337,341
Total value of all exports.....	18,785,271	17,909,155

## IMPORTS OF OILS FOR MONTH ENDING 30TH JUNE.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Cocoa-nut..... Cwt.	3,323	33,507	4,436	39,639
Olive .....	700	3,573	29,580	122,528
Palm .....	119,509	97,806	141,357	103,875
Petroleum .....	11,161,189	11,016,972	172,352	172,093
Seed .....	1,738	2,228	41,139	47,754
Train, &c.....	2,446	2,351	49,761	39,875
Turpentine .....	33,671	33,790	37,968	37,254
Other articles .. Value £	..	..	77,541	70,740
Total value of oils...	..	..	554,143	633,748

## IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDING 30TH JUNE.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Bark, Peruvian .. Cwt.	5,849	2,484	9,547	4,100
Bristles..... Lb.	456,492	310,013	71,634	45,716
Caoutchouc..... Cwt.	21,655	24,723	220,878	244,373
Gum :—				
Arabic..... "	4,448	4,470	10,901	9,533
Lac, &c..... "	12,090	10,450	58,215	54,737
Gutta-percha .... "	3,279	3,168	22,978	20,902
Hides, raw :—				
Dry..... "	29,150	43,888	87,268	104,862
Wet..... "	41,012	44,698	85,736	88,388
Ivory..... "	791	1,215	33,261	42,026
Manure :—				
Guano..... Tons	2,265	3,838	8,999	17,269
Bones..... "	2,879	3,063	12,763	13,563
Nitrate of soda... "	4,557	9,678	49,952	91,957
Phosphate of lime "	27,811	29,919	55,595	49,356
Paraffin..... Cwt.	64,399	15,520	66,105	16,124
Liñen rags..... Tons	1,223	1,030	10,794	10,450
Esparto..... "	15,388	17,668	68,950	76,684
Pulp of wood .... "	17,692	21,681	100,849	110,533
Rosin..... Cwt.	101,518	159,330	26,967	39,416
Tallow and stearin "	183,117	270,850	260,177	339,355
Tar..... Barrels	1,282	4,457	817	3,483
Wood :—				
Hewn .....	196,083	231,694	339,875	477,834
Sawn .....	153,380	745,043	972,842	1,625,142
Staves .....	7,854	20,657	34,987	67,215
Mahogany .....	4,004	6,118	32,027	49,641
Other articles.... Value £	..	..	819,728	760,182
Total value .....	..	..	3,462,845	4,562,931

Besides the above, drugs to the value of 63,797, were imported, as against 50,725, in June 1893.

IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH  
ENDING 30TH JUNE.

Articles	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Alkali..... Cwt.	8,967	29,305	8,091	13,281
Bark (tanners', &c.) ..	30,309	33,117	13,997	11,208
Brimstone..... "	30,877	19,954	8,267	5,976
Chemicals..... Value £	..	..	162,481	113,759
Cochineal ..... Cwt.	438	72	2,532	443
Cutch and gambier Tons	1,138	1,743	27,664	38,661
Dyes:—				
Aniline ..... Value £	..	..	11,556	25,713
Alizarin ..... "	..	..	27,212	14,757
Other ..... "	..	..	3,312	2,847
Indigo ..... Cwt.	738	2,250	12,069	33,729
Nitrate of potash ..	20,992	20,591	18,281	19,491
Valonia ..... Tons	2,401	2,809	31,039	34,799
Other articles... Value £	..	..	131,101	119,559
Total value of chemicals	..	..	401,262	461,124

## IMPORTS OF METALS FOR MONTH ENDING 30TH JUNE.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Copper:—				
Ore..... Tons	7,546	7,894	56,262	45,136
Regulus ..... "	11,088	7,334	261,583	152,454
Unwrought .... "	1,817	5,620	83,148	221,701
Iron:—				
Ore..... "	373,615	453,269	254,238	301,576
Bolt, bar, &c.... "	7,814	4,944	71,066	46,617
Steel, unwrought.. "	608	576	4,869	4,420
Lead, pig and sheet ..	13,013	15,339	167,771	139,187
Pyrites ..... "	33,335	52,527	91,967	90,551
Quicksilver..... Lb.	698,820	675,355	62,841	53,564
Silver ore..... Value £	..	..	269,616	272,446
Tin ..... Cwt.	35,824	41,888	156,965	148,680
Zinc ..... Tons	4,623	4,646	78,352	71,602
Other articles ... Value £	..	..	160,191	176,944
Total value of metals	..	..	1,719,400	1,727,878

EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH  
ENDING 30TH JUNE.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Gunpowder..... Lb.	719,600	491,600	16,645	9,119
Military stores... Value £	..	..	8,867	8,151
Candles..... Lb.	1,463,300	1,597,800	27,801	27,584
Caoutchouc..... Value £	..	..	56,639	51,707
Cement..... Tons	18,884	19,997	84,591	79,579
Products of coal Value £	..	..	30,263	102,523
Earthenware ... "	..	..	159,639	129,977
Stoneware..... "	..	..	13,670	13,667
Glass:—				
Plate..... Sq. Ft.	178,269	167,472	7,754	7,929
Flint..... Cwt.	6,559	6,381	18,105	14,728
Bottles..... "	55,448	56,556	26,991	28,279
Other kinds.... "	16,687	16,339	14,067	13,116
Leather:—				
Unwrought .... "	12,971	10,656	12,1945	96,825
Wrought ..... Value £	..	..	22,447	18,295
Seed oil..... Tons	6,264	4,430	138,532	94,392
Flooreloth ..... Sq. Yds.	1,509,900	1,792,500	69,791	67,529
Painters' materials Val. £	..	..	139,945	120,688
Paper ..... Cwt.	82,882	75,176	134,181	112,749
Rags..... Tons	5,169	4,584	34,748	25,969
Soap..... Cwt.	57,041	49,454	27,238	52,970
Total value .....	..	..	2,670,888	2,337,341

EXPORTS OF METALS (OTHER THAN MACHINERY) FOR  
MONTH ENDING 30TH JUNE.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Brass..... Cwt.	12,383	10,084	45,690	35,895
Copper:—				
Unwrought ..... "	61,123	37,012	147,334	82,284
Wrought..... "	35,968	20,126	101,340	54,788
Mixed metal .... "	23,463	19,696	54,287	42,980
Hardware ..... Value £	..	..	176,574	148,428
Implements..... "	..	..	105,589	95,393
Iron and steel..... Tons	296,264	266,157	2,653,466	1,644,622
Lead ..... "	4,644	5,091	51,167	52,228
Plated wares... Value £	..	..	18,942	21,684
Telegraph wires ..	..	..	28,274	34,646
Tin ..... Cwt.	13,834	8,907	63,714	30,189
Zinc ..... "	13,658	21,699	11,249	14,211
Other articles .. Value £	..	..	66,833	56,683
Total value .....	..	..	2,924,951	2,620,433

EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING  
30TH JUNE.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Alkali..... Cwt.	451,371	409,703	151,307	119,243
Bleaching materials „	154,434	96,614	65,574	38,869
Chemical manures. Tons	27,905	33,895	169,444	223,368
Medicines..... Value £	..	..	77,565	82,069
Other articles ... „	..	..	285,754	271,826
<b>Total value .....</b>	..	..	<b>719,734</b>	<b>735,366</b>

## Monthly Patent List.

\* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

I.—GENERAL PLANT, APPARATUS, AND  
MACHINERY.

## APPLICATIONS.

11,960. G. W. Brown and W. R. Skinner. Improvements in apparatus for evaporating and condensing, and for heating feed-water for steam boilers. June 20.

11,991. W. Evans. See Class XVIII., B.

12,880. G. A. Barnard. Improvements in apparatus for cooling or condensing fluids. Complete Specification. July 3.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

12,941. L. Maiche and C. Maiche. Improvements relating to the purification of water and to apparatus therefor. June 27.

14,840. M. Julien. Apparatus for effecting the intimate mixture of liquids, semi-liquids, or liquefied matters. July 4.

14,900. E. C. Buik. Improvements in apparatus for absorbing gases. July 11.

15,724. J. Harper. An improved apparatus for assisting in the drying of hops. July 18.

16,151. P. J. Worsley, W. Windus, and B. Bracey. See Class VII.

24,266. H. S. Keating. An improvement in hydrometers, saccharometers, and such like instruments. July 11.

1894.

7235. D. McGill. An improved method of and means for the compression of air or other gases. July 11.

## II.—FUEL, GAS, AND LIGHT.

## APPLICATIONS.

11,801. J. Calderwood and J. Hodges. Improvements in treating paraffin scale. June 18.

11,810. J. H. Lamprey. Improvements in apparatus for the rapid and economical production of ozone. June 19.

12,048. W. H. Biggs and R. R. Greenhow. Improvements in the manufacture of artificial fuel from anthracite coal slack and similar substances. June 22.

12,663. E. Hermite, C. F. Cooper, and E. J. Paterson. An improvement in the purification of gas. June 29.

12,761. W. P. Thompson.—From E. Scott, United States. A new or improved combustible material and process of manufacturing the same. July 2.

12,888. J. C. Fell.—From M. M. Armstrong, United States. Method of smokeless combustion of fuel, &c. Complete Specification. July 3.

13,114. E. Daus. Improvements in the manufacture of heating and illuminating gas. July 6.

13,299. J. B. Taylor. Improvements in the manufacture of fuel. July 10.

13,385. B. H. Thwaite. An improved process for generating combustible gas and in apparatus therefor. July 11.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

12,355. W. Young. Improvements in the production of illuminating gas from mineral oils, and in apparatus therefor, and for producing water-gas. June 27.

12,389. W. B. Hartridge. Improvements in and relating to the manufacture of fuel. June 27.

13,126. W. Young. Improvements in the production of illuminating gas and by-products from liquid hydrocarbons, and in apparatus therefor. July 4.

13,163. H. M. Caldwell and J. E. Tatham. Improvements in and apparatus for distilling or cracking and gasifying hydrocarbon oils and the like. July 11.

14,545. J. Smith. Improvements in the production of illuminating gas. July 18.

15,432. R. H. Laird and W. H. Laird. Improved apparatus for vaporising oil. July 18.

1894.

8588. A. G. Glasgow. Improvements in water-gas apparatus. July 4.

III.—DESTRUCTIVE DISTILLATION, TAR  
PRODUCTS, ETC.

## COMPLETE SPECIFICATION ACCEPTED.

1893.

16,769. F. W. Clark. An improved method or process for the extraction of naphthalene and other hydrocarbons from coal-tar and similar substances. July 11.

## IV.—COLOURING MATTERS AND DYES.

## APPLICATIONS.

11,739. E. von Salis. Improvements in the manufacture of bases applicable for the production of substantive cotton dyes. June 18.

11,769. W. Krauth. A process for the production of 1-phenyl-3-methyl-5-pyrazolones. June 18.

12,145. I. Levinstein and Co., Lim., and I. Levinstein. Improvements in and connected with yellow colouring matters. June 23.

12,214. O. Imray.—From The Society of Chemical Industry in Basle, Switzerland. Manufacture of polyoxothionine colouring matters dyeing on mordants. Complete Specification. June 24.

12,621. A. Ashworth and J. Bürger. Improvements in the production of new colouring matters. June 29.

12,742. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of colouring matters. June 30.

13,156. C. D. Abel.—From A. Fischesser and Co., Germany. Manufacture of naphthylendiamine sulpho acids. July 6.

13,203. C. D. Abel.—From A. Fischesser and Co., Germany. Manufacture of a new 1, 8-amidonaphtholdisulpho acid from 1, 8-naphthylendiaminetrisulpho acid. July 7.

13,260. O. Imray.—From Goerlich and Wichmann, & Co., Germany. An improvement in the manufacture of yellow prussiates from the rhodanalkalis. July 9.

13,443. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Production of fast dyes on mordanted fibre. July 11.

13,577. J. V. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements relating to the manufacture of nitrosamines. July 13.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

14,207. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brünig. Improvements in the production of colouring matters from meta-amidophenol-phthaleines. June 27.

15,269. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of amidonaphtholsulpho acids and colouring matters therefrom. June 27.

15,404. G. Tobias. Preparation of a new  $\beta$ -naphthylaminesulphonic acid by means of the salts of  $\beta$ -naphthylamine and  $\beta$ -naphthylsulfamin acid. June 27.

16,199. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation. Manufacture of 2,7-amidonaphtholdisulpho acid and of colouring matters derived therefrom. July 18.

16,780. H. E. Newton.—From The Badische Anilin und Soda Fabrik. The manufacture or production of colouring matters and materials therefor. July 11.

17,141. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of sulpho-acids of the naphthalene series. July 18.

17,141A. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of sulpho-acids derived from  $\beta_1$ -naphthylamine,  $\beta_2\beta_4$ -disulpho acid. July 18.

17,141B. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of  $\alpha_1$ -naphthylamine  $\beta_1\alpha_2\beta_4$ -trisulpho-acid, and of other sulpho-acids derived therefrom. July 18.

17,141C. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of sulpho-acids derived from naphthalene  $\alpha_1\beta_2\alpha_4$  (or  $\alpha_2\beta_2\alpha_4$ ) trisulpho-acid or analogous sulpho-acids. July 18.

17,472. B. Willeox.—From The Badische Anilin und Soda Fabrik. The manufacture and production of new colouring matters and leuco compounds related to the indigo series, and of new materials therefor. July 11.

17,572. B. Willeox.—From The Badische Anilin und Soda Fabrik. Improvements in the manufacture of betamethyleurhodine. July 18.

18,958. A. Bang.—From G. A. Dahl. Processes for the production of dyestuffs. July 18.

1894

9806. J. C. L. Durand, D. E. Huguenin, and A. J. J. d'Andicon. Manufacture of new violet, blue, grey, and black substantive colouring matters for dyeing cotton. June 27.

#### V.—TEXTILES, COTTON, WOOL, SILK, Etc.

##### APPLICATIONS

11,754. W. E. Heys.—From G. Malard, France. Improvements in the process of and apparatus for scouring and degreasing wool. Complete Specification. June 18.

13,345. W. P. Thompson.—From P. C. D. Castle, Germany. See Class XIV.

##### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

16,170. W. Littlewood and I. Ingram. Improvements in the method and apparatus for colour-classing and designing for the manufacture of textile goods. June 27.

16,698. E. J. Goodwin. Improved treatment of textile fabrics and production of material suitable for use as tracing cloth. July 11.

#### VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

##### APPLICATIONS.

12,303. H. H. Lake.—From La Société Bonnet, Ramel, Savigny, Giraud, and Marnas, France. Improvements relating to the printing of fabrics. June 25.

12,600. H. Baines. A new or improved manufacture or production of a single or multi-coloured list or selvedge on piece-dyed cloths. June 28.

12,722. C. H. Smith. Improvements in bleaching calico, cloth, and other woven fabrics. June 30.

12,762. M. Kalisch, W. A. Rosenberg, and J. Entwistle. Improvements in the manufacture or production of cloths in variegated colours. July 2.

12,866. M. Cockroft. Improvements in apparatus for dyeing stockings, gloves, and other fabrics. July 3.

13,210. H. W. Sanderson. Improvements in and relating to the printing of textile fabrics. Complete Specification. July 7.

13,443. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. See Class IV. July 11.

##### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

13,579. J. Rhodes, jun., and A. B. Perkins. Improvements in apparatus for dyeing slubbing or slivers of fibrous material. July 11.

16,038. T. P. Miller. Improvements in apparatus for treating fibrous materials, yarns, or threads with mordanting, preparing, dyeing, or other liquids. July 11.

1894.

8964. J. W. Abom. Method of bleaching paper pulp or other flocky substances. July 4.

11,077. E. Hoffmann. Improvements relating to dyeing wool with chromium lakes. July 11.

## VII.—ACIDS, ALKALIS, AND SALTS.

## APPLICATIONS.

11,859. G. Bamberg. Improvements in the manufacture of silicate of soda and carbonate of soda, and in apparatus for that purpose. June 19.

11,966. J. Brock, V. C. Driffield, and F. W. Wright. Improvements in and apparatus for effecting the decomposition of gases by means of porous or catalytic material, more especially intended for use in the production of chlorine from hydrochloric acid gas and air. June 20.

11,967. J. Brock, V. C. Driffield, F. W. Wright, and T. Davies. Improvements in apparatus for effecting the decomposition of gases by means of porous or catalytic substance, more especially intended for use in the production of chlorine from hydrochloric acid gas and air. June 20.

11,968. J. Brock, V. C. Driffield, A. Carey, and G. Ogg. Improvements in apparatus relating to the manufacture of chlorine. June 20.

11,988. G. S. Albright and J. J. Hood. Improvements in the preparation of sulphur. June 21.

12,086. H. R. Angel. Improvements in the manufacture of bicarbonate of soda. June 22.

12,218. H. Y. Castner. Improvements in the manufacture of cyanides. June 23.

12,219. H. Y. Castner. Improvements in the manufacture of cyanides. June 23.

12,384. I. A. F. Bang and M. C. A. Ruffin. Manufacture of anhydrous stannic acid. June 26.

12,634. F. Hamer. The production of nitric acid,  $\text{HNO}_3$ , and the nitrates of soda, of potash, and of lime. June 29.

12,675. A. Staub. Improvements relating to the manufacture of sulphuric acid, and to apparatus therefor. June 29.

12,708. G. Dalton and T. B. Provis. See Class XVIII., C.

12,881. H. H. Lake.—From L. Sternberg, United States. Improvements in apparatus for obtaining ammonia and ammonia-salts from nitrogenous organic matters. Complete Specification. July 3.

13,199. C. Raspe. Improvements in the manufacture of soluble metallic phosphate compounds. July 7.

13,202. H. Wilcox. See Class XIII.

13,315. W. McD. Mackey and J. F. Hutcheson. Improvements in the manufacture of ammonia. July 10.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

12,927. A. Mitscherlich. Process for utilising the lyes of sulphite cellulose products obtained in such process, and apparatus employed for that purpose. June 27.

13,406. R. Haddan.—From Compagnie Electro-chimique de St. Beron. Improvements in or relating to the treatment of sodium chloride, and to the method of utilising the product in the manufacture of white lead, soda, chlorine, and their derivatives. July 18.

16,151. P. J. Worsley, W. Windus, and B. Bracey. Improvements in or connected with pumping and absorbing chlorine gas, and apparatus for use therein. July 4.

17,846. H. W. Crowther and E. C. Rossiter. Improvements in the manufacture of sulphonylides. July 18.

1894.

10,589. O. Imray.—From P. Gredt. Process for treating blast-furnace gases for the extraction of alkaline and ammonium compounds, iodides, and iodine therefrom. July 11.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

## APPLICATIONS.

12,056. R. M. Wight. Improvements in machinery or apparatus for use in the manufacture of sheet glass. June 22.

12,291. W. Hassall and H. W. Sansom. Improvements relating to fire-places for pottery and other like kilns. June 25.

12,789. J. T. Newell. An improved method of decorating glass, earthenware, and metallic substances. July 2.

13,017. P. Shelley. Process of decorating pottery, glass, or similar goods. Complete Specification. July 5.

13,290. H. B. Barlow.—From J. J. Power, United States. Apparatus for blowing and moulding in glass. July 19.

13,293. T. W. Twyford. Improvements in means for uniting or connecting together earthenware and other non-metallic articles, structures, and objects. July 10.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

## APPLICATIONS.

11,973. J. Stow. An improved method of preparing concrete. June 21.

12,233. A. J. B. Ward. Non-slippery pavements. Complete Specification. June 23.

12,350. W. Bruch. A new or improved hydraulic cement. June 26.

12,811. J. Pfeiffer. Improved process for the manufacture of artificial sandstone. July 2.

13,029. S. J. Beaman and J. Deas. Improvements in furnaces for burning sludge, slurry, and like materials. July 5.

13,128. J. H. Lester. Improvements in treating certain waste materials to produce valuable products, such as plaster or stiffening, weighting, and whitening materials therefrom. July 7.

13,607. R. Y. Ardagh. Improvements in the manufacture of wooden blocks for paving and flooring. July 14.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

17,322. A. McLean and C. W. Wilson. Improvements relating to the manufacture of artificial stone. July 18.

1894.

9958. H. Manrer. An improved process of manufacturing porous bricks. July 4.

## X.—METALLURGY, MINING, Etc.

## APPLICATIONS.

11,797. E. D. Kendall. Improvement in the method or process of treating gold and silver ores, and a composition of matter for the same purpose. Complete Specification. June 18.

12,171. W. A. Jenkin. A method for the removal of arsenic, antimony, and bismuth from copper liquors by means of an iron salt. June 23.

12,341. H. T. Barnett. Improvements in and connected with the distillation of zinc. June 26.

- 12,582. O. Klatte. New or improved process for producing rolled bars. Complete Specification. June 28.
- 12,879. E. W. McKenna. Improved process for renewing old steel rails. Complete Specification. July 3.
- 13,038. J. Lakin and C. H. Page. Improvements in the manufacture of wrought iron. July 5.
- 13,048. J. Bedford. Improvements in the manufacture of steel. July 5.
- 13,184. T. Hampton. Improvements in the manufacture of steel. July 7.
- 13,262. A. V. Newton.—From A. Nobel, France. Improvements in welding and soldering metals. July 9.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

- 13,227. C. J. Bagley, L. Roberts, and the Moor Steel and Iron Co., Ltd. Improvements in the means employed for reheating slabs of steel or iron. July 11.
- 15,152. E. C. Broadwell. Improvements in processes of coating metals with aluminium or its diluent alloys. June 27.
- 15,569. L. Maddison. Improvements in the manufacture of iron and steel articles. June 27.
- 15,875. C. Allen and C. Davey. Improvements in apparatus for the manufacture of cast iron and steel. July 4.
- 16,421. H. Schneider. Improvements in the manufacture of armour plates and the like, and apparatus therefor. June 27.
- 16,561. C. Moldenhauer. A process for precipitating precious metals from solutions. July 11.
- 23,619. E. R. Besemfelder. Improvements in the method of separating metals from ores, and from other mixtures or insoluble substances therewith. July 11.
- 24,900. E. Weinberg, R. Carriek, and J. G. Johanson. Improvements in the elimination of the oxides of zinc, iron, and copper from cobalt-oxide, and apparatus therefor. July 18.

1894.

1472. A. H. Bell and C. Carter. An improved apparatus for precipitating gold and silver from cyanide solutions. June 27.
8087. A. A. Ackerman. An improved process for the manufacture of hard-face armour plate. July 18.
- 10,923. W. P. Thompson.—From E. Bertrand and O. Thiel. Improvements in the manufacture of steel. July 11.
- 11,148. W. P. Thompson.—From O. Nicolai. An improved flux for use in soldering aluminium and other metals. July 18.
- 11,574. H. R. Müller and F. O. Müller. A means or apparatus for producing pure castings. July 18.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

## APPLICATIONS.

- 12,045. A. Schanschieff. Improvements in secondary batteries. June 21.
- 12,483. C. P. Shrewsbury, F. L. Marshall, J. Cooper, and J. L. Dobell. Improvements in or connected with electric batteries. June 27.
- 12,650. B. M. Drake and J. M. Gorham. Improvements in storage battery plates. June 29.
- 12,993. A. Cruickshank. Improvements in the electrolytic separation of gold from ores. July 4.

- 13,358. H. H. Lake.—From I. I. Roberts, United States. Improvements relating to electrolysis and to apparatus therefor. Complete Specification. July 10.

- 13,363. W. A. R. Buckland. An improved plate for secondary voltaic batteries. Complete Specification. July 10.

- 13,377. P. Jensen.—From H. C. I. Størmer, Norway. Improvements in processes and apparatus for electrolytically treating salts in solution or in molten state. Complete Specification. July 10.

- 13,499. A. Sinding Larsen. Improvements in process and apparatus for electrolytical decomposition of alkali salts. Complete Specification. Filed July 12. Date applied for, December 12, 1893, being date of application in Norway.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

- 14,056. W. H. Longsdorf. Improvements in electrolytes for primary batteries. July 18.
- 15,355. H. S. Pyne and R. Thompson. An improved electric battery. July 4.
- 15,610. F. G. Bailly and M. Guthrie. Improvements in apparatus for the electrolytical decomposition of brine and other liquids. June 27.
- 16,851. A. Gentzsch. Improved insulating material for electrical purposes and method of producing same. July 11.

1894.

3674. W. H. Longsdorf.—From G. Hewett. Improvements in primary galvanic batteries and in the liquids used therein. July 11.
8061. H. Carnichael. Improved apparatus and process for electro-chemical decompositions. July 18.
9761. T. Craney. Improvements in electrolytic apparatus. June 27.
9949. T. Craney. Improvements in electrolytic apparatus. June 27.
- 10,169. G. R. Blot. Improvements in plates for secondary voltaic batteries. July 4.
- 10,853. J. Y. Johnson.—From La Société L'Accumulateur Fulmen. Improvements in electrical accumulators or storage batteries. July 11.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

## APPLICATIONS.

- 11,832. J. McDonald.—From E. J. Pearson, Australia. Improvements in the manufacture of soap. June 19.
- 11,885. H. Perl. Improvements in and connected with soap pieces. Complete Specification. June 19.
- 12,072. J. P. F. Field. Improvements in and relating to the manufacture of soap for laundry and other purposes. June 22.
- 12,367. J. van Raymbeke and W. F. Jobbins. Process of and apparatus for obtaining glycerine and other products from soapmakers' spent lyes. Complete Specification. June 26.
- 12,782. W. M. Walters. Improvements in the manufacture of soaps. July 2.
- 12,836. C. N. Wilson. Improvements in the method of and apparatus for extracting and refining wool-fat from spent wool-scouring liquor. July 3.
- 13,104. F. Heilbron. Improvements in or applicable to soap in tablets, cakes, or bars. Complete Specification. July 6.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

16,987. W. Spindler and C. Stautz. Improvements in extracting oil from oil-containing plants and fruits, and in the production of fodder from the residual matter. July 18.

1894.

9179. A. Wendtland. An improved process for removing the green colour from paraffin, vaseline, and lubricating oils, which have had that colour imparted to them by bodies containing oxygen. July 4.

### XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

## APPLICATIONS.

12,299. A. J. Protheroe. A new manufacture of paint. June 25.

12,595. W. B. Priest. Improvements in the manufacture of white lead. June 28.

12,872. F. Evers. Improvements in or relating to rust preventive compositions. July 3.

13,004. J. Clasen. A new or improved binding medium for size colours and method of manufacturing the same. Complete Specification. July 4.

13,145. L. E. Josset. Improvements in and in connection with luminous paint. July 6.

13,198. J. H. Lester. See Class IX.

13,202. H. Wilcox. Improvements in or relating to the manufacture of white lead and nitrate of ammonium. July 7.

## COMPLETE SPECIFICATION ACCEPTED.

1894.

10,424. P. F. Johnson. Improvements in the manufacture of plumbago or graphite, commonly called black lead. July 4.

### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

## APPLICATIONS.

12,911. P. C. Hewitt. Improvements in glue-making machinery. July 3.

12,914. R. Haddan.—From E. Avellis, Germany. Improvements relating to the colouring of greased leather. July 3.

13,195. T. H. L. Bake and H. A. Leverett. Improvements in tanning skins and hides. Complete Specification. July 7.

13,345. W. P. Thompson.—From P. C. D. Castle, Germany. Improvements in the manufacture of gum or size applicable for use with cotton, wool, silk, linen, or other textile fabrics. July 10.

13,369. P. C. Hewitt. Improvements in clarifying or removing impurities from glue and other substances. July 10.

13,414. A. Herrmann. An improved process for colouring tanned hides by means of smoke. Complete Specification. July 11.

13,596. J. P. O'Donnell.—From J. A. Schweitzer, France. Improvements in the manufacture of artificial whalebone from parchmentised skin. July 14.

### XV.—AGRICULTURE AND MANURES.

## COMPLETE SPECIFICATION ACCEPTED.

1894.

11,297. G. H. Richards. A new or improved apparatus for vaporising compounds or compositions for use in horticulture. July 18.

### XVI.—SUGARS, STARCHES, GUMS, Etc.

## APPLICATIONS.

11,871. J. Wetter.—From J. D. Riedel and Co., Germany. Improvements in and connected with sweetstuffs or sweetening materials. June 19.

12,071. W. H. Percy. Improvements in sugar boiling and in apparatus for use in connection therewith. June 22.

12,408. A. M. Clark.—From La Société Gallet, Gibou, et Cie., France. Process for the purification of caramel produced from sugar, molasses, glucoses, and other saccharine matters generally. June 26.

13,155. O. Imray.—From R. Folsche, Germany. Process and apparatus for the systematic washing of impure sugar. July 6.

## COMPLETE SPECIFICATIONS ACCEPTED.

1894.

8463. B. Lach and H. Benies. Process for purifying and decolourising saccharine juices and solutions. July 4.

9436. F. Hübner. Improvements in the manufacture of white sugar and the like from molasses and in centrifugal apparatus for use therein. June 27.

9441. C. D. Abel.—From L. König.—A process for deadening and bleaching dissolved or fused amylaceous matters. June 27.

### XVII.—BREWING, WINES, SPIRITS, Etc.

## APPLICATIONS.

12,196. A. F. Spencer. Improved preparations to be used in brewing and in the treatment of finished beer. June 23.

12,295. P. C. Vivien. Improvements in or relating to apparatus for distilling, rectifying, and condensing alcoholic and other vapours. June 25.

13,153. E. Hermite, E. J. Paterson, and C. F. Cooper. An improvement in the purification of alcohol, spirits, and the like. July 6.

13,364. C. Rach. Apparatus for the preparation of wort. July 10.

13,492. R. Zdarek. Improvements in the manufacture of alcohol. July 12.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

14,373. R. E. Evans. Improvements in syrups, extracts, wines, beers, mineral waters, and other liquids. June 27.

16,692. F. H. Collins. Improvements in apparatus for regulating the temperature of fermenting liquids. July 18.

1894.

10,043. J. T. Connell. Improvements in the treatment of hops. June 27.

# XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

## APPLICATIONS.

### A.—Chemistry of Foods.

12,690. H. Wilson. Improvements in apparatus for the preservation of perishable articles of food and the purification of the air in closed-up places. June 30.

13,003. H. H. Lake.—From F. Delangle, France. Improved food preparations. July 1.

13,031. H. L. C. Paulsen. Improvements in and relating to the preservation of food. Complete Specification. July 5.

13,587. T. H. L. Bake and H. A. Leverett. Improvements in apparatus for preserving animal and vegetable food and other substances. July 13.

### B.—Sanitary Chemistry.

11,991. W. Evans. Improvements in apparatus for cleansing and filtering sewage and impure liquids, and for separating solids or precipitated matters from liquids generally. June 21.

12,893. H. E. Newton.—From The Pinhoe Patent Refuse Destructor Co., Australia. Improvements in apparatus for destroying garbage and house refuse. July 3.

12,903. Count Heinrich von Pückler. Improved means for purifying and disinfecting air and for consuming deleterious gases and vapours. Complete Specification. July 3.

13,171. F. W. Lacey. Improvements in apparatus for the treatment of towns' refuse. July 7.

### C.—Disinfectants.

12,176. J. R. Carruthers. Improvements in preparing and packing arsenic or other materials for use in dipping animals, destroying weeds, and for like purposes. June 23.

12,708. G. Dalton and T. B. Provis. A method of utilising certain by-products of iron and copper sulphides, and other iron and copper ores for the production of persulphate of iron to be used as a disinfectant for the purification of sewage and for other purposes. June 30.

13,067. A. Blackie and E. A. White. Improved preparations for dipping sheep or other animals, for destroying erms or injurious growths and parasites on plants, and for analogous purposes. July 5.

13,092. F. M. Spence. Improvements in disinfecting powders. July 6.

## COMPLETE SPECIFICATIONS ACCEPTED.

### A.—Chemistry of Foods.

1893.

15,169. H. L. Doulton and A. W. Manger. Improvements in and relating to the softening and purifying of water and apparatus therefor. July 11.

15,434. H. B. Briggs. Improvement in the manufacture of condensed-milk preparations. July 11.

15,435. H. B. Briggs. Improvement in the manufacture of condensed-milk preparations, invalids' and children's foods. July 11.

### B.—Sanitary Chemistry.

1893.

10,458. A. P. Hope. Improvements in the treatment of sewage or analogous foul waters or matters for effecting the disinfection and purification thereof. July 4.

13,305. R. C. Tanner. Improvements in the treatment of sewage sludge. July 4.

14,586. A. McDougall. Improvements in covering lead surfaces to protect water and other potable liquids from contamination with lead dissolved in contact therewith. July 18.

15,914. S. Quilliam. New or improved sanitary material. July 4.

16,368. J. Hanson. Improvements in and apparatus for treating town refuse, or excrementitious or like matter, and obtaining useful products therefrom. July 18.

16,522. G. Jarmain. Improvements in the treatment of sewage and waste waters. July 11.

16,948. J. Bunnehr and W. Chataway. Improvements in the ventilation of sewers, and in treating the foul gases generated therein, and apparatus therefor. July 18.

1894.

5792. R. C. Sayer. Improvements in dry closets in which heat is employed to desiccate the soil. July 11.

### C.—Disinfectants.

1893.

15,914. S. Quilliam. *See* Class XVIII., B.

1894.

10,188. A. J. Bault.—From F. Haise. Improvements in insecticides. July 1.

## XIX.—PAPER, PASTEBOARD, Etc.

### APPLICATIONS.

12,978. R. Münch. Improvements in the manufacture of aluminised paper. July 4.

12,981. C. A. P. F. Tschoben. Improvements in rolls or coils of perforated paper. July 4.

13,139. S. Hahn. A process of manufacture of films imitating mother-of-pearl. Complete Specification. July 6.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

12,927. A. Mitscherlich. *See* Class VII.

16,947. A. Yockney. Improvements in the manufacture or treatment of paper. July 18.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

### APPLICATIONS.

11,774. O. Imray. — From The Farbwerke vermal's Meister, Lucius, and Brüning, Germany. Manufacture of mandelate of antipyrine. June 18.

11,952. C. D. Abel.—From Haarmann and Reimer, Germany. Process for the manufacture of vanilline from eugenol and isoeugenol respectively. June 20.

12,174. The Peptine Maltine Co., Lim. Peptonised extract of malted and other cereals. June 23.

12,504. A. Hommel. An improved process for the production of purified, sterilised, liquid hæmoglobin. June 27.

12,796. F. W. Fletcher. Improvements in cinchona preparations. Complete Specification. July 2.

12,980. C. D. Abel.—From The Fabriques de Prodnits Chimiques de Thaun et Mulhouse, Germany. Improvements in the manufacture of artificial musk. July 4.



12,987. H. E. Newton. — From the Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of chemical compounds containing iodine. July 4.

COMPLETE SPECIFICATIONS ACCEPTED.

1893.

16,708. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of chemical compounds. July 11.

16,949. O. Inray. — From The Farbwerke vormals Meister, Lucius, and Böhling. Manufacture of caffeine-sulphonates. July 11.

21,853. J. H. Baum. Improvements relating to the production of pyro catechine. July 18.

1894.

5675. C. D. Abel. — From C. Pieper. Improvements in the production of dextrins and leucocenes. June 27.

XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.

APPLICATIONS.

11,872. R. J. Crowley. A process of obtaining chemical copies of paintings, pictures, engravings, and prints. June 19.

12,269. A. B. Chatwood. Improvements in photography. June 25.

12,269A. A. B. Chatwood. Improvements in instruments for and methods of measuring the actinic intensity of light for photographic and other purposes. Filed. July 12. Date claimed 25 June 1894.

13,193. H. A. Fardell. A method of producing photographs in colours. July 7.

13,196. J. Joly. Improvements in or relating to photographing in colours. July 7.

COMPLETE SPECIFICATION ACCEPTED.

1893.

18,237. E. Edwards. — From R. Reissner and G. C. F. Hauser. Improvements in the process of making photographic films. July 18.

XXII.—EXPLOSIVES, MATCHES, Etc.

APPLICATIONS.

12,084. D. Hiekie and G. F. Bentner. Improvements in explosives. June 22.

12,649. D. Hiekie and G. F. Bentner. Improvements in detonators. June 29.

12,716. E. G. Nott. Improvements in or relating to lucifer matches. June 30.

13,256. T. Hawkins, S. H. Hawkins, and H. Hawkins. Improvements in explosives. July 9.

13,276. H. Priester. Improvements in igniting compositions for safety matches. July 9.

13,506. H. H. Lake. — From The Dynamit-Actien-Gesellschaft vormals A. Nobel and Co., Germany. Improvements relating to the manufacture of explosives. July 12.

# THE JOURNAL

OF THE

# Society of Chemical Industry:

## A MONTHLY RECORD

FOR ALL INTERESTED IN CHEMICAL MANUFACTURES.

No. 8.—VOL. XIII.]

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## NOTICES.

Notice is hereby given that, in accordance with the alterations in Rule 27 sanctioned at the Annual General Meeting in Edinburgh, all new members elected after January 1st next, and also those elected after the date of this notice who do not pay subscription for 1894, will be required to pay an entrance fee of one guinea. In accordance with the same rule the Life Composition Fee will be 20*l.*, instead of 15*l.* as heretofore after the 1st January next.

## BANKERS' ORDERS.

For the convenience of Members, the Treasurer has arranged with the Bankers of the Society that they shall collect subscriptions from Bankers in town and country; and Members, who have not already done so, are invited to fill up and sign the Banker's Order enclosed with the December number of the Journal, which should then be sent to the Honorary Treasurer, Mr. E. Rider Cook, East London Soap Works, Bow, E.

Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 41 of the bye-laws, the Society has the right of priority of publication for three months of all such papers. Infringement of this bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. EYRE and SCOTTISWOODE, the Society's printers and publishers, to whom all communications respecting them should be addressed.

Notice is hereby given that the next List of Members will contain the professions or business occupations of the Members of the Society. Any Member who desires to confirm or alter his description as given before election should communicate with the General Secretary.

## CHANGES OF ADDRESS.

Benjamin, Dr. M., 1/o Bond Street; 589, West End Avenue, New York City, U.S.A.

Blackwell, G. G., 1/o Irwell Chambers West; The Albany, Oldhall Street, Liverpool.

Cameron, Jas., 1/o Polmont; c/o the British Explosives Syndicate, Limited, Pitsea, Essex.

Dukes, T. Wm., 1/o Cape Town; c/o P. T. Petersen and Co., P.O. Box 210, Johannesburg, S.A.R.

Dyson, S.; Journals to c/o F. Dyson, 2, Exchange Place, Middlesbro'.

Emmens, S. H., 1/o San Francisco; Amador City, Cal., U.S.A.

Gray, Wm., 1/o Hall; Cliff House, Barton-on-Humber.

Hall, Allan T.; Journals to c/o Sissons Brothers and Co., Limited, Hull.

Higgins, E. Longuet, 1/o Falkner Square; c/o United Alkali Co., Limited, Muspratt's Works, Widnes; and (Journals) 79, Bedford Street South, Liverpool.

Hodgson, Wm., 1/o Victoria Buildings; 3, Barton House, Manchester.

Ivatts, H. E., 1/o Forest Gate; 13, Harrow Road, East Ham, E.

Jarvis, Talbot, 1/o Tonbridge; Castle Lodge, Bedford.  
Mackenzie, T. E., 1/o Motherwell; 4, Ethel Terrace, Mount Florida, Glasgow.

Philip, Arnold, 1/o Richmond; 4, Salisbury Villas, Cleveland Road, Barnes, Surrey.

Procter, C. (of Somerset House); Journals to 359, Upland Road, Dulwich, S.E.

Rawlins, T. Beilby, 1/o Pollokshields; Strathclyde Works, Kinning Park, Glasgow.

Tilden, Prof. W. A., F.R.S., 1/o Birmingham; 9, Lad-broke Gardens, Notting Hill, W.

Watmough, B., 1/o Leeds; c/o Brotherton and Co., Calder Vale Chemical Works, Wakefield.

White, H., 1/o Rotherham; 43, Woodstock Road, Sheffield.

White, J. Walwyn, 1/o Halebank; Public Hall, Widnes.

Whitehead, Jas., 1/o Rochdale; 1, Parkin Lane, Meltham, Huddersfield.

Woodham, K. G., 1/o Withington; 5, Lethbridge Road, Southport.

Wyld, Jno., 1/o Frizinghall; Neshells Park Chemical Works, Birmingham.

## CHANGES OF ADDRESS REQUIRED.

Carswell, T. R., 1/o Day House, Day Street, Newcastle-on-Tyne.

Chadwick, L. N., 1/o Ivy Lawn, Ponders End, Middlesex.

## ERRATUM.

(June No., 1894, p. 573.)

The changes of address of Messrs. Aldrick, Brotherton, Hopkins, Horrocks, Ray, Robinson, and Sexton, were inadvertently inserted among the names of members elected on June 22nd.

## London Section.

THE CHEMICAL SOCIETY'S ROOMS, BURLINGTON HOUSE, W.

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## Obituaries.

C. R. ALDER WRIGHT, D.Sc. (Lond.), B.Sc. (Vict.), F.R.S.

MEMBER OF COMMITTEE OF THE LONDON SECTION OF THE SOCIETY OF CHEMICAL INDUSTRY.

CHARLES ROMLEY ALDER WRIGHT was born in 1844 at Southend, and was the youngest child of Romley Wright, a civil engineer of Scotch extraction, on the ordnance survey, who was a man of great intellectual power, and singular individuality of views. He himself undertook the education of his son, and even taught himself Greek that he might impart a knowledge of that language to his boy. Thus it came about that the preliminary training of Alder Wright, whilst conducted upon a broad literary basis, was distinctly designed as that of an engineer. But the subject of this memoir was otherwise inclined, for he soon and unmistakably manifested an enthusiasm for experimental chemistry. Little wonder that on passing from home to Owens' College (1861), inheriting as he did the intellectual force and individuality of his father, together with a faculty for hard work,—he should excel with ease in everything he took in hand. Charles Romley Wright, as he was better known in the college days, was not only a well-known and distinguished figure in the chemistry classes and laboratory under Roscoe, but almost equally so on the physical, mathematical, and classical sides of the college. He even distinguished himself in the Greek Testament and Hebrew Bible classes.

In those early days of the older Owens' College, the College Union was started, and though usually taking little part in debates, none of his old fellow-students who heard it will forget a remarkable lecture and its remarkable illustrations and argument, partly read and partly delivered by him, on phrenology and mesmerism. The first piece of original research prosecuted by Wright, took place in the laboratory of Roscoe, on the *Chemical Action of Sunlight* (1866). This research completed, Wright whilst awaiting the advent of a suitable post, occupied himself at home with scientific work and in the construction of a chemical balance. Every part of this instrument he constructed himself, grinding his own knife edges, &c. The balance still remains and works to perfection. It is an example of patience, mechanical ingenuity, and skill. Just as it was completed, the post of chemist at the Weston works of the Runcorn Soap and Alkali Co., Limited, became vacant through the retirement of Mr. J. W. Kynaston, and Wright was elected to fill it (1866). Here he quickly developed as a chemical engineer as well as an investigator, notwithstanding the very considerable load of physical pain he had now frequently to bear, in consequence of a weakness of the hipjoint, which was gradually giving evidence of more serious trouble.

As the result of the energetic work referred to, in this short year in a chemical factory, several papers of considerable technical importance eventually appeared in the *Journal of the Chemical Society* (1867) and the *Chemical News* (1867). The first was on the Practical Loss of Soda in the Alkali Manufacture, the others were a series of articles on "Losses in various branches of the Alkali Manufacture and Allied Matters." Wright now left Runcorn for London, and became assistant in the laboratory of Dr. Bernays at St. Thomas' Hospital. During that brief period his energies were chiefly engaged in a series of letters and communications to the *Chemical News* on methods then in vogue for testing alkalis, and kindred subjects. Later, in conjunction with Matthiessen (1868-9), as assistant in his laboratory at the St. Mary's Hospital Medical School, a series of four papers was contributed to the *Royal*

*Society*, on "The Derivatives of Opian Alkaloids." About this period too (1869-71) he became associated with Sir Lowthian Bell, F.R.S., with whom he served as research chemist in the laboratory of his ironworks, and here Wright carried out the elaborate investigations on the chemistry of iron smelting, which form the basis of the great work eventually published by Sir Lowthian Bell. From 1871 to the time of his death, Wright was occupied as lecturer in Chemistry in St. Mary's Hospital Medical School, and during this period the following important researches were prosecuted and published by him. Nine papers on Derivatives of Morphine and Codeine, to the Royal Society (1871-74); six papers on Isomeric Terpenes, Brit. Assoc. Reps. (1873-77). Six papers on the Action of Organic Acids and their Anhydrides on Natural Alkaloids, to the Chem. Soc. (1874-80). Seven papers on Narecine, Narcotine, Cotarine and Hydro-cotarine, to the Chem. Soc. (1874-77). Four memoirs on Alkaloids of the Aconites, to the Brit. Assoc. (1877-79). Three memoirs on Alkaloids of the Veratrum, to the Brit. Assoc. (1878-79). Citric Acid in Mulberries, to the Chem. Soc. (1878). Sweet Principle of *Sambucus Eboracica*, to the Chem. Soc. (1881). During this same period an important series of papers was contributed on Inorganic Chemistry and Physical and Electro-Chemistry, among which may be recorded the "Specific Heat of Hydrogenium," (jointly with Prof. Roberts Austen, F.R.S.) (1873); "Chemical Changes accompanying Iron Smelting" (1874); Relations between Affinity and Structural Formulae (1874); series of nine memoirs on the Determination of Chemical Affinity in Terms of Electro-Motive Force to the Phil. Mag. and Proc. of Phys. Soc. (1877-85); four Reports to the Chem. Soc. on Researches on some Points in Chemical Dynamics (1878-80). Amongst a mass of work of technological interest, the following items may be cited: "Pyrites as a Source of Copper, Iron and Sulphur," to Soc. of Arts (1874); series of articles in "*Muspratt's Dictionary*" on Coal Tar Distillation and Products, &c. (1874-75); another series on "Soap, Sulphur, and Sulphuric Acid," in Thorpe's Dictionary (1893); four lectures on "Metals and their Industrial Applications," delivered at the Royal Inst. (1878); articles in "*Encyclopædia Britannica*" on Iron, Steel, &c. (1879-81); Cupro-ammonium Solution and its Use in Waterproofing Paper, &c., to Soc. of Arts (1884); three Cantor lectures on "Manufacture of Toilet Soaps" to Soc. of Arts (1885); "Chemistry of Soap;" "Fluid Specific Gravity Determination for Practical Purposes;" "Certain Aluminium Alloys" (see this Journal 1885 and 1892); nine memoirs on "Certain Ternary Alloys," to Royal Soc. (1889-1894). Of his books, "*The Threshold of Science*," a book for boys and general readers, a long life may be predicted; and a very useful one of the work on "*Oils, Fats, and Waxes*." Even now a considerable number of Wright's investigations have been omitted from reference here.

Dr. Wright was an original member of the Physical Society and of the Institute of Chemistry. He had also been a member of the Council of the Chemical Society.

For some time past Dr. C. R. A. Wright, who all his life had known more or less of pain and infirmity, had become subject to the gradual development of *diabetes mellitus*. More serious tendency had been successfully resisted up to the time of his mother's death, about a year ago. This bereavement, however, proved a terrible trial to him, and it is thought that he had shown signs of failing health from that date. No serious consequences were however anticipated until Monday the 23rd of July. On Wednesday morning, the 25th, he became comatose, and passed quietly away early in the afternoon of that day.—W. S.

## DR. HERMANN JULIUS GRÜNEBERG.

PRESIDENT OF THE COLOGNE SECTION OF THE VEREIN DEUTSCHER INGENIEURE. MEMBER OF COUNCIL DES VEREINS ZUR WAHRUNG DER INTERESSEN DER CHEM. INDUSTRIE DEUTSCHLANDS, COMMERZRIETH DER STAAT KÖLN, &c.

IN the death of Dr. Hermann J. Grüneberg, on June 1st, German chemical industry has lost one of its most distinguished representatives, and in future discussions of the Society for the Protection of the Interests of German Chemical Industry, reported so fully in their organ, "*Die Chemische Industrie*," Dr. Grüneberg will now be greatly missed, and yet more the great knowledge and wise counsel invariably associated with his name.

Hermann Julius Grüneberg was born on the 11th of April 1827, at Stettin, and was the son of Aug. Wilhelm Grüneberg, an organ-builder in that city. He soon manifested a love for natural science, and was, after leaving school, placed under the care of Dr. Ritter, a noted pharmacist, of Stettin, and thus we have another prominent example of the way in which the German Apotheke has so often proved to be the nursery of scientific greatness in the Fatherland. Later on, H. J. Grüneberg entered the works of the chemist, G. Garbe, as a volunteer. He then went to Hamburg, and again entered an Apotheke and worked there for  $1\frac{1}{2}$  years, engaged in the making of all kinds of preparations. After doing military service, by entering the Military Apotheke in Stettin, he commenced to make a definite manufacturing study of white lead, and, only at the age of 23 he started, with some friends, a small factory. This venture did not succeed, and having now lost his patrimony, he accepted the invitation of Ross of Gothenberg to go and assist him in his white lead works in Sweden. He returned in 1854 to Stettin, to join his brother-in-law, Herr Klee, in founding another white lead works. Meantime the Crimean war had broken out, and a vigorous demand for saltpetre arose. This led Grüneberg to devise a process for making artificial potassium nitrate by acting upon sodium nitrate with carbonate of potash, and this process was started in the works originally designed for white lead making. The demand for saltpetre still increased, and a second works was started by Klee and Grüneberg at Bredow, near Stettin, and thereafter a third at Altdamm. The Crimean war over, this abnormally vigorous trade in saltpetre fell. But Grüneberg now possessed means, and thus assisted, he determined to complete his studies, and whilst doing so in Berlin, he kept well in touch with those who conducted the white lead and saltpetre factory, and took occasion to consult his professors, Mitscherlich and Rose, on the theory of the processes, and sought all possibilities of improving them.

In a subsequent treatise on white lead, Mitscherlich spoke most highly of the product of Grüneberg's firm. The great demand for saltpetre being over, Grüneberg proposed a new scheme for Klee's factory, viz., the use of the lixiviate of the ashes of beetroots in the production of ferrocyanide of potassium, &c., and in consultation with Heinrich Rose he managed greatly to benefit his brother-in-law's firm, and also himself to prove an excellent student. Later on he sojourned in Paris, and heard the lectures of Boussingault, Payen, Regnault, Dumas, Frémy, and others. In 1858, Grüneberg visited England and Scotland, and saw some of the factories here, returning to Germany later on, rich in knowledge and experience. In journeying through Cologne he now met for the first time his future partner, Julius Vorster. Later on the two determined to start a saltpetre factory in one of the Rhine provinces, and seeing that an old iron foundry was for sale at Kalk, by Cologne, they purchased it, and in 1858 there was founded the chemical works under the firm, afterwards so well and widely known, of "Vorster and Grüneberg." In 1860 Grüneberg actually found time to go to Leipzig and take his Ph.D. degree, and the same year he was married. With

such a life of unremitting work, study, and experience behind him, no wonder he carried his firm forwards, and steadily advanced to prosperity; and being a man of great enterprise and wisdom, it could be no matter of surprise either, that his counsel was sought in and around the city of Cologne, which he had now made his home. But besides the untiring energy thus manifested, and which eventually proved too much for physical endurance, Dr. Grüneberg possessed an eminently kind and benevolent heart, and so it happens that the news of his death has brought sorrow not only to those within the boundaries of the Fatherland, but to many beyond those boundaries. In this country the name of Grüneberg is chiefly and most widely associated with that masterpiece of technical skill, his continuous ammonia still, which, further improved by Simon, of Manchester, is known as the Simon-Grüneberg still; and the writer can testify to the exceeding kindness with which Dr. Grüneberg, in 1886, received him in Cologne, and showed and explained to him the construction and working of this apparatus. Of this truly great and good man and citizen, the *Kölnischer Zeitung*, of July 4th, thus expresses itself:—"Dr. Hermann Grüneberg hat zu den seltenen Männern gehört, die keinen persönlichen Feind haben, weil alle ohne Unterschied der politischen und confessionellen Richtung sich vor einer Persönlichkeit beugen, die ihre glänzenden Gaben, ihr umfassendes Wissen, ihren grossen Reichtum mit einer nie versiegenden, alles erwärmenden Bescheidenheit und Liebenswürdigkeit in den Dienst des Gesamtwohls ihrer Mitbürger stellt. Sein Leben und Wirken wird in Köln nie vergessen werden.—W. S."

## Journal and Patent\* Literature.

Class.	Page
I.—General Plant, Apparatus, and Machinery .....	787
II.—Fuel, Gas, and Light .....	789
III.—Destructive Distillation, Tar Products, &c. ....	791
IV.—Colouring Matters and Dyes .....	797
V.—Textiles: Cotton, Wool, Silk, &c. ....	802
VI.—Dyeing, Calico Printing, Paper Staining, and Bleaching .....	803
VII.—Acids, Alkalis, and Salts .....	808
VIII.—Glass, Pottery, and Enamels .....	811
IX.—Building Materials, Clays, Mortars and Cements..	811
X.—Metallurgy .....	812
XI.—Electro-Chemistry and Electro-Metallurgy .....	816
XII.—Fats, Oils, and Soap Manufacture .....	819
XIII.—Pigments and Paints; Resins, Varnishes, &c.; India-Rubber, &c. ....	821
XIV.—Tanning, Leather, Glue, and Size .....	821
XV.—Manures, &c. ....	822
XVI.—Sugar, Starch, Gum, &c. ....	822
XVII.—Brewing, Wines, Spirits, &c. ....	824
XVIII.—Chemistry of Foods; Sanitary Chemistry and Water Purification; Disinfectants .....	827
XIX.—Paper, Pasteboard, &c. ....	833
XX.—Fine Chemicals, Alkaloids, Essences, and Extracts ..	834
XXI.—Photographic Materials and Processes .....	—
XXII.—Explosives, Matches, &c. ....	837
XXIII.—Analytical Chemistry .....	837

\* Any of these specifications may be obtained by post by remitting *sd.*—the price now fixed for all specifications, postage included—to Sir Henry Reader Lack, Comptroller of the Patent Office, Southampton Buildings Chancery Lane, London, W.C.

## I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

*Artificial Refrigeration from Central Stations.* D. Branson. J. Franklin Inst. **137**, 1894, 81—93.

SINCE artificial refrigeration became a practical success, many have endeavoured to devise a method of supplying the means of effecting it from a central station, and some 20 or more costly attempts have been made in various towns, including London, Paris, New York, Boston, Louisville, and Nashville. They have all, more or less, proved failures. Messrs. Starr, Thornburgh, and Branson took up the matter some five years ago, and, after careful experiments, devised a system which has been put into successful operation at Denver and St. Louis, in the United States.

The Denver plant has now been working for four years uninterruptedly, although the plant has been twice enlarged and the distributing mains extended. The St. Louis plant has been in successful operation for three years. Until the introduction of a central-station system, only those who had to effect refrigeration on a large scale could afford to install the necessary plant, and thus dispense with the use of ice as a cooling agent. The delivery of the ice, even to large consumers, is one of the main items of cost, whilst to small consumers it is by far the largest item.

The system of refrigeration adopted by the author and his colleagues is that known as the "direct-expansion system," in which liquid anhydrous ammonia under pressure is admitted into a coil, known as the "expansion-coil," where it expands and becomes gaseous. The gaseous anhydrous ammonia is then absorbed by weak *Liquor ammonia*, until the latter is saturated. The latter is then passed on to a separator or still, and anhydrous ammonia driven off and re-liquefied for use again in the expansion-coil, the weak *Liquor ammonia* left, being again used for absorption. In the present central-station system, the liquefying, absorbing, and separating are done at the station, and the "expansion" in the refrigerating chamber of the customer, so that there is no loss of cooling power in transmission. The liquid anhydrous ammonia is sent out from the central station usually at a pressure of 150 lb. per square inch, at summer temperatures, along a service main called the "liquid line." Branches from this lead to the expansion-coils in the customer's refrigerating chambers, through special valves. The temperature in the chamber is automatically controlled by an ordinary thermostat, which operates an electric regulator, the current being supplied by a dynamo and storage battery at the central station. A variation of  $1\frac{1}{2}$  up or down, is usually allowed, although in some cases it is only  $\frac{1}{2}$ . The anhydrous ammonia vapour from the expansion-coil is led back to the central station through a second pipe-line, known as the "vapour line," to be there absorbed. A third line of pipes is provided, termed the "safety line," which is one of the features of the system. It is connected through cocks, which are ordinarily kept shut, with the liquid and vapour lines, near their connections with each expansion-coil, and also in the man-holes at street crossings. In event of any repairs, alterations, or additions having to be made, the cocks on the liquid and vapour lines are closed, and those on the safety line opened. A vacuum is maintained, by means of a pump at the central station, in this line, so that the ammonia vapour in the expansion-coil flows back to the station, and loss is avoided; and the coil can be opened without any ammonia vapour escaping. In a similar manner, any section of the mains can be cut into for repairs, connections, &c., by aid of the cock in the street man-holes. Lately the safety line has been made sufficiently large to serve as a temporary substitute for either the liquid or vapour lines during any alterations, thus avoiding the necessity for even a single hour's stoppage. By careful experiments on the making of joints, all leakage of ammonia has been overcome.

The demand for the cooling medium varies largely under the daily and yearly differences of temperature with all refrigerating plants, and with a central-station plant it is tenfold. The plant must be capable of meeting the largest demand, and must consequently be lightly loaded when the

demand falls, unless some means of equalising the load be adopted. In the present case, the latter is effected by using three reservoirs at the central station: one for liquid anhydrous ammonia, another for strong *Liquor ammonia*, and a third for weak *Liquor ammonia*. The anhydrous vapour returned by the vapour line passes into the absorber, which is fed by weak *Liquor ammonia*. When this is saturated, it is automatically discharged into the reservoir for the strong *Liquor ammonia*, from which it is pumped, as required, into the separator.

These reservoirs, by taking up and giving out automatically the variations from the mean demand, enable the size of the machines to be reduced, and their being worked continuously. It also allows the machines to be shut off altogether for short periods, for repairs &c., without interrupting the supply, and night work to be dispensed with in winter, leaving the absorber and reservoirs to work automatically. With the exception of the reservoirs, the plant does not differ very much from others. The quantity of "cool" supplied to each customer is determined either by measuring the liquid ammonia by a meter, or by calculation, the internal and external temperatures at the refrigerator and its internal surface being known. The cost of laying the triple distributing pipes varied from 9,000 dols. to 22,000 dols. per mile, according to the construction of the road under which they were laid. In an average town, it is estimated to be about 15,000 dols. per mile, including 100 branches for connections. A central station, with duplicate machines, and capable of supplying two or three miles of distributing mains could be built for 50,000 dols. A large station with five or six machines, each of 100 tons capacity, suitable for five or six miles of street, would cost about 200,000 dols. Should it be required to cover a large district, it is better to erect a second station at a distance not exceeding two miles from the first. The limiting distance of distribution is about the same as with illuminating gas.—R. B. P.

*Utilisation of Waste Carbonic Acid Gas in Brewing.*

A. Marcet. Trans. Inst. Brewing, **7**, 199—223.

See under XVII., page 825.

*The Prevention of Bumping in Boiling or Distilling Liquids.* V. Genhardt. Ber. **27**, 964.

See under XXIII., page 838.

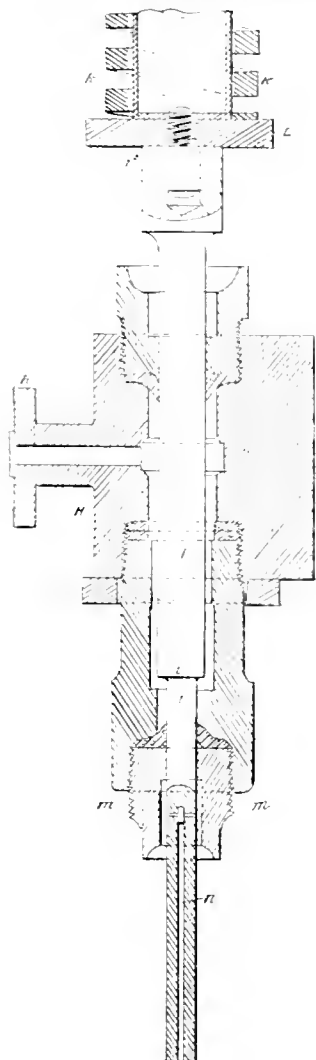
### PATENTS.

*Improvements in the Construction of Tanks or Receptacles for the Precipitation of Sewage, and for like Purposes.* E. C. Ives, Derby. Eng. Pat. 13,411, July 11, 1893.

See under XVIII.—B., page 832.

*Apparatus for Effecting the Intimate Mixture of Liquids, Semi-Liquids, or Liquefied Matters.* M. Julien, London. Eng. Pat. 14,840, August 2, 1893.

The apparatus is principally intended for mixing the constituent elements of artificial butter, so as to form a homogeneous product resembling genuine butter, and may also be used for mixing or emulsifying difficultly miscible liquids, or semi-liquids. The different constituents are supplied in regulated proportions to the cylinder II, by means of pumps worked by variable throw eccentrics. The cylinder II is traversed by a piston-rod I, of varying diameter, provided with a strong regulating spring K above, and with its lower end perforated at *n* and furnished with side inlets *m*. The roughly-mixed constituents enter the cylinder II by the inlet *h*, forcing the necked piston-rod I upwards, and thus bringing the orifices *m* above the cup-leather packing; the semi-liquid mass is discharged into these inlets under great pressure, and a thorough mixing or emulsifying takes place. The product is led away to be cooled or solidified in any well-known manner. The spring K is regulated by means of adjustable collars, so that if the pressure is insufficient the orifices *m* remain below the cup-leather packing outside the cylinder II. In a modified arrangement, a group of nozzles may be arranged in a



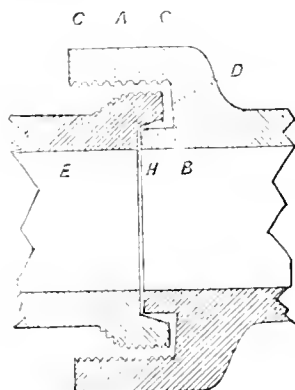
APPARATUS FOR MIXING LIQUIDS AND SEMI-LIQUIDS.

closed casing, so that the jets issuing therefrom may mix and produce the required product.—E. G. C.

#### Improvements in Earthenware and other Pipe-Joints.

R. Ewing, London. Eng. Pat. 14,926, August 4, 1893.

THE improvements made by the inventor are upon the class of joints described in specifications, Eng. Pat. 3191,



August 22, 1877, J. Rohlius; and Eng. Pat. 9028, May 31, 1889, A. Ramsden; which have cupped or recessed collars and annular grooves or double-sided sockets.

In the socket end an annular groove A is formed, with inner wall B shorter than the outer wall C, and the bottom wall projects at an acute angle from the line of the body of the pipe, so that the plastic material used for cementing the joint is locked in a tapering space D, between the two adjacent end spaces. The outer side of the spigot end is scored on the circumference at A, as is also the inner part of the adjacent socket end, thus holding the cement, which is packed into the mouth of the socket, enabling the joint to withstand a great strain.—V. C.

#### Improvements applicable to Bottles or other similar Vessels used for containing Poisonous Substances. A. Reed, Sheffield. Eng. Pat. 15,609, August 17, 1893.

A METALLIC band fastened round the neck of a bottle supports a vertical rod or wire terminating in a ring or loop, so that a bottle containing poisons and provided with this attachment may be distinguished from other bottles. The wire ring attached to its vertical support and standing over the mouth of the bottle, may also serve as a funnel-holder.

—E. G. C.

#### An Improvement in Compound Asbestos and other Fibrous Packings. A. E. H. Field, London Street, Middlesex. Eng. Pat. 15,622, August 17, 1893.

RELATES to packing of the kind in which asbestos or the like is disposed in layers with cement and caoutchouc strips or rods to form a compound packing. The improved packing has thin perforated sheets of lead or white metal introduced between the layers of fabric, so that during the rolling and pressing operations the fabric is forced into the holes, and causes the mass to cohere.—E. G. C.

#### New or Improved Process or Means for Connecting Metal to Earthenware or China. W. B. H. Drayson, London. Eng. Pat. 23,654, December 8, 1893.

See under 1X., page 811.

#### Improvements in Carbonic-Acid Pressure Apparatus for Vessels from which Liquids are Retained. N. Browne, London. From P. Beck, Augsburg, Bavaria, Germany. Eng. Pat. 1926, January 29, 1894.

THE apparatus is intended to supply carbonic acid gas to beer-barrels and the like, in quantity corresponding to the amount of liquor drawn off. The apparatus consists of a cast-iron reservoir containing carbonic acid, and provided with a reducing valve, the upper surface of which is in free communication with the barrel. When liquid is drawn off the pressure inside the barrel is reduced, and permits the reducing valve to lift and open a small outlet for the escape of carbonic acid gas into the barrel by a side passage until equilibrium is established. A safety-valve is provided to prevent the pressure in the barrel becoming excessive.

—E. G. C.

#### An Improved Mouth Blow-Pipe for the use of Gasfitters and others. W. James, Birmingham. Eng. Pat. 2370, February 3, 1894.

A RUBBER or vessel is placed between the two ends of an ordinary mouth blow-pipe, and is filled with sponge, cotton-wool, or other absorbent. This absorbent is saturated with benzoline, naphtha, or any suitable spirit, so that when the blow-pipe is being used part of the spirit passes in the shape of vapour with the breath and forms the necessary gas.

—L. T. T.

#### Improvements in Receptacles or Tanks for the Storage of Compressed Air, Gas, and Volatile Liquids. W. Timmis, Pittsburg, U.S.A. Eng. Pat. 6681, April 3, 1894.

TWO concentric cylinders are connected to one another by lugs, so that a small space is left between their sides and ends; they are further secured by stay-rods. The cylinders

are heated to a high temperature in a furnace, and while in that condition molten metal of a high density, such as lead, is poured into the annular space between the two casings so as to completely fill it. When the casings cool, the walls contract so as to fill up the pores of the metal and every opening or joint through which leakage might take place. This form of storage reservoir will sustain a very high pressure, and in the case of an explosion there will be a mere rupture, analogous to that which takes place with a copper boiler. The reservoir is especially adapted to receive compressed air, but it may be used for other purposes.—E. G. C.

#### Improvements in Apparatus for Testing Gas Cylinders.

The Scotch and Irish Oxygen Company, Limited, Polmadie, and H. Brier, Glasgow. Eng. Pat. 7914, April 21, 1894.

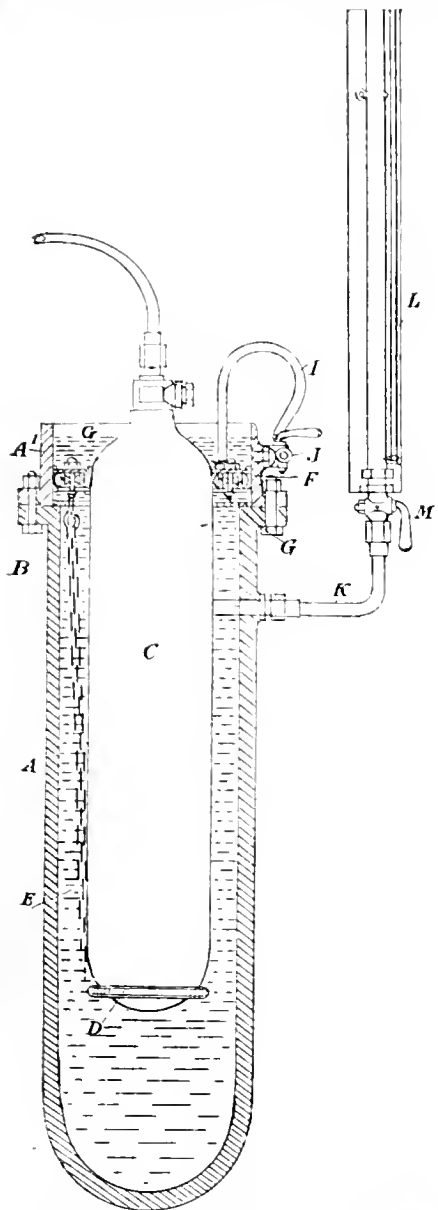
The figure shows the apparatus in sectional elevation, consisting of a cylinder A, provided with a slightly enlarged

head A' bolted thereon. The upper end of the gas cylinder C is encircled by an annular and removable ring F of india-rubber tube, which can be inflated, backed by plates G, and supported internally by an open metallic ring, which latter is kept in place by means of the clamps E, fastened to a loose ring D on the bottom of the cylinder C. Water under pressure is supplied to the ring F by means of the three-way cock J and pipe I, thereby causing the ring to press outwardly and inwardly to form a tight joint. The expansion of the cylinder C, due to internal fluid pressure, is measured by the height of the column of water in a water-gauge L, connected to the cylinder by a pipe K. A three-way cock M is provided on the pipe connection K, so that the gauge glass may be discharged, and the water-level be brought in it to the zero on the scale.—E. G. C.

#### Improvements in Heating Apparatus or Blow-Lamps for Soldering and other Purposes.

H. H. Lake, London. From B. A. Hjorth and Co., Stockholm. Eng. Pat. 9819, May 19, 1894.

This patent consists in the addition to the petroleum blow-lamp of an external metal chimney, which is contracted just above the annular vaporising or superheating tube. The object of this is to obtain a more regular and intense heating of this superheater, and so attain a more regular and intense flame.—L. T. T.



APPARATUS FOR TESTING GAS CYLINDERS.

## II.—FUEL, GAS, AND LIGHT.

*Electric Heating.* Zeits. f. Elektrotech. u. Elektrochem. 1894, 8—10.

Usual recently it has been generally assumed that heating by electricity is, on account of its cost, of little practical value. But our present experience (e.g., of electric welding) throws doubt on this assumption, and the question is one which must be settled by experiment and measurement. It is true that electrical energy is more expensive than thermal energy derived directly from combustion. But the energy has to be applied to the body which we wish to heat; and in this transference the advantage lies with the electrical form, for the losses by conduction, convection, and radiation are very much smaller. The question therefore takes the form: Which in the end is more economical, electrical energy, which is dear, but convenient and adaptable; or thermal energy, which is cheap, but wasteful in its application? Few data are available for preparing an answer to this question. J. L. Roberts has recently made a useful contribution (Elect. Engineer, Jan. 17, 1894) to our knowledge of the subject; but the writer does not regard his results as decisive, more especially because he has not fully taken into account the economies which are nowadays realised in furnaces. The writer quotes from Rohrbach the following data respecting the metals which chiefly interest electricians, viz., iron, copper, and platinum:—

According to Wagner, 165.75 kilos. of coke are required for the production of 100 kilos. of iron. According to Hering, 1,060 kilos. of coke are required for the production of 100 kilos. of 96—98 per cent. refined copper (English process). In the old Mühlbach furnaces, when ores containing 13 per cent. of copper are smelted, 691 kilos. of coke are employed in producing 100 kilos. of copper. With these results he compares the expenditure (from the smelting to the final preparation of the electrolytic copper) in the Bessemer-copper process. This amounts to only 300 kilos. of coke for 100 kilos. of pure copper. In order to melt 1 kilo. of platinum 300 litres of coal-gas and 100 litres of oxygen are required. As regards the relative cost of furnaces and other plant, the writer is of opinion that the cost of the electrical plant would be as great as, or slightly greater than, that of the usual combustion plant; but the working expenses would be smaller and



the hygienic advantages would be very great. Finally, it should be borne in mind that in smelting operations carbon plays a double part, namely, in the reduction of the ore and in the production of the temperature requisite for this. In the former function it is undoubtedly more economical than the electric current. In the latter function it probably is not; and it is in this direction that fruitful applications of the electric current must be sought.

—D. E. J.

*On the Efficiency of Heating by Electricity as compared with Heating by Combustion.* A. Wilke. Zeits. f. Elektrotech. u. Elektrochem. 1894, 2—3.

THE writer describes and discusses certain experiments made by J. L. Roberts (Electrical Engineer, 1894, Jan. 17), with the object of measuring the relative efficiency of these two methods of heating. Roberts heated masses of metal (rods of platinum and iron) to a red heat, first by combustion methods and then by the electric current. He also heated brass turnings by both methods up to the melting-point. When the substance was heated by a flame, the energy expended was calculated from the loss of weight of the combustible and the (known) corresponding calorific power; when electrical energy was used, the energy expended was calculated from the current and electro-motive force. The amount of heat transferred to the metallic mass was determined by a simple calorimetric experiment (by the well-known method of mixtures). In the first series of experiments a platinum rod, 5 cm. long and 2 mm. thick, was heated in the flame of a spirit-lamp burning absolute alcohol. The wick of the lamp could be covered with a glass cap; and it was weighed before and after the heating. This was effected by holding the platinum rod by means of tongs in the hottest part of the flame; as soon as it became red-hot it was dropped into the calorimeter (a glass beaker). The complementary experiment was made by holding the rod or wire in copper clamps and passing a current through it until it again became red-hot. The results showed that less than 0.5 per cent. of thermal energy produced in the flame was transferred to the bar; whereas 90 per cent. of the energy was utilised in the electrical method.

In the second series of experiments an iron bar weighing about a kilo. was heated, first by means of well-dried charcoal, and then electrically by a Thompson-Schweiss apparatus. In the former case about  $\frac{3}{4}$  per cent. of the thermal energy produced was transferred to the bar; in the latter case about 88 per cent.

In the third series of experiments brass turnings were heated, first by means of charcoal and then in a specially-constructed electric furnace. The efficiency was  $1\frac{1}{2}$  per cent. in the former case and about 85 per cent. in the latter case.

When coal is burned under the boiler of an electric installation, about 10 per cent. of the thermal energy reappears as mechanical energy, and about 9 per cent. as electrical energy. We thus see that in the end about 8 per cent. of the original thermal energy of the coal can be utilised in electrical methods of heating; whereas only 1 to 2 per cent. can be utilised in direct combustion methods. It is evident that the experiments above described were of a somewhat crude nature; but they show sufficiently well that, even in respect of efficiency and economy, electrical methods of heating are deserving of careful consideration.—D. E. J.

*Natural Gas: its Composition and Origin. The Origin of Petroleum. "Researches upon the Phenomena of Oxidation and Chemical Properties of Gases."* Parts 4, 5, and 6. F. C. Phillips. Amer. Chem. Jour. 16, 106—129.

1. *The Composition of Natural Gas.*—The material the author has principally worked upon has been natural gas, as supplied in Allegheny by the Allegheny Heating Company. It is the product of many wells scattered over a considerable area, and represents the

average composition of an enormous volume of gas. From 1886 to 1892 no important differences in its composition have been observed, although occasionally the odour of the higher paraffins has been more strongly marked than usual. Gas from various localities in Pennsylvania, New York, Indiana, Ohio, and British Columbia has also been examined.

In the accompanying table (1.) of analyses will be found the author's results, collated with nine results of the work of others, and these may be compared with the analyses of Russian natural gases shown in Table II. The absence of hydrogen, carbon monoxide ("nearly always stated to occur in natural gas"), the olefines, and sulphur compounds is maintained, but only after a most elaborate search for them, by the author. It is, however, to be noted that gas from Western Ohio and from Canada, which is said to contain sulphur compounds in considerable quantity (see also this Journal, 1891, 507), was not examined by the author. Except in the case of a "gas-well" near Canonsburg—where, after passing the gas through water for several hours, a feeble Nessler reaction was obtained,—ammonia was never found in the gases. Upon the authority of S. A. Ford, of the Edgar Thompson Steel Works, a case is reported where masses of solid ammonium carbonate were blown out of a well by the pressure of the gas.

The paraffins are chiefly methane, with traces of the higher hydrocarbons of the series.

Nitrogen and carbon dioxide are, on the other hand, probably always present.

2. *The origin of Natural Gas and of Petroleum.*—The author inclines to the original hypothesis put forward by the geologists to account for the formation of these substances in nature; or rather, from a study of the phenomena of the slow decay of vegetable matter under water in the absence of air, he puts forward a modification of this hypothesis, considering that, after the first somewhat sudden and tumultuous evolution of gas, vegetable tissue under the above circumstances, relapses into an extremely slow and long continued process of decay. This process may, as he shows, be artificially imitated, and, as he maintains, is the only one known which yields a gas at all similar in composition to that found in nature. Thus, the gas at first given off consists of  $\text{CO}_2$ , 18.23;  $\text{CH}_4$ , 0.30;  $\text{H}_2$ , 62.24;  $\text{N}_2$ , 19.23; while that given off in the succeeding very slow process was  $\text{CH}_4$  only.

The hypothesis of Mendelëeff is objected to, mainly on the grounds that practically no free hydrogen is found in natural gas, though this would be the case, were it the product of the reaction of superheated steam upon metallic carbides. Moreover, natural gas contains neither olefines nor carbon monoxide, yet paraffins alone cannot be produced by any known method of reacting upon iron carbide with steam. The author found the gas evolved by the action of dilute sulphuric acid upon ferromanganese to contain 6 per cent. of olefines.

With regard to Engler's hypothesis, it is pointed out: firstly, that chemically, the gases obtained by Engler, by distillation of menhaden oil and of oleic acid, are very different from natural gas, containing, as they do, from 25 to 38 per cent. of carbon monoxide, and from 12.5 to 2.3 per cent. of olefines, while the paraffins are correspondingly low; secondly, that it is very difficult to point to any known chemical method or any natural process (such as solution in water) by means of which some of the constituents of of such a gas might have been abstracted, so that the residual gas might have come to resemble natural gas, as shown; thirdly, it is pointed out that animal tissues are not capable of a two-stage decomposition, such as is indicated above for vegetable tissues, but when decay once sets in it is rapidly carried on, and without intermission, to their complete destruction.—E. R. B.

## I.—ANALYSES OF NATURAL GAS (AMERICAN).

Constituents.	1. Fredonia, N.Y.	2. Sheffield, Warren Co., Pa.	3. Kane, McKean Co., Pa.	4. Wells, McKean Co., Pa.	5. Steele, near Oil City, Pa.	6. Lyons Ranch, Murrys- ville, Pa.	7. Ravens- croft, Pa.	8. Houston, near Cameron, Louis, Pa.	9. Murrys- ville, Pa.
Nitrogen .....	9.54	9.06	9.79	9.41	4.51	2.92	9.24	15.19	4.59
Carbon dioxide .....	0.11	0.30	0.29	0.21	0.05	0.28	Trace	0.44	0.29
Hydrogen .....	0	0	0	0	0	0	0	0	0
Ammonia .....	0	0	0	0	0	0	0	0	0
Oxygen .....	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Sulphuretted hydrogen .....	0	0	0	0	0	0	Trace	0	0
Paraffins .....	90.05	90.64	90.01	90.38	95.44	97.70	90.69	84.26	95.10
	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
The paraffins contained in these gas samples have the following composition by weight:—									
Carbon .....	78.14	76.69	79.77	79.52	77.11	74.96	79.42	76.68	75.15
Hydrogen .....	21.86	23.31	20.23	20.48	22.89	25.04	20.58	23.32	24.85
	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Constituents.	10. Pittsburgh, Exhibition Grounds, Pa.	11. Cleveland, O.	12. Creighton, O.	13. Painters & Co.'s, U.S.A., Pittsburgh, Pa.	14. Baden, Pa.	15. Kokomo, Ind.	16. Allegheny City, Salt well.	17. Vancouver, British Columbia.	
Nitrogen .....	7.34	6.50	0	6.70	12.32	6.90	7.10	6.50	
Carbon dioxide .....	0.52	0.20	3.64	0.40	0.41	0.40	0.50	0.14	
Hydrogen .....	0	0	0	0	0	0	0	0	
Ammonia .....	0	0	0	0	0	0	0	0	
Oxygen .....	Trace	0	0	0	6	0	0	0	
Sulphuretted hydrogen .....	0	0	0	0	0	0	0	0	
Paraffins .....	92.18	93.50	96.36	98.90	87.27	93.70	92.90	93.56	
	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	
The paraffins contained in these gas samples have the following composition by weight:—									
Carbon .....	75.40	76.40	75.80	75.51	76.18	75.40	75.44	75.23	
Hydrogen .....	24.60	23.60	24.20	24.49	23.82	24.60	24.56	24.77	
	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	

Nos. 1, 2, 3, 4, 5, 6, 7, 8, 14, are cited from Report of Geol. Survey of Pennsylvania, 1886; the remaining analyses are by the author. Tests were made at the wells in all cases, except those of Kokomo and Vancouver.

## II. ANALYSES OF NATURAL GAS (MOSTLY RUSSIAN).

	1.	2.	3.	4.	5.	6.	7.	8.
Carbon monoxide .....	0	0	0	0	0	0	0	0
Carbon dioxide .....	0.95	2.18	3.50	0	2.47	4.44	0	0.3
Olefines .....	4.11	3.26	4.26	0	0	0	0	..
Methane .....	92.49	93.07	92.24	95.39	97.57	95.56	1.90	..
Hydrogen .....	0.94	0.98	0	0	0	0	0	..
Nitrogen .....	2.13	0.49	..	..	..	..	96.57	96.8
Oxygen .....	..	..	..	..	..	..	1.53	2.9
	100.02	99.98	100.00	..	100.04	100.00	100.00	100.00

Nos. 1, 2, 3, 4, 5, and 6: natural gas from the Caspian region. Communicated to the author by M. Behnmin of Messrs. Nobel Bros., St. Petersburg.

No. 4 is the result of a partial analysis. Nos. 7 and 8: gas from deep borings at Middlesbrough (Bedson: this Journal 17, 922).

*Liquid Fuel.* E. N. Henwood. J. Soc. Arts, 42, 703—704.

The author now contributes further remarks upon this subject, supplementary to those he made during the discussion upon Mr. Stockfleth's recent paper (see this Journal, 1894, 625).

1. He does not agree with Stockfleth's opinion, that the most primitively constructed pulverisators answer as well as the more complicated kinds: on the contrary, the atomiser he employs upon his yacht "Ruby," which is one delivering the oil-fuel as vapour, is practically noiseless, and effects considerable economy. He further states that the great "roar of the blast" is "extremely detrimental" both on vessels on the Caspian and on the locomotives on the Great Eastern Railway.

2. According to Stockfleth the results of atomising the oil by the use of compressed air, "do not materially differ from those obtained with steam": in the author's experience complete combustion is not obtained with compressed air, much soot being deposited in the boiler-tubes.

3. With regard to fire-box arrangements, the author prefers to remove the fire-bars, to entirely close the front of the furnace, and to lead the air-supply through a pipe passing down the funnel: by this procedure heated air supports the combustion, the entire surface of the fire-box may be utilised for heat absorption, and the further advantages are obtained, that the stokehole is perfectly clean, and should any water, shipped during a heavy sea, find its way here, it cannot reach the heated surfaces of the closed-in combustion chamber.

4. The author considers the Petersen's "compound tube water-tube boiler" especially suited to the requirements of liquid fuel, on account of the largely augmented heating surface of the tubes, and other advantages by which—in the author's opinion—"it surpasses all others of every kind."

5. The committee of Lloyd's, the author states, having approved of the oil-fuel system, there can no longer be any question raised as to its safety. Last year's unsatisfactory results on the Clyde are stated to have been caused by "crude arrangements."—E. R. B.

*Estimation of the Secondary Products containing Nitrogen formed during Combustion in the Air.* L. L. de N. Rosva. Bull. Soc. Chim. 11, 1894, 272—280.

It is well known that nitrous and nitric acids are among the secondary products of combustion, and, if the body burnt contains hydrogen, ammonia also. The author endeavours to show the relation between the quantity of these products formed and of the substance burnt. The substances experimented upon were hydrogen, carbon monoxide, coal-gas, wood-charcoal, and coke. Oil was excluded because much ammonia is evolved during heating previous to combustion.

The determinations included estimation of the nitrogen present in the three forms of nitrous acid, nitric acid, and ammonia. In the case of gases the time of combustion for 8—9 litres was 2—3 hours. The charcoal and coke required 8—10 hours for burning, and in this time a certain amount of atmospheric nitrogen would become oxidised to nitrous and nitric acids. A blank experiment served to determine this amount of nitrogen, which was subtracted from the total found. A source of error was traced, in that part of the ammonia formed during the burning of charcoal and coke is derived directly from the substance, and not from atmospheric nitrogen. In fact, in every way the results with charcoal and coke were less satisfactory than in the case of gases: for with the latter the modifying influence of external heat is excluded.

The results may be tabulated as follows:—

I. By burning 1 cubic metre of the gases. (The products are given in grms.)

	HNO <sub>2</sub>	HNO <sub>3</sub>	NH <sub>3</sub>	Nitrogen		Total Nitrogen.
				Contained in Oxidation Products.	Contained in Ammonia.	
Coal-gas .....	0.0212	0.1184	0.0033	0.0401	0.0027	0.0428
Hydrogen .....	0.0215	0.1022	0.0026	0.0299	0.0021	0.0320
Carbon monoxide.....	0.0500	0.0169	.. ..	0.0184	.. ..	0.0184

II. By burning 1 kilo. of the same gases.

	HNO <sub>2</sub>	HNO <sub>3</sub>	NH <sub>3</sub>	Nitrogen		Total Nitrogen.
				Contained in Oxidation Products.	Contained in Ammonia.	
Coal-gas .....	0.0408	0.2554	0.0063	0.0771	0.0052	0.0823
Hydrogen .....	0.2403	1.1411	0.0257	0.3288	0.0236	0.3522
Carbon monoxide.....	0.0339	0.0132	.. ..	0.0147	.. ..	0.0147

III. By burning 1 kilo. of wood or of coke.

	HNO <sub>2</sub>	HNO <sub>3</sub>	NH <sub>3</sub>	Nitrogen		Total Nitrogen.
				Contained in Oxidation Products.	Contained in Ammonia.	
Wood-charcoal dried at 120° C. ....	0.1085	0.3832	0.7874	0.1190	0.6481	0.7671
Wood-charcoal heated to 600° C. for two hours .....	0.1589	0.4186	0.4461	0.1279	0.3679	0.4958
Wood-charcoal heated to 900° C. for two hours.....	.. ..	.. ..	0.0278	.. ..	0.0229	0.0229
Coke heated to 600° C. for two hours .....	0.1122	0.6244	0.1566	0.1756	0.1289	0.3045

IV.—Equivalent values of the products of combustion containing nitrogen. (This table shows the proportion for each combustible between the nitrogen present in the three forms.)

	Equivalent Values.		
	$\text{-- NO}_2$	$\text{NO}$	$\text{NH}_3$
Coal-gas .....	2.4	12.4	1.00
Hydrogen .....	3.4	16.9	1.00
Carbon monoxide .....	4.1	1.0	..
Wood-charcoal heated to $120^\circ \text{C.}$ ..	1.0	2.6	19.61
Wood-charcoal heated to $600^\circ \text{C.}$ ..	1.0	1.95	7.58
Coke heated to $600^\circ \text{C.}$ .....	1.0	4.1	3.77

These figures prove:—

1. That in burning equal volumes of hydrogen and coal-gas, the same weight of nitrogen is transformed into ammonia.

2. That with equal volumes of hydrogen and coal-gas almost equal weights of nitrous acid are formed, but with carbon monoxide  $2\frac{1}{2}$  times as much.

3. In burning equal weights of the gases, by far the largest amount of nitrogen is combined in the case of hydrogen, less than one-fourth of this amount with coal-gas, and scarcely a twentieth with carbon monoxide.

4. That in burning wood-charcoal or coke the ammonia is not produced from atmospheric nitrogen, but as a direct decomposition product of the substance; it would otherwise be difficult to understand why the weight of ammonia should vary according to the heat previously applied. Probably, as a result of the true combustion of charcoal and coke, only oxygen compounds of nitrogen are formed, the weight of these being fairly constant, and the difference shown in the table for coke being due to experimental error.

5. In burning coal-gas and hydrogen the sum of the nitrogen equivalents (see Table IV.) of the acids is 14—15 times as great as that of ammonia; in the case of coke the equivalents of the acids also surpass that of ammonia, the reverse being the case with charcoal.

6. Except with carbon monoxide, more nitrogen is always present as nitric than as nitrous acid.

7. It appears that the maximum of oxidation products is formed in burning gases when the heat of combustion is least, while ammonia is formed in larger quantity as the heat of combustion increases.

The above results give us a new basis for estimating the quantity of nitrogen compounds contained in the air which can serve as plant food. The author concludes that approximately 2,900 metric quintals (1 metric quintal = 100 kilos.) of combined nitrogen pass annually into the air in the form of secondary products of combustion, and become available for the nourishment of plants. This figure is many hundred times less than that given by Boussingault, Lawes, Gilbert, Chabrier, and Bineau.

Doubtless the amount of this nitrogen varies greatly in different regions of the earth. In the author's opinion, the nitrogen present in the air combined with oxygen is in the form of nitric peroxide, with perhaps small amounts of trioxide. It is very probable that nitric peroxide is responsible for many of the effects hitherto attributed to atmospheric ozone. The phenomena of the decolorisation of certain colouring matters by gaseous products of combustion, which is generally attributed entirely to sulphur dioxide, is possibly also partly due to nitric peroxide.

—R. B. B.

*Electrical Heating by Induced Currents.* Zeits. f. Elektro-  
tech. u. Elektrochem. 1894, 93.

See under XI., page 816.

## PATENTS.

*Improved Apparatus for enabling a Person to Breathe Fresh Air while surrounded with a Poisonous or Noxious Atmosphere, more especially for use in saving Miners from being Suffocated by Choke-Damp or After-Damp.* C. A. Ash, Newport, Salop. Eng. Pat. 13,982, July 19, 1893.

This consists of a hood to fit over the head, made of india-rubber or other soft material, with glazed sight-holes, the lower part being made of india-rubber with an opening at the bottom, which is stretched to admit the head, and then closes round the neck. Reservoirs of india-rubber or light metal, and conveniently formed to carry on the arm, are inflated with air under pressure. These are connected with the hood-piece by india-rubber tubing, the whole being fitted with the necessary valves.

—L. T. T.

*Improvements in or connected with Burners for Hydrocarbon Oils.* R. Lehming, Berlin. Eng. Pat. 15,960, August 23, 1893.

With the view of removing liability of explosion, either by ignition of the body of petroleum vapour in the lamp-reservoir, or, if the lamp is upset, by the passage of oil to the wick, the patentee proposes to surround the wick with a metal tube reaching nearly to the bottom of the reservoir. This tube is detachable, and is attached to the main body of the burner (which is constructed accordingly) by a bayonet or cone connection, so as to be approximately airtight. By this means only the small quantity of vapour above the oil in this tube is in contact with the wick, and should the lamp be upset, the greater portion of the oil is effectually prevented from flowing out through the burner.

—L. T. T.

*Improvements in Round Burners for Mineral Oil.* L. Sepulchre, Herstal, near Liège. Eng. Pat. 16,989, September 9, 1893.

This invention consists in introducing into round disc-burners a second cone outside the one usually present. The second cone is of the same height, or slightly higher, than the usual cone, and is intended to cause a second or additional current of air to impinge on the outside of the flame. It is claimed that additional brilliancy of flame is thus obtained. Various forms of cones are shown.—L. T. T.

*Improvements in and relating to Incandescent Gas Lighting.* M. Bernstein and A. Silbermann, Berlin. Eng. Pat. 7372, April 13, 1894.

The claim of this patent is for a luminous body for incandescent lamps, consisting of a series of superposed inverted cones of varying diameter, made of chamotte or kaolin, or a mixture of the two, and impregnated with a fluid, leaving on heating a residue of magnesia, and called the "illuminating fluid." These cones are arranged on a rod, above a Bunsen burner, so that the edges, which are bevelled and made particularly porous, form an inverted cone just above the tube of the Bunsen. The cones may either be solid or perforated.—L. T. T.

*A New or Improved Composition for so-called Incandescers for Oil, Gas, or other Lamps.* F. H. Medhurst, London. From L. Chander, St. Petersburg. Eng. Pat. 10,427, May 29, 1894.

This composition consists of 55 parts by weight of powdered porcelain, 15 parts of asbestos, 15 parts magnesium oxide, 10 parts of chalk, and 45 parts of silicate of soda or potash. The first four constituents are made into a paste with the silicate, and this paste is moulded into the desired shape, and dried in a drying oven. This composition is said to be highly incandescent, and cheap.—L. T. T.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*The Tar of the Bark of Populus Tremula.* N. Pharmakoffsky. Jour. Ph. Ch. russe, 6, 423—429.

THE tar is a dark-coloured thick liquid of acid reaction. For the neutralisation of 1,000 grms. of it, about 30 grms. of  $K_2CO_3$  are required. Fractional distillation gave the following results:—

		Temperature,			
		100—150°	150—185°	185°—210°	210—260°
Distillate from 100 lb. of tar		3.45	12.3	11	25
		Temperature,			
		260°—330°	Above 330°	Solid Fraction.	
Distillate from 100 lb. of tar		4.6	11.6	15	

The first two fractions form a yellow oil, which turns red on standing. The fraction boiling above 360° C. deposits a green mass slightly soluble in alcohol and ether, very soluble in carbon bisulphide and benzene, and decomposing at 240° C. The rest of the fraction forms a dark-red mass, very soluble in alcohol. The substance is deposited, in the amorphous state, from this solution on cooling; the supernatant alcohol, however, preserving its red colour, which, in reflected light, is green. On treating the mass with ether, a green substance, similar to the one previously mentioned, is obtained. The whole of the tar, and especially the first four fractions, are acid. On treatment of these fractions with potassium carbonate, the salts of valericianic, propionic, formic, and benzoic acids are obtained. The presence of the last-named acid is due to the deposit of populin,  $C_{13}H_7O_7(C_7H_5O)_2$ , contained in the bark. The various fractions were washed with caustic soda, and the solutions obtained, decomposed by the addition of sulphuric acid, in order to separate the acids from the phenols. The re-distillation of the last fraction boiling above 360° C. yields two

fractions, the one being liquid, and containing a few crystals only, showing a bright colour and strong fluorescence; the other forming a compact deposit of fluorescent crystals, with very little adherent oil. Both fractions placed in the dark, in an atmosphere of carbonic acid, turned brown after some time, preserving, however, their fluorescence. The fractions boiling above 270° C. deposit crystals of paraffin, amounting to about 2 per cent. of the tar.—C. O. W.

*The Ethyl Phenols.* A. Behal and E. Choay. Bull. Soc. Chim. 1894, 11, 206.

THIS investigation was undertaken in order to identify the  $C_2$ -compounds occurring in creosote with the ethyl phenols. The ethyl phenols were obtained from ethyl-benzene, prepared according to Friedel and Crafts's method, by converting it successively into nitro-, amido-, and diazo-compounds. By boiling the latter with water, the corresponding phenols were obtained. Ortho-ethyl phenol, a liquid boiling at 203° to 204° C., specific gravity 1.0371 at 0° C. Meta-ethyl phenol, a liquid crystallising at about -4° C., boiling at 214° C., and of a specific gravity 1.0250 at 0° C. Para-ethyl phenol, melting at 46° C., boiling at 215° to 216° C., and of a specific gravity 1.0403.—C. O. W.

*Present State of the Petroleum Industry.* M. P. de Boisieu. Bull. Soc. Chim. 11, 1894, 453.

CRUDE petroleum varies very greatly in its chemical nature, and its technical treatment correspondingly varies; moreover, the increasing number of products obtained from it, and their augmented purity, has of late years necessitated modifications and elaborations of the processes employed; the author has therefore contrasted the state of the art as practised in different countries and with different oils.

1. *America.*—The first fractionation of the crude petroleum is conducted in cylindrical stills of 500—600 barrels capacity, made of  $\frac{1}{4}$ "— $\frac{3}{8}$ " steel plate, and set horizontally in brickwork, like ordinary boilers.

After leaving the stills the vapours are conducted through long iron tubes kept as cool as possible by abundance of water, for it is found that the losses during refining are greatly diminished by adopting this method and by thus working with properly cooled distillate.

At the works of the Standard Oil Company at Bayonne, near New York, six fractions are collected at this first distillation, as shown by the following tabular statement:—

No.	Name of Fraction.	Specific Gravity.	Destination of Fraction and its Products.
1	Light naphthas .....	Up to 0.705 .....	a. Sold as it is, as crude naphtha. b. Sold after re-distillation by steam.
	Heavy naphthas .....	0.705 to 0.744 .....	c. Fractionated into gasoline (sp. gr. 0.696—0.675) and naphtha (sp. gr. 0.675—0.720). d. Fractionated into many products: cymogene, rhigolene, gasoline, light naphtha, naphtha for lighting and heating, and heavy naphtha (substitute for turpentine).
	Extra heavy naphthas (light distillate).	0.744 to 0.765 .....	Fractionated into benzine (added to heaviest fraction of No. 1) and light distillate.
4	"Water white" .....	0.765 to 0.795 .....	Mixed in certain proportions with 5, and refined; the product being "export oil" (flashing point 21° to 24° = 69.8 to 75.2 F.).
5	"Heavy distillate" .....	0.795 to 0.825 .....	After refining is white lighting oil.
6	"Slops" .....	0.825 to the point when oil turns very brown.	.. .. .. ..
7	Residue .....	.. ..	c. Sold, as gas oil, to the gas companies. b. Added to the crude petroleum next to be distilled.
8	{ Tar .....	.. ..	This, while still boiling, is pumped into a vessel where, upon cooling, it separates into:—
9	{ Coke .....	.. ..	Treated subsequently for paraffin and lubricants; and
			.. .. .. ..

The refining of lighting oils is carried out in large reservoirs, constructed of iron plate lined with lead, the funnel-shaped bottoms of which terminate in cocks for

drawing off the waste liquors. To the oil, which is kept in a state of agitation by a current of air, and the temperature of which does not exceed 15°, there is added, little by

little, one half of the sulphuric acid to be used, and the agitation is continued as long as the temperature rises, when the mud-like waste acid ("sludge") is drawn off. The operation is repeated with the second half of the acid, the waste acid being now merely coloured. It can be used a second time. The now straw-coloured oil is thoroughly washed by water, which is forced into the oil from apertures in a tube which passes all round the upper part of the reservoir, and, when acidity has almost disappeared, a solution of soda, of 15 B., is introduced with agitation. The oil is now conducted to the settling tanks, where it may be warmed, to assist the clarifying, by worms suitably placed. Many works, however, prefer to filter through sawdust or coke-dust, as there is risk of colouring the oil yellow by warming it. In some works the oil is washed after the treatment with alkali. Dilute ammonia is sometimes used as alkali. The soda solution employed is about 1 per cent., and the acid about 1.5 per cent. of the volume of the oil to be refined.

The very sulphurous Lima oils are treated by the Standard Oil Company at Whiting, near Chicago, by two recently invented processes. The first of these consists in passing the vapour of crude oil over copper oxide, while with the second the crude oil is distilled in the ordinary way, and the distillates so obtained are redistilled from stills containing copper oxide, the still-contents being kept violently agitated during the distillation. This process is supplanting the other: both require a large excess of copper oxide. The residue, consisting of tar and copper sulphide, is drained, filter-pressed, and calcined, little heat being required for the latter operation, when the oxidation has once set in.

Most of the by-products are utilised; thus, the gases given off at the commencement of distillation are used as

fuel, and so also are the heavy oils, not used for the preparation of lubricants and paraffin. Sulphurous tars are mixed with water and the tar separated from the dilute acid, which is then employed for the manufacture of ferrous sulphate. Sulphuric acid is recovered from sulphur derivatives of petroleum by treatment of these with superheated steam. Other acid and alkaline residues and coke are utilised by ordinary methods.

2. *Russia.* In contrasting the Russian petroleum industry with the American, the following fundamental differences are to be noted:—

(a.) The Russian industry is in the hands of many owners, while the Standard Oil Company produces 85 per cent. of the American petroleum.

(b.) Most of the Russian oil is raised in a small territory around Baku, while there are several American oil-fields, of great extent and considerable distances apart.

(c.) Chemically the oils differ greatly; the Russian containing no paraffin, practically no sulphur, and very little petroleum-spirit, but, with great constancy, 10 per cent. of lighting oil and 40 per cent. of lubricating oil. Further, it is made up of naphthenes and a small quantity of aromatic hydrocarbons, while American petroleum is composed of fatty hydrocarbons, and varies considerably between oils rich in light products, such as those of the Washington and MacDonald Fields; oils rich in heavy products, such as those of Bradford; and sulphurous oils, such as the Lima oils.

(d.) The method adopted in the distillation of the crude petroleum is also different.

Ordinary Baku crude oil has a specific gravity of 0.868—0.885, a flashing point of 35—45°, and a firing point of 15—57°, and is separated, at the first distillation, into the fractions indicated by the following tabular statement:—

No.	Name of Fraction.	Specific Gravity.	Flash-Point.	Products and Uses of the Fractions.
1	Gasolines and spirits,....	0.718—0.760	Under 28	Export forbidden by Russian Government (being a petroleum product containing substances flashing under 28°).
2	Kerosenes,.....	0.760—0.800	28°—55°	Fractions mixed or kept separate according to kerosene required. Usually "Russian kerosene" has specific gravity = 0.822—0.823 and flashing point = 28° ("Kerosene No. 1"). Unlike the American parallel products, never contain substances flashing from 20°—25°.
3	Solar oils,.....	0.866—0.875	Above 100°	Generally mixed with 7, the whole constituting liquid fuel. With a specially designed lamp is excellent illuminant. Also highly suitable for gas-making, on account of high content of aromatic hydrocarbons. (100 kilos. give 2,100 cub. ft. of gas 3½ times stronger than coal-gas—Schibaneff.) This fraction constitutes 15—18 per cent. of the production.
4	Spindle oils,.....	0.892—0.896	153°—163°	Lubricating oil with viscosity = 2.21—2.16 and firing point of 171°—181.
5	Machine oils, No. 1.....	0.906—0.908	170°—175°	Lubricating oil with viscosity 6.5 and firing point 210°—214.
6	Cylinder oils, No. 0.....	0.911—0.912	205°—207°	Lubricating oil with viscosity 9 to 11 and firing point 236°—239.
7	Tar (astatki).....	.. ..	.. ..	1. Liquid fuel. 2. Mixed with 3: the product being used as liquid fuel and in gas manufacture. 3. Caulking of ships. 4. Briquette-making. 5. Constituent of wazgon grease, &c.

The majority of the works only turn out lighting oil; and their residuum, known as "Mazout," is taken in hand by the makers of lubricating oil.

Baku crude petroleum only contains from 33 to 40 per cent. of lighting oil; by very careful manipulation, however, the percentage may be raised to 44 per cent., though the average practice of the Baku works barely exceeds 38 per cent. The yields therefore are:—

1. Spirit, &c.,.....	2
2. Lighting oil.....	38
3. "Mazout".....	100
	100

"Mazout" yields:—

a. Spindle oil.....	12—13
b. Machine oil.....	27—28
c. Cylinder oil.....	1—5
d. Tar and coke.....	17—14

60 60

The most important improvement which has been introduced into the distillation process is the employment, which is becoming very general, of apparatus which can be kept constantly charged. Great advantages are presented by this method over the old intermittent charging, with its occasional spontaneous ignition of the vapours of "Mazout" when the still was emptied, its injuries to the still through the latter being alternately exposed to high and to low temperatures, and its waste of the time necessary for cooling the still, before it was safe to re-charge it with fresh crude oil of initially low boiling point.

Several continuous methods, all based on the same principle, have been proposed; that of Intkisch, one of the most recent, being conducted as follows:—

A number of stills, of like capacity, and each provided with its own heating and steam-injecting apparatus and condensing plant, are arranged in file and connected up, so that the overflow of the first is the supply-pipe of the second,

and so on. The first still is fed from a large tank, placed above it, and containing the stock of crude oil. In this tank is a worm, through which passes the hot overflow oil from the last still: the crude oil entering the first still is therefore warm.

The temperature and the rate of oil-flow are carefully regulated, with the result that each still yields constantly a distillate of a given density. The results are excellent and the apparatus very manageable. Refining is conducted much as in America.

Several attempts have been made to manufacture benzene, anthracene, and other aromatic hydrocarbons, from Baku petroleum, especially in connection with the manufacture of gas from this petroleum, and the author himself conducted experiments, in 1891, at St. Denis (see Bull. Soc. Chim. 1892—3), whereby he was able to show a production of 80 per cent. benzene, at 40 fres. per 100 kilos.; but the heavy fall in price of this substance, from 120 fres. in 1891 to 50—60 fres. per 100 kilos. at the present time, and the anticipation that still further reduction will take place in the price, has deprived this work of the commercial importance it would otherwise have had. (This Journal, 1892, 104.)

3. *France*.—Here crude oil is distilled, by the naked flame, into fractions with the following densities:—

1. Up to 0.725 = 15 per cent. of the charge.
2. 0.725 to 0.740
3. 0.740 to 0.810 } = 70 " "
4. Above 0.810
5. Residue of tar = 15 " "

The tar is submitted to "cracking," in special retorts ("black pots"), by which means about 75 per cent. of useful hydrocarbons are obtained from it, while the loss (gas and coke) is about 25 per cent. After rectification, the bulk of these hydrocarbons are obtained, as an oil of sp. gr. 0.786.

The author acknowledges his indebtedness to the report of MM. Riche and Roume, who in 1892 investigated the state of the American petroleum industry, at the instance of the French Ministry of Commerce and Industry.

—E. R. B.

#### *Petroleum in Sumatra.* J. Soc. Arts, 42, 704.

THE petroleum deposits, discovered in the province of Langkat, at the north of the island, and along the coast of the Malacca Straits, are being rapidly developed. The Netherlands-Indian Government have granted 318 square miles of concessions to Dutch and English capitalists, but only the former are at work.

The wells are put down close to the coast, where there is an excellent harbour. The costs of transit and of shipment are not heavy, the quality of the oil is very good, and it is believed that this portion of the island is very rich in petroleum. Sumatra may therefore, before long, enter into competition with Russia and with America as to petroleum.—E. R. B.

*Asphalt Paving.* Report to the Department of Public Works, Philadelphia, U.S.A. (Pamphlet.) 1894. (This Journal, 1893, 265.)

AN investigation has recently been made by a committee of experts to decide upon the merits of different kinds of asphalt for street paving. The comparative tests carried out for the purpose of the investigation are here abstracted.

The work was divided into three groups—

1. *Chemical Examination*.—On account of the abstention of certain asphalt companies who were invited to offer their raw materials for examination, only two distinct kinds of asphalt and their corresponding paving mixtures were exhaustively dealt with. The following table affords a comparison between the two varieties:—

	Refined Trinidad Asphalt.	Refined Bermudez Asphalt.
Specific gravity at 60° F. ....	1.373	1.471
Bitumen soluble in carbon bisulphide....	61.507 %	97.22 %
Mineral matter (ash).....	34.51 %	1.50 %
Non-bituminous organic matter .....	3.983 %	1.28 %
Portion of total bitumen soluble in alcohol	8.21 %	11.66 %
" " " " ether..	80.01 %	81.63 %
Loss at 212° F.....	0.65 %	1.37 %
" " " " 100° F. in 10 hours.....	7.98 %	17.80 %
" " " " on total bitumen.....	12.811 %	18.308 %
Evolution of sulphuretted hydrogen at...	410° F.   437° F.	
Softening point .....	166° F.	113° F.
Flowing point .....	192° F.	150° F.

The sample of Trinidad asphalt is rather better than the average (cf. Sixth Annual Report of Inspector of Asphalts and Cements, Washington. This Journal, 1893, 267).

2. *Mechanical Tests*.—Mixtures of the asphalts under examination were prepared by heating the asphalt and the appropriate quantity of oil to 300° F., and adding the mineral ingredients, which, like the moulds, rammers, &c., were all kept hot. The mixtures prepared in this manner were filled into cement briquette-moulds and kept for 24 hours at various temperatures. The mixtures were made up with the following proportions:—

	Trinidad.	Bermudez No. 1.	Bermudez No. 2.
Asphalt .....	12.5	9.1	9.1
" Oil residuum " (heavy petroleum oil) .....	2.5	1.0	1.0
Sand .....	58.0	75.0	60.0
Crushed stone .....	23.2	..	..
Powdered limestone .....	3.8	15.0	30.1
	100.0	100.1	100.1

The following is a record of the results of experiments with these mixtures:—

#### *Tensile Tests.*

	Trinidad.	Bermudez No. 1.	Bermudez No. 2.
	Lb. per Sq. In.	Lb. per Sq. In.	Lb. per Sq. In.
At 32° F.....	146.5	399.0	508.5
At 64° F.....	55.5	116.5	204.0
At 108° F.....	19.0	20.0	44.5

#### *Compression Tests.*

	Lb. per Sq. In.	Lb. per Sq. In.	Lb. per Sq. In.
At 32° F.....	1,027.5	1,750.0	1,750.0
At 65° F.....	517.5	..	667.5

The conclusions drawn from all these data may be summarised as follows:—

1. Bermudez asphalt is nearly pure bitumen, containing, moreover, a large proportion of "petrolene" (the portion soluble in a solvent such as petroleum ether). In these respects it is said to be better than Trinidad asphalt.

2. The small proportion of mineral matter in Bermudez asphalt makes it capable of yielding a larger amount of paving mixture than does an equal weight of Trinidad asphalt. The noteworthy amount of organic non-bituminous matter in Trinidad asphalt causes it to decay, especially in street gutters, more readily than Bermudez asphalt.

3. Mixtures made with Bermudez asphalt have a greater mechanical strength than corresponding mixtures containing Trinidad asphalt.—B. B.

*Natural Gas: its Composition and Origin. The Origin of Petroleum.* F. C. Phillips. Amer. Chem. Jour. **16**, [6], 406.

See under H., page 790.

*Acidimetric Estimation of Naphthalene, Acenaphthene,  $\alpha$ - and  $\beta$ -Naphthol, &c.* F. W. Küster. Ber. **27**, 1101.

See under XXIII., page 844.

*Liquid Fuel.* E. N. Henwood. J. Soc. Arts, **42**, 703.

See under H., page 792.

#### PATENT.

*Improved Process for the Extraction of Naphthalene and other Hydrocarbons from Coal-Tar and similar Substances.* F. W. Clark, South Norwood Hill, Surrey. Eng. Pat. 16,769, September 6, 1893.

COAL-TAR, or preferably the distillate obtained in the process of separating pitch from coal-tar, is mixed with naphtha or other light hydrocarbon and passed through an evaporator, which is maintained at a sufficiently high temperature to expel the solvent containing the naphthalene and other hydrocarbons. The naphtha is conveyed by a suitable carrier, such as air, into a condenser, the pitch remaining in the evaporator being drawn off into suitable vessels, whilst the naphtha containing the hydrocarbons is collected in other receptacles. When coal-gas is used as a carrier its illuminating power is increased, and instead of being passed through a condenser it may be conducted through mains to places where it is to be used for lighting purposes. The apparatus employed comprises an evaporator, wherein the liquid to be distilled runs through inclined tubes surrounded by a heating medium and a condenser, and means for circulating a carrier, such as air, through the evaporator and condenser.—D. B.

## IV.—COLOURING MATTERS AND DYES.

*Brasilin and Hæmatoxylin.* J. Herzig. Monatsh. Chem. **15**, 139—146.

By methylation brasilin furnishes a trimethyl derivative, which gives rise to mono-acetyl-trimethyl brasilin, when treated with acetic anhydride and sodium acetate. Tetramethyl brasilin can be similarly produced by heating the trimethyl ether with solid caustic alkali and methyl iodide in sealed tubes at  $130^{\circ}$ — $140^{\circ}$  C., or with alcoholic potash and methyl iodide for 7—8 hours in the water-bath. When purified it melts at  $137^{\circ}$ — $138^{\circ}$ , and cannot be made to yield an acetyl derivative.

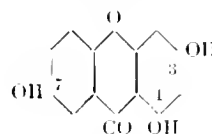
In similar fashion, hæmatoxylin furnishes a tetra-methyl derivative, which by acetylation gives rise to mono-acetyl-tetramethylhæmatoxylin, melting at  $178^{\circ}$ — $180^{\circ}$ , and by pushing the methylation process to the utmost, to pentamethylhæmatoxylin, melting at  $144^{\circ}$ — $147^{\circ}$ . In examining

products of this class, Zeisel's methoxyl process furnishes a convenient method of determining pretty sharply the number of methoxyl groups present.

Previous results by methylation go to show that both hæmatoxylin and brasilin behave quite similarly. Other similarities (alkyl derivatives) are observed also, which are only noticed in substances standing in close relationship to xanthone. Beyer has also shown that fluorescein and eosin, with respect to a hydroxyl group, behave exactly like the substances of the xanthone group, and R. Meyer has further pointed out that fluoran, the root substance of fluorescein, is a body of perfectly analogous constitution to xanthone. In future studies of brasilin and hæmatoxylin, the circumstance must, then, be ever kept in view, that in these substances we have compounds similarly constituted in all probability to xanthone or fluoran.—C. R. A. W.

*Synthesis of Gentisin.* St. V. Kostanceki and J. Tarabor. Monatsh. Chem. **15**, 1—8.

GENTISIN,  $C_{15}H_{10}O_4$ , the colouring matter of gentian root, loses methyl on boiling with hydriodic acid, forming *gentisein*,  $C_{14}H_{10}O_4$ , in which three hydroxyl groups are found to be present by acetylation: the parent body, gentisin, only containing two. On methylation, gentisin gives a mono-methyl ether, the same substance being formed by analogously treating gentisein, two methyl groups being introduced during the process; in either case the third hydroxyl group can be substituted by acetylation or benzylation, yielding the same product, whether gentisin or gentisein be the starting point. On fusion with caustic potash gentisein breaks up, forming phloroglucinol and hydroquinone carbonic acid, whence the constitution of gentisein must be that of the trihydroxyxanthone—



The reproduction of gentisein, which dyes upon alumina mordants, from these two decomposition products is somewhat difficult, most of the materials becoming carbonised; by carefully working with small quantities at a time, using acetic anhydride as water-withdrawing agent, the authors have succeeded in synthesising a sufficient quantity for identification. By careful methylation gentisein has been converted into a monomethyl ether, identical in all respects with gentisin; as yet the data extant are not sufficient to decide whether the methyl group thus introduced is situated at position No. 3 or No. 7, but it appears to be at one of the two.—C. R. A. W.

*Some Reactions of Orthotolidine.* H. Schiff and A. Ostro-govich. Annalen. **278**, 375—379.

WHEN benzidine dihydrochloride is boiled in dilute aqueous solution it is partially converted into monohydrochloride, which is sparingly soluble. This reaction does not take place with tolidine dihydrochloride, and the authors only succeeded in obtaining tolidine monohydrochloride by boiling equimolecular proportions of the dihydrochloride and of sodium acetate. In the case of the phenylene and tolylene diamines this reaction produces the hydrochlorides of the mono-acetyl derivatives of the bases. (H. Schiff, Ber. **25**, 2210.) The method which gave the best result was to dissolve 45 grms. of tolidine dihydrochloride in 120 cc. of water, mix this whilst hot with a hot solution of 13 grms. of anhydrous sodium acetate in 60 cc. of water, and boil for two hours, when the reaction becomes almost complete. The monohydrochloride is purified by recrystallisation from water, and forms colourless shining plates, which reddens in the air and are sparingly soluble in cold water. When heated to  $220^{\circ}$  C. the compound becomes coloured, and decomposes without melting above  $300^{\circ}$  C. The dihydrochloride decomposes without melting above  $340^{\circ}$  C. By boiling the monohydrochloride with acetic anhydride it is



converted into dihydrochloride and diacetylolidine, which melts at 304 C., corr. 311 C. Equimolecular proportions of the monohydrochloride and of sodium acetate give tolidine when boiled in aqueous solution. The authors were unable to obtain the hydrochloride of mono-acetylolidine, nor were they able to produce mono-acetylolidine by boiling tolidine with dilute acetic acid. The monohydrochloride treated in the cold with potassium cyanate gave tolidine, and CO<sub>2</sub> was evolved, but no urea derivative was formed. With chlorocarbonic ether the monohydrochloride gives dihydrochloride and tolidine diurethane, melting at 190 C. Compared with benzidine, tolidine behaves as a much weaker base, and with regard to the acetate and the monohydrochloride of the latter the authors assume that these salts are dissociated in aqueous solution. In support of this view as regards the hydrochloride they point out that on boiling its aqueous solution, hydrochloric acid is given off and tolidine crystallises out on cooling. As regards the solubilities of the mono- and dihydrochlorides, the latter dissolves in 17.34 times its weight of water at 12° C., whilst the former at the same temperature requires 112.4 times its weight. Hence the hydrochlorides of benzidine are about five times more soluble at ordinary temperatures than those of tolidine. The method of separating the monohydrochloride is proposed as a means of purifying tolidine, and attention is called to the fact that the solubility of this salt is less in brine than in pure water.

—T. A. L.

*Condensation of Aldehydes with α-Naphthaquinone and α-Naphthaquinol.* J. Wurgaft. J. prakt. Chem. [2], 49, 551—552.

WHEN a mixture of α-naphthaquinol (2 mols.) and benzaldehyde (1 mol.) is dissolved in glacial acetic acid and saturated in the cold with hydrochloric acid gas, the solution soon becomes dark red, and after 4 hours golden lustrous crystals separate. These on analysis are found to have the empirical formula C<sub>22</sub>H<sub>12</sub>ClO<sub>4</sub>·H<sub>2</sub>O. The same compound being obtained by the action of benzotrichloride on α-naphthaquinol in glacial acetic acid solution, it appears that the phenomena are similar to those observed by Doebner (Annalen, 217, 227) on the formation of aurin; the new compound may be regarded as *tetrahydroxydinaphthylphenylcarbin chloride*. By varying the conditions a non-chlorinated product of a similar colour may be obtained; it is the anhydride of a tetrahydroxydinaphthylphenylcarbinol, C<sub>20</sub>H<sub>12</sub>C(OH)<sub>4</sub>·[C<sub>10</sub>H<sub>5</sub>(OH)<sub>2</sub>]<sub>2</sub>. The acetyl derivative (found C = 76.25; H = 4.3 per cent.) melts at 246°, and is converted into the original colouring matter by acids; the acetyl derivative of the leuco base, which latter is very difficult to prepare, gave on analysis C = 79.24; H = 4.4 per cent.

Cuminal and nitrobenzaldehyde yield similar derivatives. α-Naphthaquinone reacts with benzaldehyde, forming a red dyestuff, the constitution of which is at present uncertain. It gave C = 77.0; H = 3.75; Cl = 5.83 per cent.

α-naphthaquinone does not react with benzotrichloride in glacial acetic acid solution.—A. R. L.

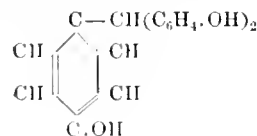
*Preparation of Quinoline.* J. Walter. J. prakt. Chem. [2], 49, 549—550.

ACCORDING to Skraup's instructions for preparing quinoline (Monatsh. 2, 141), nitrobenzene (24 grms.), aniline (38 grms.), and glycerol (120 grms.), are mixed with sulphuric acid (100 grms.) in a flask of 2 litres capacity, and the mixture heated therein on a sand-bath, a reflux condenser being attached. The large flask is rendered necessary because of the tendency of the reacting mixture to froth. The author finds, however, that when the nitrobenzene is heated to boiling in a globular flask connected with a reflux condenser and provided with a dropping funnel into which latter the mixture of glycerol, aniline, and sulphuric acid, previously heated on the water-bath, is introduced in portions and dropped therefrom into the boiling nitrobenzene, the frothing is entirely repressed, and

it is possible to work with double the quantities of reagents above mentioned in a flask of 800 cc. capacity. The addition of the mixture of aniline, glycerol and sulphuric acid to the nitrobenzene should extend over a period of  $\frac{3}{4}$  hour. If a large amount of material is being operated upon, it is best to prevent the separation of the liquid into layers by a mechanical mixing arrangement. When all the reagents have been mixed the boiling point is found to be 157°—160°; the whole is then maintained in gentle ebullition for two hours, after which the boiling point sinks to 138°—140° by reason of the formation of water. The unaltered nitrobenzene is then removed by passing a current of air through the liquid. In working on a technical scale enamelled iron boilers are used. The product is now made alkaline and distilled in a current of steam from a capacious cast-iron pot.—A. R. L.

*Aurin.* J. Herzig and Th. v. Smoluehowski. Monatsh. Chem. 15, 73—84.

AURIN and acetyl aurin have been examined by several experimenters with varying results. The authors conclude from their own observations that aurin is C<sub>19</sub>H<sub>14</sub>O<sub>3</sub>; but on acetylation the elements of water are taken up, so that acetylaurin is derived from C<sub>19</sub>H<sub>16</sub>O<sub>3</sub>. On saponification this reproduces C<sub>19</sub>H<sub>14</sub>O<sub>3</sub>. Acetyl aurin contains three acetyl groups, but no hydroxyl group; on reduction it gives rise to triacetyl leucaurin. Aurin, they regard as possessing the structure—



—C. R. A. W.

*Comparison between the Coloured and the Colourless Derivatives of Di- and Triphenylmethane.* A. Rosenstiehl. Bull. Chim. 1894, 11, 213.

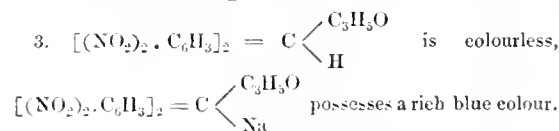
1. THE colourless leucaurine is converted into coloured magenta by the substitution of hydrogen by some electro-negative (acid) radical—



2. The compounds—



however, being both colourless, it follows that the amido groups of magenta must contribute essentially to the colouring of this compound. The assumption that this action of the amido group is due to its chemical function, as opposed to that of the electro-negative radicals, is thus borne out.



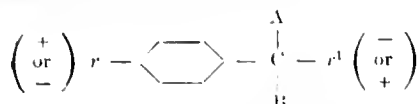
Further, trinitrotriphenylmethane is colourless; the sodium salt (NO<sub>2</sub>.C<sub>6</sub>H<sub>4</sub>)<sub>3</sub>.C—Na is coloured. Also in this case the opposite chemical functions of the nitro group and of the sodium, coincide with the appearance of colour in the compound.

4. In sulphonated colouring matters the free colour acid, in contradistinction to the previous observation (above), is coloured, and the alkaline salts are colourless. It appears therefore that the introduction of the sulpho group into the molecule suspends the basic character of the amido group present.

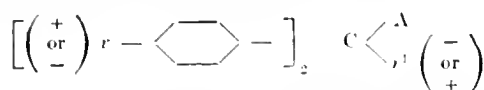
5. In magenta, as in all coloured compounds of the same order, the amido groups occupy the para-position towards the methane carbon atom. Only two of the three phenyl

groups require the amido group in para-position. The third may even contain no amido group at all. One amido group is, however, already sufficient to produce the chemical antagonism required for the formation of coloured hydrochlorides, although only those colouring matters are of practical interest which possess at least two phenyl groups substituted in the para position.

5. In order to convert triphenylmethane into colouring matters, at least one of the phenyl groups must be substituted in the para position by either  $\text{NH}_2$ ,  $\text{OH}$ , or  $\text{NO}_2$ , and the methane carbon atom must be in combination with a radical of respectively opposite chemical functions. This may be expressed by the formula—



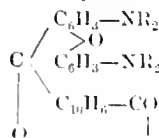
in which A and B may be phenyl groups substituted in the para position or not,  $r$  and  $r'$  representing radicals of chemically opposite functions. With two para-substituted phenyl groups the formula becomes—



7. In amido derivatives of this class, influences weakening the antagonism between  $\text{NH}_2$  and  $\text{Cl}$  also weaken the dyeing properties of the compounds. For this reason excess of acid or base weakens the colouring power of these dyes, whether sulphonated or not.—C. O. W.

*Naphthalene of Substituted Amidophenols.* F. Ulzer and A. Höller. Mitt. k. k. Tech. Gew. Museums, 4, 1894, 179—181.

One molecule of naphthalic acid and two molecules of dimethyl-*m*-amidophenol were mixed with an equal weight of anhydrous zinc chloride, and heated for from four to five hours in an iron crucible at from 190° to 210° C. The melt was boiled with water to extract the zinc chloride, the residue collected on a filter and washed with boiling water. It possessed a high metallic lustre after being dried. The substance so obtained was treated with dilute ammonia, the solution filtered, and the residue dissolved in hot 50 per cent. alcohol, with addition of some hydrochloric acid. On cooling, some resinous matter separated out, from which the solution was poured off. The latter was then evaporated until the alcohol had almost entirely been expelled, when, on cooling, the solution formed a pulpy mass, on the surface of which, on re-heating, the colouring matter separated out in the form of a semi-solid cake. This was separated from the mother liquor, which, on further concentration, yielded some more colouring matter, but of inferior purity. The dyestuff thus obtained is purified by precipitating its aqueous solution with caustic soda, shaking the precipitate with ether, and precipitating the base from its ethereal solution in the form of the hydrochloride by shaking it with hydrochloric acid. Diethyl-*m*-amidophenol yields a similar dye as dimethyl-*m*-amidophenol, but the yields are very unsatisfactory. The fact of naphthalic acid producing rhodamines is a further proof that dibasic acids, the carboxyl groups of which are in peri-position (1:8), are capable of the same reactions as orthodicarboxylic acids. The formula of the above-described colouring matter is therefore analogous to that of ordinary Rhodamine.



—C. O. W.

*The Action of Light upon Dyed Colours.* Rep. of the Committee (T. L. Thorpe, J. J. Hummel, W. H. Perkin, W. J. Russell, W. de W. Abney, W. Stroud, and R. Meldola). Drawn up by J. J. Hummel. Brit. Assoc. Sect. B. Oxford, 1894.

See under A VI., page 803.

#### PATENTS.

*Improvements in the Production of Colouring Matters from Meta-amidophenolphthalenes.* O. Imray, London. From "The Farbwerke vormals Meister, Lucius, and Brüning," Hoechst-on-the-Maine, Germany. Eng. Pat. 11,207, July 22, 1893.

Red colouring matters soluble in water are produced by condensing alkylated *m*-amidophenolphthalenes with secondary and tertiary bases, such as mono-methyl- and mono-ethyl-aniline, mono-ethyl-*o*-toluidine, methylphenylamine, ethylbenzylamine, dimethyl- or diethyl-aniline, and the shades produced from the tetra-alkylated *m*-amidophenolphthalenes are bluer than those from the dialkylated derivatives. The condensation is effected by means of phosphorus oxychloride, and the following example gives the quantities employed. About 15 to 20 kilos. of ethylbenzylamine are mixed with 10 kilos. of tetra-ethyl-*m*-amidophenolphthalene and 1 kilo. of phosphorus oxychloride are gradually added. The reaction is somewhat violent, and in order to control it, the melt is kept at 60—80° C. for an hour, then dissolved in dilute hydrochloric acid, and the colouring matter is precipitated from the filtered solution with salt. It forms when pure a dark red-brown powder easily soluble in water, alcohol, and glacial acetic acid, and dyes cotton mordanted with tannin in shades fast to soap. In order to obtain the condensation products from primary amines it is only necessary to boil the components together. Obtained in this way the compound from tetra-ethyl-*m*-amidophenolphthalene and aniline melts at 222° C., from *o*-toluidine at 179° C., and from *p*-toluidine at 196° C. These are almost colourless substances, and are precipitated from their solutions in mineral acids by sodium acetate.—T. A. L.

*Improvements in the Production of Green and Bluish-Green Colouring Matters.* O. Imray, London. From "The Farbwerke vormals Meister, Lucius, and Brüning," Hoechst-on-the-Maine, Germany. Eng. Pat. 11,671, July 31, 1893.

This is an extension of Eng. Pat. 12,796 of 1888 (this Journal, 1889, 701), and claims the preparation of colouring matters of the Patent blue series. These are the sulphonic acids of *m*-hydroxydibenzylidiamidotriphenyl carbinol, *m*-hydroxydibenzylidiamido-di-*o*-tolylphenyl carbinol, and *m*-hydroxy-tetrabenzylidiamidotriphenyl carbinol, which are obtained according to the process described in the above-mentioned patent from symmetrical *m*-amidodibenzylidiamidotriphenylmethane, symmetrical *m*-amidodibenzylidiamido-*o*-tolylphenylmethane, and *m*-amidotetrabenzylidiamidotriphenylmethane, or from benzylamine, benzyl-*o*-toluidine, and dibenzylamine, by sulphonating the hydroxy-leuco bases obtained therefrom and oxidising the leuco-sulphonic acids. An alternative method is to condense *m*-hydroxybenzaldehyde with the sulphonic acids of benzylamine, benzyl-*o*-toluidine, or of dibenzylamine, and after sulphonating, to oxidise the leuco-sulphonic acids thus produced to the blue colouring matters (see also this Journal, 1890, 286).—T. A. L.

*The Manufacture or Production of Dye-stuffs.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 15,045, August 5, 1893.

These colouring matters, which belong to the class of the oxazines, are obtained by condensing the nitroso-derivatives of secondary and tertiary amines with acetylated or benzoylated (1,2)-amido-naphthols, and subsequently removing the acetyl group or not, as may be desired. A solution of 20.1 kilos. of (1,2)-acetanidonaphthol and 57 kilos. of

nitrosodimethylaniline hydrochloride in 300 kilos. of alcohol or acetic acid is heated to 60–100° C. When the nitroso-compound has disappeared the melt is poured into water and the dyestuff salted out. It dissolves to a blue solution in water, and dyes a reddish-blue on cotton mordanted with tannin. If the acetyl-group be removed by boiling with acids a colouring matter is obtained which dyes mordanted cotton a greenish-blue, and in general the colouring matters produced according to this invention are of a redder shade when they contain an acid radicle than after saponification.

—T. A. L.

*Improvements in the Manufacture or Production of Naphthylamine-sulpho Acids.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 15,223, August 9, 1893.

By sulphonating with fuming sulphuric acid the two acids numbered II. and III. (i.e. (1.4.3')- and (1.4.2')-naphthylamine disulphonic acids) of Ger. Pat. 41,957, at a temperature of 50°–100° C., two new  $\alpha$ -naphthylamine trisulphonic acids are produced, the new sulphonic acid group entering the molecule ortho to the amido group. The same acids are produced from the  $\beta$  and  $\delta$  Clève acids: the former, which is (1.5')-naphthylamine sulphonic acid, giving (1.2.4.3')-naphthylamine trisulphonic acid; and the latter, which is (1.2')-naphthylamine sulphonic acid, yielding (1.2.4.2')-naphthylamine trisulphonic acid. The quantities employed are as follows:—One kilo. of Clève's  $\delta$  acid is stirred into 3.5 kilos. of fuming sulphuric acid containing 25 per cent. of anhydride, the melt being kept cool. The temperature is then raised to 50°–60° C. until a sample dissolves easily in water. After cooling, 1 kilo. of 70 per cent. anhydride is added and the melt heated to 80°–90° C. until a test gives no coloration with diazotised sulphanilic acid. The melt is then limed, the calcium salt converted into the neutral sodium salt, and the acid sodium salt is precipitated by adding hydrochloric acid. On treatment with a mixture of nitrous and nitric acids naphthol yellow S is formed.—T. A. L.

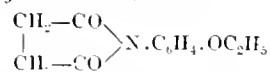
*The Manufacture or Production of Amido-naphthol-sulpho Acids and Colouring Matters therefrom.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 15,269, August 10, 1893.

The two amido naphthol sulphonic acids described, of which one is new, contain the amido and hydroxyl groups in the (1.1') or peri position. They are obtained by fusing with caustic alkalis the (1.4'.1')- and the (1.3'.1')-naphthylamine disulphonic acids. In the case of both these acid-sulphonic acid group in the peri position to the amido group is replaced by hydroxyl yielding (1.1'.4')-amido-naphthol sulphonic acid and (1.1'.3')-amido-naphthol sulphonic acid. Of these the former has already been described in Eng. Pat. 4613 of 1893 (this Journal, 1894, 244), and no claim is made to it or to the colouring matters therefrom. The starting point for the (1.1'.3')-acid is (1.1'.3')-naphthylamine disulphonic acid, which is prepared by sulphonating acetylated (1.1')-naphthylamine sulphonic acid, removing the acetyl group, and separating the (1.1'.3')-naphthylamine disulphonic acid from the isomeric (1.2.1')-naphthylamine disulphonic acid by the difference in solubility of the sodium salts, that of the latter acid being much less soluble. The constitution of the new (1.1'.3')-acid has been determined by removing the amido group by the hydrazine method and converting the disulphonic acid produced into its sulpho-chloride. The naphthalene disulphonic chloride so produced melts at 138° C., which is the melting point of the (1.3)-sulpho-chloride prepared by Armstrong and Wynne. As already mentioned, when the (1.1'.3')-naphthylamine disulphonic acid is fused with caustic potash it is converted into (1.1'.3')-amido-naphthol sulphonic acid, which can be used for the preparation of dyestuffs by combining it with various diazo and tetrazo compounds in the usual manner.

—T. A. L.

*Production of Para-ethoxy- and of Para-methoxy-phenyl succinimides.* O. Imray, London. From The Farbwerke vormals Meister, Lucius, and Brüning, Höchst a/Main, Germany. Eng. Pat. 15,327, August 11, 1893.

*p-Ethoxy-phenyl-succinimide.*—



prepared by heating together in molecular proportions, succinic acid, and (a) *p*-amido-phenetol hydrochloride, or (b) *p*-acet-amido-phenetol, and subsequently recrystallising the product from hot alcohol. It forms colourless or slightly yellow prismatic needles which fuse at 155° C.

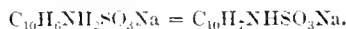
*p-Methoxyphenyl-succinimide.*—This substance resembles the preceding, and is prepared in a similar manner from *p*-amido-anisol, or acet-*p*-amido-anisol. Melting point, 165° C.—H. T. P.

*Improvements in the Production of Iodo-derivatives of the Succinimide and of Substituted Succinimides.* O. Imray, London. From The Farbwerke vormals Meister, Lucius, and Brüning, Höchst a/Main, Germany. Eng. Pat. 15,328, August 11, 1893.

The process in general consists in adding a strong solution of iodine and potassium iodide to the succinimide, dissolved in warm glacial acetic acid. On cooling, the iodo-derivative separates out in the crystalline state. The iodo-derivatives of succinimide, and of *p*-ethoxy- and *p*-methoxy-phenyl-succinimide, are described.—H. T. P.

*Preparation of a New  $\beta$ -naphthylaminesulphonic Acid and of  $\beta$ -naphthylamine, and the Preparation of Salts of  $\beta$ -naphthylsulphamic Acid from the former.* G. Tobias, Berlin. Eng. Pat. 15,404, August 12, 1893.

By the action of sulphuric acid on  $\beta$ -naphthol, the product of the reaction is not, according to the inventor,  $\beta$ -naphthyl sulphate, but (2.1)- $\beta$ -naphthol sulphonic acid. By heating it under pressure with ammonia it is converted into  $\beta$ -naphthylamine sulphonic acid, which on further heating yields  $\beta$ -naphthylamine. When the salts of  $\beta$ -naphthylamine sulphonic acid are heated they are converted into the corresponding salts of  $\beta$ -naphthyl sulphamic acid which are decomposed by mineral acids yielding  $\beta$ -naphthylamine. As to the properties of the (2.1)- $\beta$ -naphthol sulphonic acid, the sodium salt dissolves easily in 80 per cent. alcohol or in water, whilst the potassium salt forms sparingly soluble scales. Both salts are stable when heated in neutral solutions, but the acid is rapidly decomposed when an excess of a mineral acid is present. A neutral solution of the acid gives an indigo-blue coloration with ferric chloride. By the action of diazobenzene sulphonic acid on a strong solution of the sodium salt in presence of sodium carbonate, the sulphonic acid group is eliminated, and there results sulpho-benzene-azo- $\beta$ -naphthol. When the sodium salt is heated with 4–5 times its weight of a 20 per cent. ammonia solution for 10–20 hours to 220°–230° C.,  $\beta$ -naphthylamine sodium sulphonate is produced, together with a little  $\beta$ -naphthylamine. The free acid crystallises in scales or needles, and the sodium salt also separates from water or can be precipitated by means of salt. It forms a yellow crystalline diazo compound which on boiling with dilute sulphuric acid yields  $\beta$ -naphthol sulphonic acid, and on conversion into dichloro-naphthalene it yields the 1.2 isomer, melting at 34° C. When the sodium salt is heated to 230° C. the following conversion takes place:—



This salt is soluble in water, and can be precipitated by adding salt. Mineral acids decompose it, forming  $\beta$ -naphthylamine. It is claimed for the  $\beta$ -naphthylamine made in this way, that it is free from dinaphthylamine and from naphthol.—T. A. L.

*The Production of New Acetnaphthylene-diamine-sulphonic Acids and of Colouring Matters derived therefrom*. S. Pitt, Sutton. From L. Cassella and Co., Frankfurt-on-the-Maine, Germany. Eng. Pat. 15,411, August 14, 1893.

THE acids are obtained by nitrating and reducing the acetyl derivatives of (1.3')- and (1.2')-naphthylamine sulphonic acids, when derivatives of (1.4)-naphthylene diamine are obtained which can be employed for the manufacture of dyestuffs by combining them with diazo or tetrazo compounds. Other dyestuffs are obtained by diazotising the acetyl compound and combining this with a diazotisable amine such as  $\alpha$ -naphthylamine or  $\gamma$ -amido-naphthol sulphonic acid, rediazotising, combining with an amine or phenol, and finally saponifying the product. The acetylation of the naphthylamine sulphonic acid is carried out by heating the sodium salt with glacial acetic acid, and the crude product thus obtained is nitrated in sulphuric acid by adding nitro-sulphuric acid. On diluting with water and adding salt the sodium salts of the nitro acids are precipitated, that from the (1.2')-acid crystallising in needles, whilst the (1.3')-acid crystallises in rhombic plates. Both are easily saponified by mineral acids and the (1.4.2' or 3')-nitro-naphthylamine sulphonic acids obtained dye wool a greenish-yellow from an acid bath. In order to reduce the acids they are gradually added to a boiling mixture of iron-borings, water, and acetic acid. When the solution has become colourless the dissolved iron is precipitated with soda and the new acids are precipitated from the filtrate by adding hydrochloric acid. The acids will combine with diazo compounds, or the acids themselves may be diazotised and combined with phenols and amines. In the latter case, if a diazotisable amine be employed and the azo compound be first saponified, a tetrazo compound is produced when treated with nitrous acid. The following example gives the quantities employed for the formation of one of the dyestuffs referred to in the patent. The diazo compound obtained from 30.2 kilos. of (1.4.3')-amido acetnaphthalide sulphonic acid by means of 7 kilos. of sodium nitrite and 24 kilos. of hydrochloric acid, is added to an alkaline solution of (1.4)-naphthol sodium sulphonate. The dyestuff which separates is filtered off and boiled with 500 litres of 10 per cent. soda-lye until a sample mixed with acetic acid gives a bluish-violet colour. After adding hydrochloric acid and cooling to 0° C., it is rediazotised by the addition of 7 kilos. of sodium nitrite, and the sparingly soluble brown diazo compound is added to an alkaline solution of 38 kilos. of  $\beta$ -naphthol disulphonic acid B. The colouring matter which separates, dyes wool a deep blue from an acid bath.—T. A. L.

*The Manufacture and Production of New Acid Colouring Matters*. B. Willeox, London. From "The Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 16,370, August 30, 1893.

THIS is an extension of Eng. Pat. 3487 of 1893 (this Journal, 1894, 115), referring to the production of acid dyestuffs by condensing the nitroso derivatives of dialkylated  $m$ -amido-phenol with  $\alpha$ -naphthylamine mono- and disulphonic acids. Similar dyestuffs can be obtained from the sulphonic acids of aniline, toluidine, and  $\beta$ -naphthylamine, especially when condensed with diethyl- and dimethyl- $m$ -amido-phenol. The acids most suitable for the purpose are,  $p$ - and  $m$ -sulphanilic acid, aniline disulphonic acid,  $o$ -toluidine- $m$ -sulphonic acid, the so-called  $\alpha$ -,  $\beta$ -,  $\gamma$ - and  $\delta$ - $\beta$ -naphthylamine mono-sulphonic acids,  $\beta$ -naphthylamine disulphonic acid G, and (2.4'.2')- $\beta$ -naphthylamine disulphonic acid. About 69 kilos. of nitroso-diethyl- $m$ -amido-phenol, 50 kilos. of crystallised sodium sulphanilate and 6 kilos. of 30 per cent. acetic acid, are heated on the water-bath for about 12 hours. The melt is then diluted with 1,000 litres of hot water, alkali is added to neutralise the greater part of the acid, and the dye is precipitated with salt. After filtering and washing with brine it is pressed and dried. It dyes wool from an acid bath, giving bluish-violet shades fast to light and alkalis and dyes on very evenly.—T. A. L.

*The Manufacture or Production of Condensation Products from Anines, or Derivatives thereof, by Oxidation in Alkaline Solution*. H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 16,708, September 5, 1893.

ACCORDING to Eng. Pat. 19,061 of 1891 (this Journal, 1892, 809), certain yellow dyestuffs can be obtained by treating dehydrothio- $p$ -toluidine sulphonic acid and its homologues with alkaline oxidising agents, resulting in the formation of azo or azoxy compounds. Certain other amines react in the same way, and the present invention describes the preparation of azo and azoxy compounds of  $o$ -,  $m$ -, and  $p$ -nitraniline and of their homologues, of benzidine sulphonic acid and its homologues, and of diamido-stilbene disulphonic acid. Finely-divided  $p$ -nitraniline is mixed with an excess of sodium hypochlorite and dilute caustic soda, and the mixture is allowed to stand until on treatment with nitrite and acid very little colouring matter is formed with naphthol. The product is then filtered off and freed from unaltered  $p$ -nitraniline by washing with dilute hydrochloric acid. The compound is scarcely soluble in water and only slightly in alcohol, but dissolves easily in acetone and chloroform, and crystallises from the latter in reddish-brown crystals with a bluish reflex, melting at 221° C. On reduction with ammonium sulphide it forms a diamido compound which, after diazotisation, combines with amines and phenols, giving substantive azo colouring matters. When completely reduced it yields  $p$ -phenylene diamine. By applying the same process to benzidine  $m$ -mono-sulphonic acid, a product having the constitution—



is obtained. It dyes unmordanted cotton yellowish-orange and can be diazotised on the fibre and combined with amines and phenols. Diamido-stilbene disulphonic acid behaves similarly to alkaline oxidising agents, and the product obtained also gives yellowish orange shades on cotton. The oxidising agents mentioned are hypochlorites or ammonium persulphate, hypobromites, potassium permanganate, or potassium ferriyanide.—T. A. L.

*The Manufacture or Production of Colouring Matters and Materials therefor*. H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 16,780, September 6, 1893.

THIS is an extension of Eng. Pat. 8898 of 1893 (this Journal, 1894, 511), which referred to the production of di-alkylated naphthalene diamines by heating  $o$ - or  $\beta$ -naphthylamine sulphonic acids (more especially those containing the substituting groups in the meta position) with a primary amine and one of its salts, with or without the addition of benzoic acid. It has now been discovered that certain  $\alpha$ - and  $\beta$ -naphthol mono-, di-, and tri-sulphonic acids will react in the same way, giving di-alkylated naphthylene diamines or their sulphonic acids. Thus the (1.3)-diphenyl-naphthylene diamine described in Eng. Pat. 8898 of 1893 can also be obtained by mixing 1 kilo. of (1.3)- $\alpha$ -naphthol sodium sulphonate with 4 kilos. of aniline and 1 kilo. of aniline hydrochloride, and heating the melt to 150°–170° C. for about four hours, after which it is poured into dilute hydrochloric acid, when the diphenyl-naphthylene diamine separates out, and can be filtered off. The naphthol sulphonic acids suitable for this purpose are the following: (1.3)  $\text{C}_{10}\text{H}_7\text{OH}(\text{SO}_3\text{H})$ , of Ger. Pat. 37,910; (1.3.3') and (1.3.2')  $\text{C}_{10}\text{H}_6\text{OH}(\text{SO}_3\text{H})_2$ , of Ger. Pat. 38,281; (1.3.1')  $\text{C}_{10}\text{H}_5\text{OH}(\text{SO}_3\text{H})_3$ , of Ger. Pat. 45,776; (1.3.3'.1')  $\text{C}_{10}\text{H}_4\text{OH}(\text{SO}_3\text{H})_4$ , of Ger. Pat. 56,058; (2.4.1')  $\text{C}_{10}\text{H}_7\text{OH}(\text{SO}_3\text{H})$ , the acid C, of Ger. Pat. 65,997; and the hitherto unknown (2.4.2')  $\text{C}_{10}\text{H}_6\text{OH}(\text{SO}_3\text{H})_2$ , obtained by boiling the diazo compound or the corresponding  $\beta$ -naphthylamine disulphonic acid with water. The di-alkylated naphthylene diamines and their sulphonic acids produced as above are to be employed for the manufacture of dyestuffs by condensing them with

the nitroso derivatives of secondary or tertiary aromatic amines in a similar manner to that already described in Eng. Pats. 8898 and 11,892 of 1893 (this Journal, 1894, 511 and 631). The nitroso compounds can be replaced by other bodies which act in a similar manner, such as quinone dichloride and its analogues, and also certain azo compounds. Or, in place of these, alkylated *p*-diamines or their sulphonic acids may be oxidised together with the diphenyl-, di-*o*-tolyl-, or di-*p*-tolyl-(1,3,4')-naphthylene diamine sulphonic acid in presence of a suitable solvent by means of an oxidiser, such as ferric chloride, potassium dichromate, manganese dioxide, or the like.—T. A. L.

*The Manufacture and Production of New Colouring Matters and Leuco Compounds related to the Indigo Series, and of New Materials therefor.* B. Wilcox, London. From "The Badische Anilin and Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 17,472, September 16, 1893.

THE colouring-matters referred to in this patent are greenish-blue dye-stuffs which are carboxylated indigo derivatives. The leuco-compounds are obtained by fusing phenyl glycecol dicarboxylic acid with caustic alkalis. This new acid is produced by interacting with (1,2,3) amido-phthalic acid on chloro-acetic acid. The leuco-compound, when oxidised, gives a carboxylated indigo, which can be used as a dye, or sulphonated by treatment with sulphuric acid. About 125 kilos. of the sodium salt of (1,2,3) amidophthalic acid, and 58.5 kilos. of calcined soda are dissolved in 200 litres of water, then mixed with 120 kilos. of chloro-acetic acid, and the whole evaporated on the water-bath. The sodium salt of the phenyl-glycecol dicarboxylic acid so obtained is a syrup, which gradually solidifies. It is readily soluble in water, and dyes wool yellow from an acetic acid bath. In order to convert it into the leuco-compound, 1 kilo. is quickly added to 4 kilos. of fused caustic potash at 250° to 260° C., and kept at this temperature until the brown colour of the melt no longer increases in intensity. The melt, after cooling, is dissolved in 60 litres of cold water, and the leuco-compound may be precipitated by adding hydrochloric acid, but it is preferable to separate the colouring-matter directly, by adding ferric chloride. The dyestuff so formed is filtered off, and purified by dissolving in ammonia and precipitating with a mineral acid. In order to produce a sulphonic acid of this compound, 1 kilo. is dissolved in about 10 kilos. of fuming sulphuric acid, containing about 12 per cent. of anhydride. The melt is kept at about 65° C. for five hours, or until a sample is completely soluble in hot water. The melt is then poured into 100 litres of water, when part of the dyestuff separates, the rest being precipitated by adding salt. It is purified by dissolving in sodium acetate, and re-precipitating with a mineral acid. The sulphonic acid thus produced resembles indigo-carmin, but dyes wool from an acid-bath, giving rather greener shades. Still greener shades can be obtained by more energetic sulphonation by the use of sulphuric acid containing a higher percentage of anhydride.—T. A. L.

*Manufacture of New Violet, Blue, Grey, and Black Substantive Colouring Matters for Dyeing Cotton.* J. C. L. Durand, D. E. Huguenin, and A. J. J. d'Andiran, Basle, Switzerland. Eng. Pat. 9806, May 19, 1894.

THIS is an extension of Eng. Pat. 8511 of 1893 (this Journal 1893, 671), and also of Eng. Pat. 12,704 of 1893 (this Journal 1893, 824), describing the preparation of mixed tetrazo compounds from the unsymmetrical bases described in the second patent. Different colouring matters are produced according to the order in which the combination takes place, so that, whereas the colouring matter obtained by converting formaldehyde-dianisidine-aniline into its tetrazo compound, and combining it first with (1,4)-naphthylamine sulphonic acid, and secondly with (1,4)-naphthyl sulphonic acid dyes cotton a bluish violet shade, that obtained by carrying out the combination in the reverse order, gives reddish violet shades on unmordanted cotton.

Another series of colouring matters is produced by combining the tetrazo compounds of the symmetrical or unsymmetrical formaldehyde bases with (1,3,3',1')-amido-naphthol disulphonic acid or with (2,3',1') amido-naphthol sulphonic acid. The resulting colouring matters which dye cotton grey and bluish grey can be further diazotised and combined with amines and phenols and their sulphonic acids giving very dark shades. Thus the tetrazo compound from 3,4 kilos. of formaldehyde-tolidine-*o*-amido-phenol when combined with 4.8 kilos. of (2,3',1') amido-naphthol sulphonic acid in presence of sodium carbonate gives a colouring matter which dyes unmordanted cotton grey. After diazotisation and combination with *m*-phenylene diamine, the colouring matter dyes cotton a greyish black which, when dyed on 5 per cent. gives practically a black. (See also Eng. Pats. 20,301 and 20,790 of 1893, this Journal 1894, 32 and 146).—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Agave Americana Fibre.* J. Soc. Arts, 42, 702.

*Agave americana* grows in all soils in the Coimbatore district of the Madras presidency. This district has a mean temperature of 78°, with maximum and minimum temperatures of 97° and 64° respectively, and the annual rainfall is 21 inches, half of which falls during the monsoon (October to December).

The plant must be freely exposed to the sun, shade rendering it unhealthy.

The fibre is extracted, either by scraping or by maceration from the old green outwardly-drooping leaves of plants six or seven years old.

*Scraping process.*—Here, the spines, and about six inches of the apex of the leaf having been cut off, the leaf is split longitudinally into four or five pieces, which are next beaten with a mallet.

The "pulp" is then removed by a bamboo scraper and the fibre dried in the sun. This fibre, without either working or bleaching, is very clean, but the staple is not long. It is made into thread, which is used in weaving grass mats.

*Maceration process.*—The leaf is trimmed as before but not split. It is then beaten with the mallet and thrown, in bundles, into tanks, where it is left for 14–20 days, or until the pulp is quite decomposed. It is then taken out and exposed to the sun. The fibre so obtained is longer than, but not nearly so clean as that obtained by scraping.

If a demand arose, there would be a large supply, and then the present price of 2 annas per 1 lb. would no doubt be reduced.—E. R. B.

## PATENTS.

*An Improved Fabric for Garments.* F. Linneborn, Hagen, Germany. Eng. Pat. 12,390, June 23, 1893.

THE improvement claimed consists in knitting or manufacturing the fabric in two layers, the one of linen and the other of wool, flaked together, so that one side is entirely of linen and the other of wool. The layer of linen lying next to the skin keeps it cool, perspiration passing through the interstices between the linen fibres, because these do not absorb it, into the wool fibres, which first receive it and then allow it to evaporate immediately. In this way all drying of perspiration on the skin is avoided.

—E. G. P. T.

*Improvements in Apparatus or Machinery for Washing Wool or other Fibres.* J. Dawson, Rochdale. Eng. Pat. 13,874, July 18, 1893.

AN improved scouring apparatus is invented, in which the fibres are passed through the liquor by means of rakes,

forks, or prongs, mounted upon endless chains or bands, and the special object is to vary or regulate the angle of the rakes to the chains, particularly at the delivery end of the apparatus. To clean the bottom and sides of the wash bowl, a brush is employed, approximately of the size or shape of a cross-section of the bowl, and secured to one of the rakes.—E. G. P. T.

*Improvements in or applicable to Machinery for Securing and Washing of Wool and other Fibrous Materials.*  
J. and W. McNaught, Rochdale. Eng. Pat. 15,082, August 5, 1893.

An invention relating to improvements upon machines described in a former specification, Eng. Pat. 983, 1882, and involving the use of plates, grids, or sieves termed "immersers," which have perforated bottoms and turned-up sides and are made to sink into and rise out of the liquor in the trough in which the fibres operated upon are scoured.—E. G. P. T.

*Improved Treatment of Textile Fabrics for Stiffening same, and Production of Material suitable for Use as Tracing Cloth.* E. J. Goodwin, Castleton. Eng. Pat. 16,698, September 5, 1893.

The process of treating a textile fabric having a close texture, with sulphuric acid of 115 Tw. at a temperature of 64° to 65° F., as described in Eng. Pat. 22,566, 1892, is now extended to loose fabrics such as lace, and is found to impart to them a stiffness and finish that renders them suitable for use after washing and drying without the necessity of starching. Sheet material of a translucent character, suitable for tracing purposes, can also be produced by treating a finely woven cloth, such as lawn or muslin, with dilute sulphuric acid of 128—132 Tw. at a temperature varying between 64 and 72° F.—E. G. P. T.

## VI.—DYEING, CALICO PRINTING, PAPER-STAINING, AND BLEACHING.

*The Action of Light upon Dyed Colours.* Rep. of the Committee (T. E. Thorpe, J. J. Hummel, W. H. Perkin, W. J. Russell, W. de W. Abney, W. Stroud, and R. Meldola). Drawn up by J. J. Hummel. Brit. Assoc. Sect. B. Oxford. 1894.

DURING the past year the work of this committee has been continued, and a large number of wool and silk patterns, dyed with various natural and artificial orange and yellow colouring matters, have been examined with respect to their power of resisting the fading action of light.

The general method of preparing the dyed patterns, and the manner of exposing them under glass, with free excess of air and moisture, were the same as already adopted.

The patterns were exposed at Adel, near Leeds, in the grounds of James A. Hirst, Esq., to whom the best thanks of the Committee are again due for his kind permission.

Each dyed pattern was divided into six pieces, one of which was protected from the action of light, while the others were exposed for different periods of time. These "periods of exposure" were made equivalent to those adopted last year, by exposing, along with the patterns, special series of "standards" dyed with the same colouring matters as were then selected for this purpose. The standards were allowed to fade to the same extent as those which marked off the "fading period" of last year, before being renewed or removing a set of dyed patterns from the action of light. The patterns exposed during the past year are therefore comparable, in respect of the amount of fading which they have experienced, with the red dyes already reported upon.

The patterns were all put out for exposure on June 8, 1893, certain sets being subsequently removed on the following dates: July 1, July 31, August 26, 1893; February 19, June 12, 1894. Of the five "periods of exposure" thus marked off, periods 1, 2, 3 were equivalent to each other in fading power, whereas periods 4 and 5 were each equivalent to four of the first period in this respect; hence five patterns of each colour have been submitted respectively to an amount of fading equal to 1, 2, 3, 7, and 11 times that of the first "fading period" selected—viz. June 8 to July 1, 1893.

The dyed and faded patterns have again been entered in pattern-card books in such a manner that they can be readily compared with each other.

The following tables give the general result of the exposure experiments made during the year 1893-94, the colours being divided, according to their behaviour towards light, into the following five classes: very fugitive, fugitive, moderately fast, fast, very fast.

The initial numbers refer to the order of the patterns in the pattern-books. The S. and J. numbers refer to Schütz and Julius's "Tabellarische Uebersicht der künstlichen organischen Farbstoffen."

The colours marked thus (\*) appear to be somewhat faster than the rest of the class in which they are placed.

In the case of colouring matters requiring mordants, the particular mordant employed is indicated in brackets after the name of the dye-stuff.

### CLASS I. VERY FUGITIVE COLOURS. (Wool.)

The colours of this class have faded so rapidly that at the end of the first "fading period" (June 8 to July 1, 1893) only a very faint colour remains, or it has become very materially altered in hue. At the end of the fifth period (one year) all traces of the original colour have disappeared, the woollen cloth being either quite white or merely of a yellowish or brownish tint.

#### Nitro Colours.

Wool Book III.

- |               |     |  |
|---------------|-----|--|
| Acid Yellows. | 9.  | Aurantia. Ammonium salt of hexanitro-diphenylamine. S. and J. 14.                                      |
| "             | 32. | *Brilliant Yellow. Sodium salt of dinitro- $\alpha$ -naphthol- $\alpha$ -sulphonic acid. S. and J. 12. |
| "             | 37. | *Naphthol Yellow. Sodium salt of dinitro- $\sigma$ -naphthol. S. and J. 9.                             |
| "             | 38. | *Naphthol Yellow S. Sodium salt of dinitro- $\alpha$ -naphthol- $\beta$ -sulphonic acid. S. and J. 11. |
| "             | 43. | Picric acid. Tri-nitro-phenol. S. and J. 1.  |

#### Azo Colours.

Wool Book IV.

- |                  |     |   |
|------------------|-----|---|
| Mordant Colours. | 14. | Wool Yellow (Cr.). From azo derivative of aniline and maelurin. S. and J. 32. |
|------------------|-----|---|

Wool Book III.

- |                        |     |  |
|------------------------|-----|--|
| Basic Yellows.         | 4.  | Chrysoidine. From aniline and <i>m</i> -phenylene diamine. S. and J. 21.   |
| Direct Cotton Colours. | 1.  | Terra Cotta F. From primulin and <i>m</i> -phenylene-diamine-azo-naphthionic acid.   |
| "                      | 9.  | Direct Orange RR. Constitution not published.  |
| "                      | 26. | Thiazol Yellow. From azo derivative of dehydro-thio-toluidine-sulphonic acid, and dehydro-thio-toluidine-sulphonic acid. S. and J. 98. |
| "                      | 28. | Mimosa Yellow. From azo derivative of primulin, and ammonia.   |
| "                      | 30. | Direct Yellow TS. Constitution not published.  |
| "                      | 36. | Direct Orange R. Constitution not published.   |
| "                      | 39. | Direct Yellow A-C. Constitution not published.   |

*Diphenylmethane Colours.*

- Wool Book III.  
Basic Colours. 7. Auramine. Imido-tetra-methyl-diamido-diphenyl-methane-hydrochloride. S. and J. 260.

*Triphenylmethane Colours.*

- Acid Colours. 48. Trauin A. Sodium salt of fluorecein. S. and J. 315.

*Quinoline Colours.*

- Basic Colours. 42. Quinoline Yellow (sol. in spirit). Quino-phthalone. S. and J. 378.

*Acridine Colours.*

1. Acridine Orange R. extra. Zinc salt of tetra-methyl-diamido-phenyl-acridine.  
2. Acridine Orange NO. Zinc salt of tetra-methyl-diamido-acridine. S. and J. 381.  
3. Phosphine. Diamido-phenyl-acridine nitrate. S. and J. 382.  
6. Benzoflavine. Diamido-phenyl-dimethyl-acridine hydrochloride. S. and J. 383.

*Thiobenzoyl Colours.*

- Basic Colours. 9. Thioflavin T. Dimethyl-dehydro-thio-toluidine-methyl chloride. S. and J. 384.  
Acid Colour. 29. Thioflavin S. Sodium salt of dimethyl-dehydro-thio-toluidine-sulphonic acid. S. and J. 385.  
Direct Cotton 31. Primulin. Sulphonated product of the interaction of sulphur and *p*-toluidine. S. and J. 386.

*Natural Colouring Matters.*

- Non-mordant Colours. 1. Annatto. Pulp from fruit of *Bixa orellana*.  
2. Saffron. Stigmata of the flower of *Crocus sativus*.  
3. Turmeric. Rhizome of *Curcuma tinctoria*.  
Mordant Colours. 1. Young Fustic (Al.). Wood of *Rhus cotinus*.  
8. Tesu (Al.) Flowers of *Butea frondosa*.

NOTES.—Certain of the nitro colours show extreme sensitiveness to light by rapidly altering in hue. During the first "period of exposure," the rich red-orange colour of Aurantia, for example, soon changes to brown, and the pure lemon-yellow of Pieric Acid changes to orange-yellow; in both cases these altered colours fade slowly without any further change in hue, and might almost be placed among the "moderately fast colours." Brilliant Yellow, Martins Yellow, and Naphthol Yellow S behave somewhat like Pieric Acid, but the alteration in hue is much less pronounced. Thiazol Yellow, Mimosa Yellow, Thioflavin T and S, and Primulin all fade rapidly during the first "period of exposure" to a yellow-buff, which then appears to be "moderately fast."

## CLASS II. FUGITIVE COLOURS. (WOOL.)

The colours of this class show very marked fading at the end of the second "fading period" (July 1 to July 31, 1893), and after a year's exposure they have entirely faded, or only a tint remains.

*Azo Colours.*

- Wool Book III.  
Acid Colours. 1. Orange R. From xylydine-sulphonic acid and  $\beta$ -naphthol. S. and J. 81.  
6. Orange I. From *p*-sulphanilic acid and  $\alpha$ -naphthol. S. and J. 72.  
14. Narcein. Sodium bisulphite compound of Orange II. S. and J. 103.  
35. Phenotlavin. From *m*-sulphanilic acid and diamido-phenol-sulphonic acid.  
Direct Cotton 4. Benzo Orange R. From benzidine, salicylic and naphthionic acids. S. and J. 173.  
5. Salmon Red. Sodium salt of diamido-diphenyl-urea-disazo-naphthionic acid. S. and J. 143.  
6. Tolylene Orange R. From *o*-toluidine and *m*-tolylene-diamine-sulphonic acid. S. and J. 197.  
8. Cloth orange. From benzidine, salicylic acid, and resorcinol. S. and J. 170.

*Quinoline Colours.*

- Acid Colours. 41. Quinoline Yellow S. Sodium salt of quino-tha'one-disulphonic acid. S. and J. 379.

*Natural Colouring Matters.*

- Wool Book IV.  
Mordant Colours. 1. Tesu (Cr.).  
3. Young Fustic (Cr.).  
15. Anthracine (Cr.). Composition not published.  
2. Quercitron Bark (Al.). Bark of *Quercus niger*.  
3. Old Fustic (Al.). Wood of *Morus tinctoria*.  
6. Flavin (Al.). Quercetin prepared from Quercitron Bark.  
7. Persian Berries (Al.). Fruit of *Rhamnus saxatilis*.  
2. Persian Berries (Sn.).  
3. Young Fustic (Sn.).  
4. Flavin (Sn.).  
5. Quercitron Bark (Sn.).  
6. Old Fustic (Sn.).  
7. Tesu (Sn.).

NOTES.—The fugitive character of Narcein as compared with Orange II., of which it is merely the sodium bisulphite compound, is very pronounced. The bright orange of Flavin with tin mordant changes rapidly during the first exposing period to an olive-yellow, which may be regarded as "moderately fast." A similar change is noticed in the case of Quercitron Bark and Old Fustic with the same mordant, the faded colour of the latter being, however, very dull.

With aluminium and tin mordants Anthracine gives bright but very fugitive colours.

## CLASS III. MODERATELY FAST COLOURS. (WOOL.)

The colours of this class show distinct fading at the end of the second period (July 1 to July 31, 1893), which becomes more pronounced at the end of the third period (July 31 to August 26, 1893). A pale tint remains at the end of the fourth "period of exposure" (August 26, 1893, to February 19, 1894), and at the end of a year's exposure the colour has entirely faded, or, at most, mere traces of colour remain.

*Azo Colours.*

- Wool Book III.  
Acid Colours. 2. Orange GT. From toluidine and  $\beta$ -naphthol-mono-sulphonic acid S. S. and J. 41.  
1. \*Mandarin GR extra. From *o*-toluidine-mono-sulphonic acid and  $\beta$ -naphthol. S. and J. 78.

## Wool Book III.

- Acid Colours. 5. \*Orange II. From *p*-sulphanilic acid and  $\beta$  naphthol. S. and J. 73.
- „ 7. \*Orange III. From *m*-nitraniline and  $\beta$ -naphthol-disulphonic acid R. S. and J. 33.
- „ 10. Dimethylaniline Orange. From *p*-sulphanilic acid and dimethylaniline. S. and J. 74.
- „ 11. \*Diphenylamine Orange. From *p*-sulphanilic acid and diphenylamine. S. and J. 75.
- „ 12. Tropaeolin Y. From *p*-sulphanilic acid and phenol. S. and J. 70.
- „ 15. \*Metanil Yellow. From *m*-sulphanilic acid and diphenylamine. S. and J. 77.
- „ 16. Resorcinol Yellow. From *p*-sulphanilic acid and resorcinol. S. and J. 71.
- „ 18. \*Acid Yellow OO. Constitution not published.
- „ 19. \*Fast Yellow N. From *p*-toluidine *o*-sulphonic acid and diphenylamine. S. and J. 79.
- „ 28. \*Curcumein. Nitro derivative of Diphenylamine Orange. S. and J. 101.
- „ 29. \*Azodavin S. Same as Curcumein, but more highly nitrated. S. and J. 102.
- „ 34. \*— Bromine derivative of Metanil Yellow.
- „ 45. Persian Yellow (G.). Constitution not published.
- Direct Cotton Colours. 2. Salmon Red. Constitution not published.
- „ 3. Congo Orange R. From tolidine,  $\beta$ -naphthylamine-disulphonic acid R and phenol (ethylated). S. and J. 202.
- „ 7. Congo Orange G. From benzidine,  $\beta$ -naphthylamine-disulphonic acid R and phenol (ethylated).
- „ 10. Toluylen Orange G. From tolidine, *o*-cresotinic acid, and *m*-toluylenediamine sulphonic acid. S. and J. 196.
- „ 23. Carbazol Yellow. From diamido-carbazol and salicylic acid. S. and J. 181.
- „ 24. \*Cotton Yellow G. Sodium salt of diamido-diphenyl-urea-disazo salicylic acid. S. and J. 144.

## Wool Book IV.

- Mordant Colour. 24. Mordant Yellow (Cr.). Constitution not published.

## Triphenylmethane Colours.

## Wool Book III.

- Acid Colours. 33. Aurotin. Sodium salt of tetra-nitro-phenol-phthalein. S. and J. 314.

## Natural Colouring Matters.

## Wool Book IV.

- Mordant Colours. 5. Weld (Al.). *Reseda luteola* (whole plant).

NOTES.—The following colours become somewhat duller and apparently darker during the first and second periods of exposure:—Diphenylamine Orange, Metanil Yellow, Fast Yellow N, Azo-flavin S, Acid Yellow OO, and Aurotin. This appearance is only observed when the patterns are examined “underhand,” i.e., by looking down into the fabric; when they are examined “overhand,” i.e., by glancing along the surface, a normal fading of the colours is observed. This darkening is probably due to the presence of the diphenylamine group in the first four colours mentioned, and to the presence of the nitro group in the case of Aurotin, of which the alteration in hue reminds one of the change occurring in pieric acid yellow, though it is less pronounced.

Mordant Yellow with aluminium and tin mordants gives colours which may well be classed with the fast colours.

## CLASS IV. Fast Colours. (Wool.)

The colours of this class show comparatively little fading during the first, second, and third periods. At the end of the fourth “period of exposure” a pale shade remains, which at the end of the year’s exposure still leaves a pale tint.

## Nitro Colours.

## Wool Book III.

- Acid Colours. 3. \*Palatine Orange. Ammonium salt of tetra-nitro-7-diphenol. S. and J. 8.

## Hydrazon Colours.

- Acid Colours. 26. Lartrazin. Sodium salt of diphenyl-*p*-sulphonic acid osazone-dioxytartaric acid. S. and J. 19.
- „ 30. Nitrazin Yellow. Sodium salt of di-nitro-dixyl-*p*-sulphonic acid osazone-dioxytartaric acid.

## Azo Colours.

- Acid Colours. 23. \*Acid Yellow. Sodium salt of amido-azo-benzene-disulphonic acid. S. and J. 21.
- „ 24. \*Fast Yellow. Sodium salt of amido-azo-tolocene-disulphonic acid. S. and J. 22.
- „ 25. Brilliant Yellow S. Sulphonated diphenylamine Orange. S. and J. 76.
- „ 31. \*Milling Yellow OO. Constitution not published.
- „ 34. \*Milling Yellow. From  $\beta$ -naphthylamine- $\alpha$ -sulphonic acid and salicylic acid.
- Direct Cotton Colours. 11. Titan Yellow R. From thio-*p*-toluidine sulphonic acid. (Constitution not published.)
- „ 12. Chrysamin R. From *o*-tolidine and salicylic acid. S. and J. 195.
- „ 13. Cresotin Yellow R. From *o*-tolidine and *o*-cresol-carboxylic acid.
- „ 16. Chrysophenin. Ethylated Brilliant Yellow from diamido-stilbene-disulphonic acid. S. and J. 156.
- „ 17. Cresotin Yellow G. From benzidine and *o*-cresol-carboxylic acid.
- „ 19. Diamine Yellow N. From ethoxy-benzidine, phenol, and salicylic acid (ethylated). S. and J. 204.
- „ 21. Chrysamin G. From benzidine and salicylic acid. S. and J. 166.
- „ 22. \*Oriol Yellow. From dehydro-thio-*p*-toluidine-sulphonic acid and salicylic acid. S. and J. 99.
- „ 38. Titan Yellow Y. From thio-*p*-toluidine sulphonic acid. Constitution not published.

## Wool Book IV.

- Mordant Colours. 12. Chrome Orange (Cr.). Constitution not published.
- „ 13. Yellow for wool AF (Cr.). Constitution not published.
- „ 20. Chrome Yellow (Cr.). Constitution not published.

## Oxyketone Colours.

- Mordant Colours. 25. Galloclavin (Cr.). Oxidation product of gallic acid. S. and J. 242.
- „ 26. Alizarin Yellow A. (Cr.). Tri-oxo-benzophenone. S. and J. 237.

## Natural Colouring Matters.

- Mordant Colours. 2. Persian Berries (Cr.). S. Weld (Sn.).

NOTES.—In Palatine Orange we meet with the first example of a colour fast to light, the manufacture of which



has already been abandoned; possibly some difficulty or expense connected with its manufacture may account for this circumstance.

Yellow for wool AF, applied with aluminium mordant, is very fugitive, while Chrome Orange seems quite as fast as with chromium. Chrome yellow with aluminium mordant may be classed as a "moderately fast" colour.

Gallodavin with aluminium and tin mordants gives fugitive colours, more especially with aluminium.

#### CLASS V. VERY FAST COLOURS.

The colours of this class show a very gradual fading during the different periods, and even after a year's exposure a moderately good colour remains.

##### Azoxy Colours.

- Wool Book III.  
Direct Cotton Colours. 20. Carunin S. Sodium salt of azoxy-stilbene-disulphonic acid. S. and J. 16.  
" 33. Mikado Orange 3 RO. Constitution not published. S. and J. 18.  
" 35. Mikado Orange GO. Constitution not published. S. and J. 18.

##### Azo Colours.

- Acid Colours. 13. Orange GG. From aniline and  $\beta$ -naphthol-disulphonic acid G. S. and J. 28.

- Wool Book IV.  
Mordant Colours. 10. \*Alizarin Yellow R (Cr.). From *p*-nitraniline and salicylic acid. S. and J. 35.

- " 17. \*Anthracene Yellow C (Cr.). Constitution not published.

- " 18. \*Diamond Yellow R (Cr.). From *o*-amido-benzoic acid and salicylic acid. S. and J. 231.

- " 19. \*Alizarin Yellow GGW (Cr.). From *m*-nitraniline and salicylic acid. S. and J. 34.

- " 21. \*Gambine Yellow (Cr.). Constitution not published.

- " 22. \*Diamond Yellow G (Cr.). From *m*-amido-benzoic acid and salicylic acid. S. and J. 230.

- " 23. \*Flavazol (Cr.). From *p*-toluidine and salicylic acid.

- Direct Cotton Colours. 14. Brilliant Yellow. From diamido-stilbene-disulphonic acid and phenol. S. and J. 149.

- " 15. Hessian Yellow. From diamido-stilbene-disulphonic acid and salicylic acid. S. and J. 154.

- " 37. Chloramine Yellow. Oxidation product of dehydro-thio-toluidine sulphonie acid.

##### Oxyketone Colours.

- Mordant Colours. 9. \*Alizarin Orange W (Cr.) (Al.).  $\beta$ -nitro-alizarin. S. and J. 251.

##### Natural Colouring Matters.

- Mordant Colours. 4. \*Flavin (Cr.).  
" 6. \*Quercitron Bark (Cr.).  
" 7. \*Weld (Cr.).  
" 8. \*Old Fustie (Cr.).  
" 16. \*Xanthanin (Cr.). Composition not published.

NOTES.—The brownish-red given by Alizarin Orange W with chromium mordant becomes, during the first "fading period," distinctly bluer in shade, and hence apparently darker; the altered colour then fades so slowly that even after a year's exposure the faded colour appears almost as dark as the original.

The azo colours in this class which have been dyed on chromium mordanted wool leave, at the end of a year's exposure, faded colours of greater body and fulness than those applied without mordant; this is no doubt due to the inferior fastness of the latter, the faded colours of which are covered with a thin layer of perfectly bleached fibres.

All the artificial azo-mordant-colours in this class were fixed with aluminium as well as chromium mordant, and found to be equally fast to light. They were also applied with a tin mordant, but only in a few cases were satisfactory level colours thus obtained, and these seemed to be inferior to those applied with an aluminium mordant, in point of brilliancy as well as of fastness to light.

#### SILK PATTERNS.

Most of the foregoing colours were also dyed on silk, and the patterns were exposed to light along with those on wool. The relative fastness of the various colours was, for the most part, the same as on wool, the differences observed being too unimportant to necessitate a special classification for silk. In Class IV., Yellow for wool AF (Cr.) proved to be much more fugitive on silk, whereas Chrysamin R and G, Titan Yellow R and Y, Oriol, Cresotin Yellow R and G, and Chrysophenin appeared to be somewhat faster. In Class III. the same remark applies to Cotton Yellow G.

The Indian dyestuff Kamala was an additional one applied to silk, and found to belong to the fugitive class, being very little faster than Annatto.

#### GENERAL OBSERVATIONS.

The first thing which strikes one when examining these orange and yellow patterns is the comparatively large number of satisfactorily permanent colours.

In the more or less fugitive class are to be found all the basic colours, all the nitro-phenols, with the exception of Palatine Orange, and all the bright yellows derived from the natural colouring matters by means of aluminium and tin mordants, with the exception of those obtained from Weld. Comparatively few azo colours are met with in this group.

The marked alteration in colour from yellow to orange shown in the case of picric acid has long been known, and is ascribed to a reducing action of the light. The equally striking change from orange to brown, shown by Aurantia, does not, however, seem to have been previously recorded.

By far the largest number of yellows, ranging from "moderately fast" to "very fast," are to be found among the azo colours. Specially important are those in which salicylic acid is a constituent element, since not only does this impart to the colour the power of forming more or less stable lakes with chromium and aluminium mordants, but it appears frequently to give the colours the quality of fastness to light, even when no mordant is applied. It is a fact of some importance that the colours obtained with aluminium are practically as fast as those fixed with chromium, since the first-named mordant gives much brighter and purer yellows. The tin mordant, so useful in the production of the most brilliant orange and yellow colours obtainable from the natural colouring matters, seems, however, to be of little or no advantage in connection with most of these azo-mordant-colours, no doubt because they are susceptible to the reducing action of the mordant usually employed for wool—viz., stannous chloride.

Very interesting in point of fastness to light are the azoxy colours, and although unfortunately apt to dye wool somewhat irregularly, giving speckled-looking colours, they are admirably adapted for silk and cotton.

Another interesting little group is that which includes Tartrazin, a colour not only noteworthy for its fastness to light, but also because of its brilliancy and purity.

The fastness of alizarin orange is worthy of special mention, for it is probably greater even than that exhibited by most other colours of the alizarin group, and it shows the peculiar darkening action exerted by the light, probably in consequence of the presence of the nitro group.

It is remarkable how few really fast yellows are derived from the natural colouring matters, and these are chiefly

the olive-yellows obtained with chromium mordant. The only fast, and at the same time bright, natural yellows are those derived from Weld, and since this dye-stuff is now of little general importance to the dyer its cultivation has become extremely limited, and is gradually being given up; it is fortunate therefore that science has been able to replace it by efficient substitutes, so far, at least, as permanency towards light is concerned.

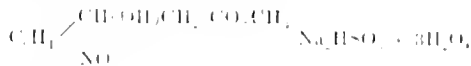
Our experiments have already abundantly proved that the popular opinion that the coal-tar dye-stuffs include only such as yield more or less fugitive colours is entirely false; indeed, it is perfectly safe to assert that coal-tar is the source from which the greatest number of colours fast to light are derived at the present time, and this seems to be specially true of the red and yellow colours.

*On the Influence of the Structure of the Textile Fibres upon the Absorption of Colouring Matters.* G. V. Georgievics. Mitt. k.k. Tech. Gew. Museums, 1894, 4 (4, 5 and 6) 163.

THE influence of the structure of the textile fibre upon the behaviour of the latter in the process of dyeing, has never been investigated, although it is well known that different kinds of wool show marked differences on dyeing, which is also the case with the various cellulose fibres, particularly flax, which are much more difficult to dye than cotton. Of the various fibres, cotton being most easily obtainable in a structureless "disorganised" form, the experiments were first tried with this material. The "disorganised" cellulose was prepared by dissolving Swedish filter paper in Schweitzer's reagent, and reprecipitating with an acid. The cotton used was pure surgical wadding. The dyes employed were benzopurpurin 4 B and benzoazurin G, the first being chemically pure, the second of 75 per cent. strength. All the dye-trials were made simultaneously, and under the same conditions, so that the colorimetric examination of the residual dye-baths at once revealed which of the two forms of cellulose employed, had taken up more of the colouring matter. Carrying out these experiments with benzopurpurin, cotton was found to absorb more of the dye than precipitated cellulose, whilst in the case of benzoazurin the latter took up as much, or but very slightly less than the former. On a similar experiment with methylene blue, the cotton had absorbed distinctly less of the dye than the precipitated cellulose, but the latter was again found to take up less tannic acid than the former. These results are therefore not available for any definite conclusions regarding the influence of structure upon the absorption of colouring matter. It is possible that the solution of the cellulose in the ammoniacal solution, is accompanied by some chemical change, so that the structureless body precipitated from this solution by acids cannot be considered the chemical equivalent of ordinary cotton. A repetition of these experiments with mercerised cotton and precipitated and mercerised cellulose, revealed the fact that the former absorbed much more dye than the latter. In all cases the dye was found to be much better fixed by the cotton than by the precipitated cellulose. In order to test wool in a similar manner, weighed quantities of wool and wool dust prepared from loose wool, were dyed under the same conditions with cyanine B. The powdered wool was found to absorb much less dye than the ordinary wool fibre, and also in this case the dye was faster to washing upon the latter. Soft, woolly asbestos fibre was converted into a fine powder, and both fibre and powder were dyed with benzopurpurin under the same conditions. The result of this experiment showed that powdered asbestos takes up very small quantities of benzopurpurin, while fibrous asbestos absorbs even more of the dye than mercerised precipitated cellulose. Altogether these experiments prove that the fibrous structure of textile materials not only favours the absorption of larger quantities of dyestuff, but also favours the better fixation of the latter upon them.—C. O. W.

*A New Compound for the Production of Indigo upon the Fibre.* G. Ulrich. Mitt. k.k. Tech. Gew. Museums, 4, 1894 (4, 5 and 6) 182.

KARL'S "indigo salt," the sodium bisulphite compound of ortho-nitrophenyl ketotone—



is sold as a paste which is easily soluble in cold water. On heating it above 50° C., the free acetone separates out. On adding alkalis to its aqueous solution indigo is readily formed. For practical purposes a solution of caustic soda of 28 B<sup>e</sup> (14.47 per cent. NaOH) was found especially suitable, but to avoid mercerisation, solutions of from 16 to 18 B<sup>e</sup> (10 to 12 per cent. NaOH) are often used. In printing, the cloth is either padded with the solution of the indigo salt, and the pattern produced by printing with a mixture of 500 parts of British gum thickening, and 350 parts of caustic soda at 40 B<sup>e</sup> (35 per cent. NaOH), or the solution of the indigo salt is suitably thickened, printed on the calico, and the latter subsequently passed through caustic soda. The pieces padded or printed with indigo salt should not be exposed to light before developing the blue. After developing, the pieces are washed, soured, again washed and dried. The blue obtained is of a fine shade and very fast to rubbing. From 20 to 25 grms. of indigo salt per 1,000 cc. of padding liquor or printing colour produce light blue, from 200 to 300 grms. dark blue. Addition of neutral salts to the caustic soda, although minimizing its mercerising influence is detrimental to the blue. Schlieper and Baum's well known Turkey-red discharge styles on indigo, may be produced with this indigo salt, by padding the oiled cloth with a mixture of aluminium bisulphite and indigo salt, and drying. On subsequent printing with caustic soda, discharge of the alumina mordant and development of the blue take place simultaneously, and the Turkey-red is subsequently produced upon these pieces in the usual manner, or Turkey-red cloth is padded with the indigo salt, dried, printed with caustic soda, dried, steamed, soured, washed, soaped, again washed and dried. The last-named method is also applicable in combination with all those dyes, the lakes of which are not decomposed by caustic soda, or the pieces may be prepared with naphthol, and printed with a diazo-compound and indigo salt simultaneously, dried and passed through caustic soda. Combinations with tannin lakes are obtained by padding with a mixture of tannic acid and indigo salt, drying, printing with caustic soda, passing through tartar emetic, washing and dyeing with a basic dye. Also combinations with "Oxidation aniline-black" are possible, and many others.—C. O. W.

*The Constitution of Turkey-Red Oils.* P. Juillard. Bull. Soc. Chim. 11, 1894, 286.

See under XII., page 820.

PATENT.

*Method of Bleaching Paper Pulp or other Flocky Substances.* J. W. Abom, Stockholm. Eng. Pat. 8964, May 5, 1894.

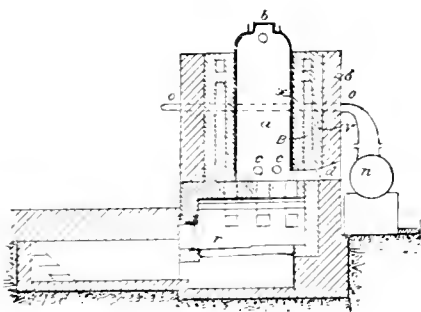
See under XIX., page 834.

## VII.—ACIDS, ALKALIS, AND SALTS.

*The Manufacture and Refining of Carbon Bisulphide at Zalatna (Hungary).* J. Farbaký. Zeits. angew. Chem. 1894, 226—231.

CASE-IRON retorts *a* (Figs. 1 to 3) of elliptical section, 36 in. by 20 in., and  $1\frac{1}{2}$  in. thick, are used. They are provided with a jacket of refractory material, in which near 4 in. flues *b* (Fig. 1) are arranged to ensure the uniform heating of the retort. The part *γ* of the brickwork also consists of refractory material (firebricks), the rest of ordinary bricks *δ* (Figs. 1 and 3). The openings at *b*, through which the coal is fed into the retorts, are provided with lids and water-joints. The sulphur is added through the openings at *c*, while the openings *d* serve for the cleaning out of the retorts. The firebars are arranged as shown at *r* (Fig. 1), the flue gases passing through *f* into the chimney (Figs. 2 and 3). The iron retorts before being brought into use are lined with a refractory coating by placing within them a jacket of sheet iron of about 2 in. less diameter than that of the retort. The annular space between jacket and retort is then filled with a warm liquid mixture of the dust of fireproof bricks and treacle. To prevent warping or bending outwards of the sheet iron, it is supported by wooden wedges or blocks. The inner space of the jacket is then filled with medium-sized pieces of limestone and the retort heated for two or three days. After this time the refractory lining is converted into a hard mass, and the wooden wedges often charred. The lime is then removed without disturbing the iron jacket which is readily destroyed in the first working of the retorts, when its *débris* and that of its supports can be removed. Each of these retorts has an average lifetime of eight months, during which it produces about 86 tons of carbon bisulphide. If the retorts are not provided with the refractory lining described, they only last from  $1\frac{1}{2}$  to 3 months. (See this Journal, 1889, 118.) When used for the first time the retorts are moderately heated for about 12 days, and are then filled through the opening *b* with pieces of charcoal about 2 in. in diameter. The heat being now raised, and whilst fumes appear at the end of the condenser, sulphur is added through short funnel-shaped tubes placed in openings *c* (Fig. 1). The sulphur is added in powder or in lumps of about  $\frac{1}{2}$  in. diameter.

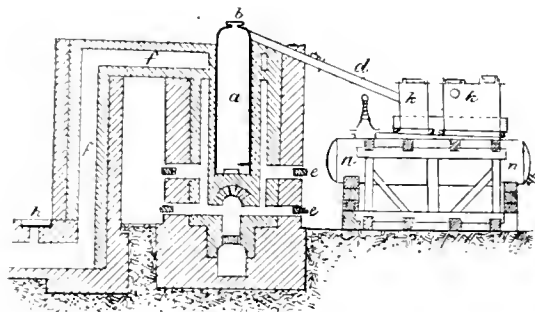
Fig. 1.



The openings *c* are then closed with clay plugs. For every 90 lb. of charcoal contained in the retorts 40 lb. of sulphur are added, and this weight of sulphur produces from 250 to 300 lb. of crude bisulphide. The charcoal used, before being added, is well dried and ignited. Nevertheless it still contains hydrogen and oxygen, which in the process cause formation of sulphuretted hydrogen and other gases. Charcoal made from beech-wood is preferable to that made from pine wood, since owing to its higher specific gravity the retorts will hold more of it and consequently require fewer charges. This means a considerable saving of time, as two hours before adding fresh charcoal, the feeding of sulphur into the retort is stopped in order to have all the sulphur in the retort converted into bisulphide, and so to prevent losses while the charcoal is being added. The daily working of

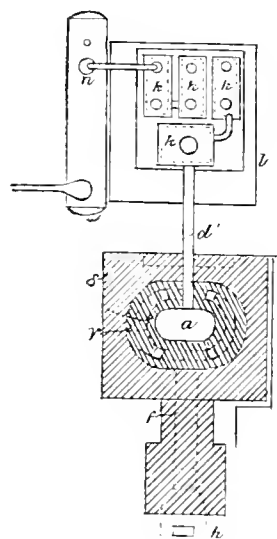
the retorts is carried on in the following manner:—The retorts are filled with charcoal (which takes about five minutes), they are then heated for two hours without adding any sulphur. Next the sulphur is fed in—a process continuing for the next 20 hours. Two hours before adding another charge of charcoal the feeding in of the sulphur is stopped. Throughout these 24 hours the temperature of the retort is maintained evenly at bright redness. Five men attend to two furnaces. At least once every fortnight the retorts are cleared of ashes and coal dust. During this operation tube *d* connecting the retort and condenser is closed with a plug to prevent

Fig. 2.



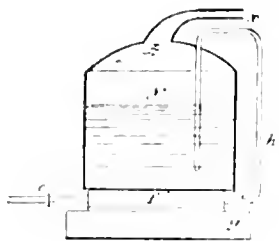
possible explosions. The waste charcoal withdrawn from the retorts is allowed to fall into water to prevent the bisulphide contained therein, from evaporating. It is then dried and sieved and used over again. The condensation of the carbon bisulphide takes place in the bell-shaped receivers *k* (Figs. 2 and 3) which are arranged in a pan *l* filled with water and acting as a condenser. These receivers, of which four are used for each retort, have a capacity of from 0.32 to 0.384 cubic metres. They are provided with

Fig. 3.



lids of from 20 to 40 cm. diameter, resting in water-lute grooves, which are, however, filled with a mixture of water and fine sand. The bisulphide distils from the retort through the pipe *d* into the condensers *k*. Any vapours not condensed there, first pass through the boiler-shaped vessel *n*, which is about 9 ft. long and 2 ft. in diameter, and travel then through three U-shaped tubes which are kept cool in a water-tank. From these tubes a pipe is

carried into the open air, with a downward bend at its end, and a zinc jug is placed underneath it in order to collect



any bisulphide which might have escaped. The bisulphide in the morning of each day is drawn from the pan *l* of the condensers and stored in iron tanks. Of the difficulties encountered in this branch of manufacture requiring special mention, the clogging up of the tube *d* by sulphur distilling over with the bisulphide, often causes interruption in the working. Experiments with retorts of different slopes and materials showed that distillation of sulphur occurs in the case of every kind of retort. To prevent explosions in attempting to remove distilled sulphur from the pipe *d* or the first condenser during the working of the retort, the pipe *d* is heated from the outside. The molten sulphur then flows into the first condenser and the pan *l*, from which it can be removed. To obtain the best results from the charges, the distillation should be conducted throughout at a bright red heat, *i.e.* about 1,000 °C. The following tables show the working results obtained:—

Year.	12 Hour Shift.	Charges.						Yield.			Fuel & Wood used.			Cost of Pro- duc- tion of 1 Hk. of CS <sub>2</sub> .	Cost of Total Produc- tion.
		Total Sulphur used.			Total Charcoal used.			Total.	Per Day.	Per Shift of 12 Hours.	From 1 Hk. of S.	Quan- tity.	Value.		
		Quantity.	Price.	Value.	Quantity.	Value.									
1882	256	Hk. 294'5	Fl. Kr. 11 32	Fl. Kr. 3,335 00	Hk. 114	Fl. Kr. 117 60	Hk. 217'68	Hk. 1'935	Hk. 0'960	Hk. 0'841	Chm. 154	Fl. Kr. 735 48	Fl. Kr. 19 93	Fl. Kr. 1,528 47	
1886	103'5	103	9 78	1,005 60	180	111 60	81'65	1'577	0'788	0'792	191	311 18	28 67	2,341 06	
1887	360	515	7 0	3,605 0	895	533 42	475	2'610	1'320	0'925	1,018	1,697 76	18 72	8,994 53	
1888	380	589	7 0	4,123 0	1,053	536 40	536	2'846	1'432	0'910	1,215	1,968 30	17 42	9,937 0	
1889	600	1,107	5 45	7,398 68	1,566	733 42	977	3'25	1'621	0'88	1,370	2,219 40	15 33	13,020 68	
1890	752	1,569'36	6 12	9,963 38	1,959	861 96	1,435	3'84	1'92	0'92	1,928	3,181 58	10 85	15,80 18	
1891	730	1,159	9 25	11,390	2,113	973 72	2,149	5'86	2'93	0'97	2,336	3,770 96	13 79	28,871 59	
1892	628	1,597	9 25	14,863 75	1,649	725 56	1,522	1'62	2'31	0'91	1,755	2,965 95	14 79	21,522 21	
1893	730	3,391	8 47	28,899 27	3,550	1,562 00	3,120	8'54	4'27	0'92	3,491	6,308 0	13 11	40,069 19	

The crude bisulphide contains from 6 to 10 per cent. of dissolved sulphur. It possesses a brownish colour and penetrating smell. It is purified by distillation from an iron still *y* (Fig. 4), 6 ft. high and about the same in diameter. The still is enclosed in a wooden jacket and the space between filled with cinders. The distillation temperature of 48° C. is raised by means of steam injected into the space under the still, forming a false-bottom *f*<sup>1</sup>, or by an improved process it is passed through *f*<sup>1</sup> and the pipe *h* directly into the still *y*. The bisulphide vapours pass through *x* into a system of four U-tube condensers, the distillate being collected under water. *z* is an opening for filling as well as for cleaning the still. The still being charged with from 40 to 45 hk. of crude bisulphide, steam at about 8 lb. pressure is admitted into the jacket. Two hours afterwards the distillation begins, preceded by a hissing noise, due to the rush of sulphuretted hydrogen which is at first evolved. The distillation takes about six hours. The removal of the residual sulphur from the still is a very disagreeable operation, resulting in suffering to the workmen from headache, vomiting, fits of weeping, pains in the limbs, general weakness of body and mind, and especially loss of memory. These affections may develop into actual insanity. To protect the workmen from the bisulphide vapours, the still *y*, after the distillation, is filled three-fourths full with water, and the latter is heated to boiling by means of directly injected steam, which is admitted through *h*. After about 20 hours' boiling all bisulphide is driven off, and the sulphur is now easily removed from the still. This recovered sulphur is subsequently used again in the manufacture. The bisulphide drawn from the condensers is stored under water in iron tanks holding from 30 to 80 hk. of it. It is sold in iron drums holding 100 or 500 kilos. each. In the former 12, in the latter 2'5 litres of air-space are allowed for the expansion of the carbon bisulphide.—C. O. W.

*The Function of Calcium Chloride, and the Formation and Constitution of the "Stiff Charges" in the Weldon Manganese Recovery Process.* J. Wiernik. Zeits. angew. Chem. 1894, 257—263.

It is well known that the addition of more than 1'5 mols. of lime to every mol. of manganese chloride greatly facilitates the oxidation of manganese solutions by means of air. The drawback of using an excess is that such large quantities of lime are likely to produce stiff charges. It is here that the beneficial influence of calcium chloride comes in. Solutions containing 2'5 to 3 mols. of calcium chloride may be safely treated with the above excess of lime, and the resulting mud will be better oxidised without having recourse to excessive blowing, and settles better than mud obtained under other circumstances. The explanation of the process cannot be given with certainty. The explanation propounded by Lunge and Zahorsky (this Journal, 1892, 882), in which stress is laid on the solvent action of calcium chloride on lime and manganese peroxide, and on the more rapid formation of calcium manganite from such a solution by the action of air, is likely to be the correct one.

Determinations of the relative quantities of calcium chloride and lime which may be present in a manganese chloride solution without producing stiff charges, show that at a temperature of 60 °C. the quantity of calcium chloride should not fall below 2'4—2'5 mols., and the lime ought not to exceed 1'7 mols. for every mol. of manganese chloride.

It is not new to practical men that the setting of the stiff charges in the oxidisers may be avoided by powerful blowing. The author fixes the period at which the charge is likely to stiffen, at the beginning of the oxidation process, and finds that the danger is great before the lapse of 1½ hours after completed precipitation of the manganese hydrate. At this juncture 30—35 per cent. of the manganese is oxidised; but the greatest danger may be expected

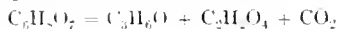
with an oxidation of 35–39 per cent. When the oxidation has advanced to 55–65 per cent., the conditions for a stiff charge are no longer present.

Judging from the period of its formation, the proportion of  $\text{MnO}$  to  $\text{MnO}_2$  in a stiff charge is 2 mols. of the former to 1 of the latter, or perhaps 5:3. Lime will be also among the constituents, but not calcium chloride or calcium oxychloride, as assumed by Post. The determination of the composition of a stiff charge by means of analysis is a difficulty which the author has not surmounted to his own satisfaction. As a likely composition he mentions 41.64 per cent.  $\text{MnO}$ , 25.51 per cent.  $\text{MnO}_2$ , 32.85 per cent.  $\text{CaO}$ , equal to  $2\text{MnO} \cdot 1\text{MnO}_2 \cdot 2\text{CaO}$ , or to  $\text{Mn}_2\text{O}_3 \cdot 2\text{CaO}$ .—H. A.

*The Decomposition of Tartaric and Citric Acids by Sunlight.* W. Seekamp. *Annalen*, **278**, 373–374.

LIEBIG observed that manganese peroxide decomposes a tartaric acid solution with separation of carbonic acid and a substance which reduces cupric salts, that malic acid when similarly treated yields aldehyde and carbonic acid, and citric acid forms acetone and carbonic acid (*Annalen*, **113**, 1). The author has investigated the action of sunlight on solutions of two of these acids, containing in addition uranium oxide, and finds that a decomposition similar to that observed by Liebig occurs after an exposure of several months. In the case of the tartaric acid, the solution contains 5 per cent. of the acid and 1 per cent. of uranium oxide. It is noticed that shortly after insolation, gas is evolved, and the liquid becomes green owing to the reduction of the uranium oxide. After several months the solution was distilled, and the distillate contained aldehyde. After removing the aldehyde, the solution showed an acid reaction, and was neutralised with freshly precipitated lead hydrate, which, on decomposition with sulphuretted hydrogen, yielded unaltered tartaric acid, malic and succinic acids. The mother-liquor, after removing the excess of lead, reduced cupric salts, but no definite compound having this property could be separated from the solution. A barium salt obtained from the solution consisted probably of propionic acid, but the quantity was too small for analysis. The author considers that the reaction takes place as follows:—Tartaric acid is decomposed first into oxalic acid and a substance,  $\text{C}_4\text{H}_4\text{O}_2$ , which reduces cupric salts. The oxalic acid then splits up into carbonic acid, carbonic oxide, and water, and the carbonic oxide liberated reduces the tartaric acid to malic acid, and this further to succinic acid; at the same time part of the malic acid decomposes into aldehyde and oxalic acid, and the succinic acid gives propionic acid and carbonic acid.

The same phenomena are observed in the case of solutions of citric acid containing uranium oxide. Gas is evolved, the solution becomes green and has a peculiar smell, and gives, after several months, acetone on distillation. The decomposition is represented by the equation—



but the further products of the reaction have not been investigated.—T. A. L.

*Yellow Prussiate of Potash and the Detection of Hydrocyanic Acid in the Presence of Ferrocyanides.* W. Autenrieth. *Arch. Chem. Pharm.* **213**, 99.

See under XXIII., page 843.

*Utilisation of Waste Carbonic Acid Gas in Breweries.* A. Marcet. *Trans. Inst. Brewing*, **7** and **8**, 199.

See under XVII., page 825.

*A Problem in Electro-chemical Economy.* A. W. Zeits. f. Elektrotech. u. Elektrochem. 1894, 95.

See under XI., page 817.

## PATENTS.

*An Improved Method of and Apparatus for Producing Carbonic Acid Gas.* H. Lane, Birmingham, and J. Pullman, London. Eng. Pat. 12,995, May 3, 1893.

Air is passed into an iron-lined clay retort, set vertically in a heating chamber, and charged with charcoal. The carbonic oxide produced passes through a valved tube into a horizontal retort, heated by gases from the first-mentioned heating chamber, so as to traverse a layer of copper oxide (for instance). The outlet tube for the carbonic acid gas formed, has a valved outlet to the air, beyond which it is connected to a blower, and the outlet tube from this has set into it a tube leading back to the vertical retort. The inventors allege that when the carbonic oxide is converted into carbonic acid by passing through the copper oxide, it is "considerably amplified in volume, the relative proportion of nitrogen being lessened." Part of the gas is allowed to escape into the air, and "the remainder of the mixture is forced back to and through the first retort."

—E. S.

*Improvements in or connected with Apparatus for the Manufacture of Chlorate of Potash by Electrolysis.* T. Hurter, Liverpool. Eng. Pat. 15,396, August 12, 1893.

The solution of potassium chloride is electrolysed in a metallic vessel, which forms the cathode. This is coated inside with a mixture of Portland cement, salt, sand, and water, which forms a porous diaphragm. The anode consists of a sheet or sheets of platinum. The cells so arranged may be connected in series or used separately.

—J. H. C.

*Improvements in Apparatus for the Electrolytical Decomposition of Brine and other Liquids.* F. G. Bailey and M. Guthrie, Liverpool. Eng. Pat. 15,610, August 17, 1893.

See under XI., page 818.

*Improvements in or connected with Pumping and Absorbing Chlorine Gas, and Apparatus for use therein.* P. J. Worsley, W. Windus, and B. Bracey, Bristol. Eng. Pat. 16,151, August 26, 1893.

CHLORINE, and especially dilute chlorine, such as that resulting from the "Deacon" and other processes, is well dried before presenting it for absorption by milk of lime or other agent, since the inventors have found that chlorine gas in a dry condition, does not act readily on ordinary metals, and can therefore be pumped by an ordinary air-compressor.

The chlorine is passed through a lead or earthenware vessel charged with coke or the like, over which a flow of sulphuric acid is maintained. The gas has exit through a liquid seal, at the upper part of the desiccator, into a pipe connected to a pump of ordinary construction, which, as well as the inlet and outlet valves, may be made of metal. Or the pump cylinder and piston may be lined with ebonite, and india-rubber valves may be used. The lubricator should be a pure mineral oil not acted upon by chlorine gas. The pipes and distributor may be of cast iron or earthenware. The distributor, placed near the bottom of the absorbing tank, is a system of pipes, slotted on the under side, so that the gas may rise through the absorbing liquid in bubbles or fine streams. An agitator is provided.—E. S.

*Improvements in the Methods of Effecting the Electrolysis of Saline Solutions.* L. A. P. and H. E. A. Liénard, Paris. Eng. Pat. 6046, September 22, 1893.

See under XI., page 818.

*Improvements in Electrolytic Apparatus.* T. Craney, Bay, U.S.A. Eng. Pat. 9761, May 18, 1894.

See under X1., page 819.

*Process for Treating Black Furnace Gases for the Extraction of Alkaline and Ammonium Compounds, Iodides, and Iodine therefrom.* O. Murray, London. From P. Gredt, Esch-sur-Alzette, Belgium. Eng. Pat. 10,589, May 31, 1894.

See under X., page 816.

## VIII.—GLASS, POTTERY, AND ENAMELS.

### PATENTS.

*Improvements in the Manufacture of Earthenware or Porcelain Filters and Analogous Articles.* A. da S. Prado, Paris, and B. Medina-Santurio, Houilles, France. Eng. Pat. 14,132, July 21, 1893; date of first foreign application, February 17, 1893.

THE inventors have departed from the ordinary method of moulding or casting filters and analogous articles, and in lieu of such processes they compress the materials of which the articles are made round a core of fusible material, such as a metallic alloy. The core is provided with three or more projections, which serve to keep it in place and which provide a channel of escape for the fusible material of which the core is composed, when the article is placed in the furnace. The materials used for the filter or other analogous article are such as are usually employed, e.g., kaolin, clay, asbestos, and the like. The materials are compressed in a moist state, but not in the condition of a paste. The core may be composed of various materials, almost anything which melts or vaporises or burns away when heated may be employed, provided that it be practically incompressible. Besides alloys, sawdust with gelatin, charcoal, and even ice are described as suitable materials.—V. C.

*Process of Producing Intensely-Brilliant Pictures, Ornamentations, Inscriptions, or Drawings by Colour-Printing on Glass, and with a Backing Stamped in Relief.* A. Gortitz, Zürich. Eng. Pat. 7844, April 20, 1894.

THE design or inscription is first engraved on a printing-plate, for which rubber is a suitable material; the design being engraved *positively*, that is to say, in the same way as that in which it will be afterwards seen. The plate is then coated with varnish colour, and pressed upon a glass plate. The glass plate is strewn with bronze-powder, sheet aluminium, or other suitable material, the portions forming the design or inscription remaining empty, and being therefore transparent. The glass plate is then placed in a frame having a backing of strong paper-board, on the front face of which is mounted a brilliant sheet of tinfoil or tinplate, provided with prominent squares placed in suitable positions. The design is thus shown by a brilliant reflected light visible through the transparent part of the glass, the other portion of the glass forming a backing stamped in relief.—V. C.

*Improvements in the Production of Enamelled Letters and Designs on Glass, Porcelain, Stoneware, and other Surfaces.* C. Hodan, London. Eng. Pat. 11,513, June 12, 1894.

HERETOFORE raised enamelled writing and designs in relief on glass have been produced by means of a brush and thin enamel-paint. The inventor uses stencil-plates, preferably

of tinfoil or other flexible material, and a composition made of glass-powder, made up to the consistency of treacle with turpentine and "glaze." The composition is applied with a spatula through the openings of the stencil-plates, and the article is then fired. If the surface on which the design is to be produced is irregularly curved, or is curved both ways, the stencil is applied to sized paper, and the design is transferred from the dampened paper to the glass or ware. —V. C.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Asphalt Paving.* Report to the Department of Public Works, Philadelphia, U.S.A. 1894. (This Journal, 1893, 265.)

See under III., page 796.

### PATENTS.

*New or Improved Process or Means for Connecting Metal to Earthenware or China.* W. B. H. Drayson, London. Eng. Pat. 23,651, December 8, 1893.

THE process is applicable more particularly to the connection of lead pipes with the earthenware of closet basins or lavatory basins. The portion of the earthenware with which connection is to be made being unglazed, or the glaze having been removed, it is coated with plumbago, and placed in an electrolytic bath, whereby a firm metallic coating is obtained. The lead pipe is then soldered to this coating by a plumber's "wiped" joint. By this means are avoided the imperfect joints made with india rubber sleeves, washers, or putty.—V. C.

*Artificial Lumber or Composition Material.* J. C. Fell. From C. A. Smith, Minneapolis, U.S. Eng. Pat. 6678, April 3, 1894.

THE new material is produced in the form of boards of great strength, inflexible, and suited for all climates, not swelling or warping, and capable of taking a high polish. The boards are made up of layers of paper and wood, secured by adhesive material. The central strip or body of the board consists of sheets of wood laid end to end, above and below which are thinner sheets or veneers of wood having the grain transverse to the direction of the grain of the thicker sheets. The thinner sheets or veneers are provided with serrated edges, which provide a large surface for the thin and even spreading of a quick-drying adhesive material, which serves to weld the whole into a firm mass. On either side of the nucleus or body, consisting of the "sheets" and the "veneers" of wood, are thick sheets of paper, thick heavy straw- or pulp-board. Before using, the whole is subjected to great pressure, thereby disseminating the adhesive material, and subsequently dried and heated in a kiln. The finished material, although inflexible, can be worked by ordinary carpenter's tools.

By the employment of suitable materials, the composite board may be made waterproof, or fire-proof, or both.

—V. C.

*Improvements in the Manufacture and Treatment of Fire-proof Plastic Material for Cement.* R. Stone. Eng. Pat. 8549, April 30, 1894.

THIS improvement relates to a process described in Eng. Pat. 16,789, 1890 (see this Journal, 1891, 932), and consists in adding pearlsh, potash, soda, or sodium silicate to the petroleum or other inflammable oil which is used as a flux in the manufacture of the cement. About 7 cwt. of the

pearlash. No. is mixed with 1 cwt. of the flux, and the whole is then mixed with about one ton of the raw material, which consists of granite, slate, shale, clay, or similar substances.—V. C.

*An Improved Process of Manufacturing Porous Bricks.*  
H. Maurer, New York. Eng. Pat. 9948, May 22, 1894.

Porous bricks have formerly been made from clay and straw, but the inventor finds that a better quality is produced by employing the dung of animals which feed upon straw or hay. Equal parts of dung and clay make a good hard brick; five parts of dung to three of clay make a good soft brick.

The improved brick has strength sufficient for use in all the ordinary ways, and is especially adapted for fire-proofing and for interior use in all places where nails and screws are needed.—V. C.

## X.—METALLURGY.

*The Austin Process for the Treatment of Pyritic Minerals by Fusion.* Bull. Soc. Chim. 11, 1894, 448.

THE precious metals are often associated with sulphurous ores, especially in minerals obtained from deep mines, and such minerals often lend themselves, where the accompanying sulphides are those of copper, lead, or iron, or where these may be cheaply added to the mineral, to a fusion process, by means of which a *matte* of sulphides may be obtained; such *matte* collecting the precious metals, formerly diffused over a large bulk of ore, into a relatively small bulk. In this way many ore-deposits—for example, several in Transylvania, Sweden, and North America, which are either refractory, relatively poor, or remote as to locality—may be concentrated into a bulk capable of justifying the cost of transit to a metallurgical centre, and so be utilised.

At the present time this production of *matte* is always accomplished by aid of heat derived from the burning of coke, charcoal, anthracite, &c., the price of which therefore dominates the problem.

Mr. Austin proposes to employ as source of heat, elements other than carbon, and which are to be found in the mineral itself or in others in the immediate locality; for example, by means of pyrites only as fuel, he smelted *matte* from the refractory argentiferous and auriferous quartz ores of Colorado, Montana, and Arizona, which he was able to send to New York, Baltimore, &c., to be worked up.

The possibility of this procedure has been questioned, for it has been pointed out that iron pyrites ( $\text{FeS}_2$ ) only yields 2,253 calories, and copper pyrites but 1,700.

Mr. Austin's reply to these criticisms has been reproduced in the *Berg u. Hüttenm. Zeit.*

Iron, sulphur, and arsenic give out light and heat during oxidation, if the duration of the reaction is sufficiently shortened; there is therefore no reason why pyrites—a compound, as it is, of two combustible bodies—should not, with suitable plant, furnish the heat necessary in metallurgical operations; similarly, both blende and galena may be employed. The *rationale* of the operation is as follows:—Iron sulphide, burning at a high temperature, in presence of strongly heated silica, yields a very fusible slag of ferrous silicate, while the excess of the sulphides produces a *matte* which collects the gold and silver. The necessary attendant conditions are: a sufficiently high temperature at the tuyères; sufficient amount of hot air-blast to prevent chilling the furnace; a sufficiently open charge to allow of free circulation of the blast. The furnace is therefore peculiar from the fact that its atmosphere is always an oxidising one. Part of the S escapes as  $\text{SO}_2$ , and carries away some heat; but this, according to Austin, is small compared to that lost by the CO and  $\text{CO}_2$  in the ordinary (C) process of *matte*-making.

Pyrites ( $\text{FeS}_2$ ) loses an atom of S at a dull red heat ( $700^\circ$ ), so that only FeS is present in the zone of fusion, and Austin considers that the theoretically maximum temperature, T, attained, at or near the tuyères, when the theoretical quantity of blast acts upon the charge, is shown by the expression—

$$T = \frac{\Sigma ag + atq}{\Sigma pc - C}$$

where  $a$  = heat of combustion of each element (Fe and S);  $g$ , their weights;  $atq$ , heat introduced by the blast, of weight  $q$ , at temperature  $t$ ;  $\Sigma pc$ , the sum of the products of the specific heats of the products of combustion by the corresponding weights (FeO,  $\text{SO}_2$ , N); and C, specific heat of FeS.

Taking the temperature of the blast at  $400^\circ$ , Austin finds  $T = 3,553^\circ$ . Of the total heat, 11 per cent. is contributed by the blast.

It is considered that the co-efficients employed by Austin are somewhat high, but yet, seeing that the internal temperature of the furnace ought not to exceed  $1,500^\circ$ , there remains a large balance of heat; one certainly sufficient to heat the silica and to fuse both slag and *matte*. The comparative utilisations of the heat may be estimated by contrasting the total calories evolved by the combustible in question with those remaining in hand after allowing for the raising to  $1,500^\circ$ , of the volatile products of combustion.

Taking  $\text{FeS} + 3\text{O} = \text{FeO} + \text{SO}_2$  ( $34.5 + 34.6 - 11.9$ ) = 57.2 calories or 1,298 calories per 1 kilo. of FeS are evolved: the heat introduced by the blast being deducted. Hence, for heating the slag (FeO,  $\text{SiO}_2$ ) and the *matte*, there remains—

$$1,298 - 1,500 [0.7272 \times 0.1553 + 1.82(\text{N}) \times 0.2438] = 164 \text{ calories} = \frac{164}{1,298} \text{ or } 12.6 \text{ per cent. of the total heat involved, or, including the heat (11.9 calories) introduced by the hot blast, 16 per cent.}$$

Taking now the case of carbon. In burning to  $\text{CO}_2$ , 3,600 calories are available, and to CO, —69 calories; so that, supposing that at the tuyères in a lead or copper smelting furnace, 45 per cent. burns to  $\text{CO}_2$ , and 55 per cent. to CO, there would be available,  $0.45 \times 3,600 = 0.55 \times 69 = 1,852$  calories, or a little less than 20 per cent. of the calorific value of the carbon involved.

Greater possibilities are therefore shown by the pyrites furnace.

The advantages of the Austin process may be summarised as follows:—

1. A means of melting very refractory mineral without the necessity of using *matte*s containing either lead or copper; iron pyrites serving both as melt and fuel.
2. Saving cost of preliminary roasting.
3. Saving cost of transit of fuel.
4. Utilisation of deposits, unworkable otherwise.
5. The greater heat at disposal allows of use of more siliceous slags; hence diminution in the weight of the charge and saving of labour.

The principal disadvantage is the necessity of employing fuel to heat the blast, and this fuel is additional to that which will be consumed in blowing, if there is no waterfall available. Austin estimates the consumption of fuel for this purpose at 5 tons for the air necessary to melt 1,000 tons—a very trifling matter.

The process undoubtedly possesses the greatest interest, and appears to have before it a brilliant future. Its field would be still further enlarged if the escaping sulphurous gases were utilised.—E. R. B.

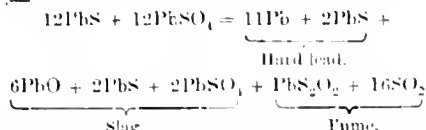
*The Interaction of Sulphide with Sulphate and Oxide of Lead.* J. B. Hannay. Proc. Chem. Soc. 1894, 151. (This Journal, 1894, 639.)

In this paper the author examines the equations suggested by Percy to account for the smelting of lead in the reverberatory furnace. The two main reactions which are supposed to occur are usually represented by the equations  $\text{PbS} + \text{PbSO}_4 = 2\text{Pb} + 2\text{SO}_2$ , and  $\text{PbS} + 2\text{PbO} = 3\text{Pb} + \text{SO}_2$ ; and although Percy relies on the first as representing what

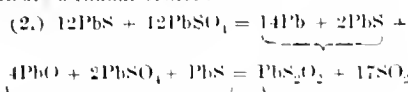


takes place, he also says that a mixture of the sulphate and oxide reduces the sulphide. The author finds that a much more complex reaction takes place, since metallic lead, when formed, attacks the remaining sulphate, producing litharge, which in its turn reacts with the sulphide, while some of the sulphide is removed by being dissolved in the metallic lead, and some is volatilised as the volatile compound  $PbS_2O_2$ . The result varies according to temperature and the method of mixing the substances. The following equations correspond with the results the author has obtained:—

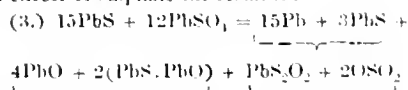
(1.) Lead sulphide and sulphate mixed in molecular proportions and very finely ground, and brought to calm fusion:—



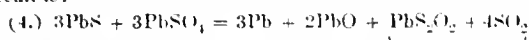
34.8 per cent. of lead was obtained instead of 76.3, as required by Percy's question. When the sulphide of lead is in small pieces the reaction yields more lead, but otherwise pursues a similar course:—



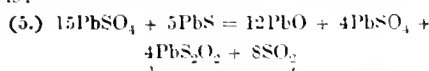
With excess of sulphate the result is:—



On fusing lead sulphide and adding solid sulphate, the result is:—



On fusing the sulphate and adding solid sulphide, the result is:—

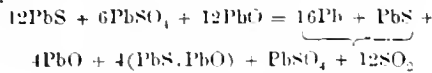


The temperature in this last case was much higher owing to the high melting point of the lead sulphate, and thus a larger amount of fume was produced.

On mixing sulphate and oxide in the proportions which ought to form lead and sulphur dioxide, as by the equation:—



no such result was obtained, but the reduction took this form:—



The author has shown in another paper, that the sulphide and oxide do not react to form lead, but combine together to form the substance  $PbS.PbO$ , which is the basis of lead slags.

Professor Roberts-Austen said, in discussion, that Mr. Hannay's statements presented themselves under two main aspects. It might be well either (1) to test the accuracy of his views as to the action of atmospheric air on sulphide of lead, or (2) to endeavour to obtain evidence as to the existence of volatile gaseous compounds of sulphide of lead. The speaker had adopted the latter course. He stated that he had understood Mr. Hannay to say distinctly, that if a mixture of sulphide and sulphate of lead were heated strongly no reaction ensued; hence Prof. Austen's appeal in favour of the accuracy of certain of the equations which were given by Percy and accepted by metallurgists. He was glad, however, to find that Mr. Hannay did not maintain this extreme view. Prof. Austen had experimentally confirmed the fact that the following equation is

substantially correct. —  $PbS + 3PbSO_4 = 4PbO + 4SO_2$ . This is a very important point, as it is one of Percy's main equations; but he had pointed out on the previous occasion, that both this equation and the one which has hitherto represented the reaction between oxide and sulphide of lead are strongly endothermic, and in the case of an endothermic equation, some explanation had to be sought; energy in some form had to be imported into the system, and an endothermic equation would represent a reaction which would be less likely to take place at a low than at a high temperature. In the case of the well known endothermic interaction between carbon and the oxides of iron, it was probable that the iron carbonyl played an important part, which certainly pointed to the possibility of similar action on the part of *poisons metallic* compounds in the metallurgy of lead. The well-known statement, "that the volatilisation of sulphide of lead is promoted by currents of gas, such as proceed from the combustion of fuel," a statement made by Dr. Percy in 1870, might well be taken as a starting point. There could be no doubt that when galena is strongly heated in a porcelain tube between two plugs of asbestos in a current of sulphur dioxide, the galena is volatilised, and is driven right through the plugs, condensing on the other side of them. Such experiments were made in the School of Mines Laboratory, the temperature being carefully measured by the aid of a thermocouple, and it was found that at a temperature of 1,357° C., 9 grms. of galena lost 38.2 per cent. of its weight in sulphur dioxide in 20'; while 9 grms. of galena lost 37.7 per cent. in nitrogen in 20'. At a still higher temperature, 1,434° C., 9 grms. of galena lost 50.2 per cent. of its weight in sulphur dioxide in 20'; while 9 grms. of galena lost 72.3 per cent. of its weight in nitrogen in 20'. This result, showing the volatility of galena in ordinarily pure nitrogen, was not a little surprising; but Mr. Rose had made a perfectly independent experiment, the result of which quite confirmed the volatility of galena in nitrogen. Prof. Austen had also ascertained the fact that at 1,200° galena may be rapidly distilled *in vacuo*, and, so far, he had been unable to obtain any evidence in favour of the view that sulphur dioxide promotes the volatilisation of lead sulphide more than nitrogen does. He had, however, tried a further experiment. A kind of air thermometer was arranged, with a porcelain tube for a bulb, in which a certain amount of galena was placed in an atmosphere of sulphur dioxide. The tube was then heated strongly, and the expansion of the gas carefully measured by means of a moving index of mercury in a horizontal capillary tube. The presence of galena produced no abnormal change in the volume of sulphur dioxide such as might be expected to occur if a gaseous compound of sulphur dioxide and galena were formed. It should be remarked, however, that sulphur dioxide alone appears to exhibit contraction about 1,000°, and the cause of this fact is being examined; but the presence of galena did not increase the observed condensation. Metallurgists would welcome the proof of the existence of gaseous compounds of lead sulphide; but although Mr. Hannay had undoubtedly shown that the whole metallurgy of lead should be further investigated, the existence, composition, and nature of the volatile compounds could not, as yet, be considered to be established.

Mr. Rose had tried to isolate the supposed compound  $PbS_2O_2$  by means of Deville's hot and cold tubes, galena being heated to about 1,250° in a stream of sulphur dioxide, but had been unsuccessful, the sublimate on the cold tube consisting entirely of lead sulphide. It was to be remembered that several substances, such as ozone and oxide of silver, which, although more or less unstable at moderate temperatures, could exist at a white heat, had been prepared in this way, and the method had appeared to be the most likely one to succeed in effecting the isolation of  $PbS_2O_2$ . The failure was therefore not without significance.

Mr. H. C. Jenkins said that lead sulphide (galena) was easily volatile in a vacuum at a temperature only a few degrees above its melting-point. In a current of sulphur dioxide the rate of volatilisation was, as would be expected, much increased, but a similar current of nitrogen gave even better results than sulphur dioxide. Very careful experi-



ments were made with mixtures of galena and sulphate of lead, but, although some of the products resembled those that Mr. Hannay had described, yet the evidence obtained did not point to the existence of a volatile compound  $PbS \cdot O_2$ . In some cases only the acting compounds were present; in others the experiments were performed in an atmosphere of sulphur dioxide in the presence of furnace gas. Metallic lead was only seen as a separate product in the latter case. But it was found that pure metallic lead is capable of decomposing sulphur dioxide, forming sulphur and oxide. Experiments were still in progress with a view to obtain quantitative knowledge of the change. The existence of volatile lead compounds would, if proved, offer an easy explanation of the cause of lead fume, but in view of the volatile character of well-known compounds of lead at furnace temperatures, much more experimental evidence than has yet been afforded is needed to demonstrate the formation of new compounds.

Dr. Armstrong said the experiments which he had witnessed certainly favoured the conclusion that sulphur dioxide exercised a specific effect in volatilising lead sulphide; but assuming that the evidence that combination took place was satisfactory, he could not regard Mr. Hannay's observations on the extent to which volatilisation took place as satisfactory determinations of the composition of the volatile compound; such a compound would probably be dissociable, and, if so, it was not to be expected that the gas and galena would pass over and be collected in exact molecular ratios. The strongest evidence was undoubtedly that derived from passing air into molten galena; the manner in which galena volatilised and passed away as lead sulphate under such conditions was extraordinary, and still more remarkable was the fact that, as Mr. Hannay stated, just half the lead was obtained in the metallic state. Experiments made in his laboratory under Mr. Hannay's direction had given this result, and it was certainly a remarkably coincidence if the result were but accidental. It appeared to him that, although Mr. Hannay could not be held to have established his point, he had called attention to peculiarities in the behaviour of lead sulphide which were of great interest to metallurgists, and that he had clearly made out a case for inquiry.

Mr. Hannay, in reply, said that he was glad to find it was now recognised that the simple equations of the textbooks did not adequately represent the facts, and that nearly all the furnace reactions of lead compounds were very complex. As to the volatility of the sulphide and the formation of a definite compound with sulphur dioxide, this conclusion was based on the action of air on molten galena, an action which always proceeded in the same manner, and was represented by the equation  $2PbS + O_2 = Pb + PbS \cdot SO_2$ , as exactly one-half of the galena is reduced to lead and left in the metallic state, and one-half is volatilised with the sulphur dioxide. Whether this was a definite compound or a mere chance volatility in molecular proportions was a question which might still admit of experimental investigation, and it was to be hoped that some of those who had spoken would carry the work further. As showing that sulphur dioxide when set free in intimate contact with molten galena, does act in a special manner, it might be stated that while nitrogen and hydrogen gases passed at the rate of 1 litre in three minutes through the molten sulphide, carried over the galena as vapour at the rate of 3.5 grms. per litre of gas passed, air under the same conditions carried over 5.2 grms. per litre. Now if the amount carried over by the nitrogen of the air were the same as that carried over by pure nitrogen, there is a large excess carried over by the sulphur dioxide (formed by the combination of the oxygen of the air with the sulphur of the galena), and this excess is present in nearly the proportion indicated by the formula  $PbS \cdot SO_2$  or  $PbS \cdot O_2$ . All the evidence pointed to the fact that when sulphur dioxide was formed in intimate contact with molten galena, every molecule of sulphur dioxide rendered volatile one molecule of galena, and subsequently deposited it on cooling. The volatility is so profoundly modified by temperature, that experiments are not comparable unless carried out at the same temperature, a result difficult to obtain; but Mr. Hannay hoped that Prof. Roberts-Austen would test the

matter and lay down accurate data for future work, as all statements of temperature had hitherto been only the roughest approximations.

#### *Hempel's Experiments on Zinc Smelting in Blast Furnaces.* Bull. Soc. Chim. 1894, 12, 248—252.

THE reduction of the zinc oxide in a blast furnace is quite possible if access of air be prevented, either by using steam or an excess of carbonic acid. The results achieved in the lead furnaces employed in the Harz district prove this assertion. On account, however, of the volatility of zinc, the furnace gases carry away large quantities of this metal, and in order to avoid enormous losses, it becomes necessary to precipitate it from the gases. The usual means for this purpose are insufficient in this case, but the problem has been solved by submitting the gas to centrifugal action, when the volatilised metal is obtained in the form of bead-like granules at a temperature of about 500° C. The experiments were carried out with a mixture of 1 part of oxide of zinc, 0.65 part of limestone, and 3 parts of coal. This mixture, heated in a crucible, gave a coke-like mass, containing about 22.7 per cent. of zinc and 49.7 per cent. of carbon. This product was treated in a small furnace working with a cold blast and provided with a Parry's hopper for feeding in the charge. The furnace gases were passed through a centrifugal ventilator, making 1,500 revolutions per minute, and then travelled through a vertical condensing flue. The pyrometer indicated a temperature for the gas of 470° C., and a content of 4 per cent. of carbonic acid was found. After some hours working only little zinc dust was found near the ventilator, more being found on the top of the condensing flue. Most had been carried away. This small quantity of zinc found near the ventilator proves that the condensation of the metallic vapour can only be carried out successfully at temperatures below 470° C. Experiments made by passing currents of different gases at varying speeds over the surface of zinc at a white heat demonstrates the fact that the formation of zinc dust depends upon the dilution of the metallic vapours as much as upon the chemical nature of the diluent gases. These experiments further showed that the zinc is volatile at a temperature very little above its melting point, i.e., about 500° below its boiling point. This explains why zinc smelting in a blast furnace where the fused metal is necessarily in contact with considerable volumes of gases, leads to the formation of zinc dust. The condensation of volatilised substance results in the formation of solid masses or coherent liquids, only if the dilution of the volatilised substance by indifferent gases, does not exceed a certain limit. Contrary to general opinion the reduction of the oxide of zinc takes place much below the boiling point of the metal. These facts suggested an attempt to manufacture very dense zinc-grey by the use of a polygonal screen or jacket against which the condensed metallic dust would be thrown. 17.5 kilos. of the above-mentioned coke-zinc were used. Between furnace and ventilator 1,010 grms. of zinc dust containing 72 per cent. of zinc were deposited; from the ventilator 2,370 grms. containing 90 per cent. of zinc were obtained; in a bag behind the ventilator 458 grms. containing 82 per cent. of zinc were collected. The escaping gases were free from zinc, and contained from 1.8 to 4 per cent. of carbonic acid. The rest of the zinc not accounted for above was absorbed by the lining of the furnace. This shows that it is possible to manufacture zinc-grey of high density in a blast furnace and to collect it by means of a ventilator. An interesting experiment was made by adding to the coke-zinc an argentiferous lead ore and ferric oxide. The latter is either reduced or scorified. All the lead and silver distil with the zinc. From ores rich in lead and silver, these metals can therefore be obtained partly in the metallic state, partly in the form of dust, and, if a satisfactory method for the treatment of this dust can be found, it is evident that the whole process would become of considerable practical importance. For the partial separation of these metals distillation was tried. The distillation of the dust, however, offering great practical difficulties, it was submitted to a pressure of 100 atmospheres, whereby it was obtained in the form of solid blocks, the volume of which is about one-tenth that of the original dust. On distilling the

blocks without addition of carbon very pure zinc to the extent of two-thirds of their weight was obtained. The residue contained  $\text{SiO}_2$ , 11.6;  $\text{Fe}_2\text{O}_3$ , 2.9;  $\text{CaO}$ , 0.6;  $\text{Zn}$ , 33.6;  $\text{S}$ , 8.1;  $\text{PbS}$  +  $\text{Ag}_2\text{S}$ , 1.05.

This residue, if the proportion of silver present be sufficient, can be very satisfactorily treated by the usual processes. The distillation of the compressed dust is preferable to the distillation of zinc oxide with carbon, as there is evolution of no gas, and therefore no loss of metal from this source. There is also a saving of space and fuel, owing to the conductivity of the compressed metal being much greater than that of the dust. From the above it follows that the treatment of zinc ores in blast furnaces requires three operations: (1) manufacture of zinc dust, deposited by centrifugal force; (2) compression of the zinc dust; (3) refining of the zinc dust by distillation. The ores, sulphides, or carbonates must be well roasted. Ferruginous ores should be reduced as much as possible with a reducing flame. Calcined fluxes would have to be used which, together with bituminous coal, are mixed with the ore and coked in an Appolt furnace, from which the red-hot coked mixture is at once transferred to the blast furnace. The gases, cooled to  $50^\circ\text{C}$ ., pass subsequently through one or more centrifugal ventilators provided with polygonal jackets. No more than 4 per cent. of carbonic acid should be contained in the gases. The distillation is carried out in crucibles of slightly greater diameter than that of the cylindrical blocks of compressed dust.—C. O. W.

*On New Alloys of Copper (Ducta Metal).* G. v. Knorre. *Zeits. angew. Chem.* 1894, 238.

THE specific gravity of this metal is 8.077 at  $16^\circ\text{C}$ ., its tensile strength 58 k. per sq. mm., its coefficient of elasticity 48 k. per sq. mm., its elongation under tensile strain 14 per cent. in 100 mm. of original length. The analysis of this alloy gave the following figures:—

	I.	II.	III.	IV.	V.	Mean.
Tin and antimony ..	2.25	2.21	2.20	..	..	2.22
Iron .....	1.70	..	..	1.73	..	1.71
Aluminium .....	1.72	1.69	..	..	..	1.70
Copper .....	64.79	64.80	64.80	64.72	64.77	64.78
Zinc .....	29.49	..	29.51	..	..	29.50

According to the statement of the makers, cadmium is an essential component of this alloy, but analysis showed it to be free from this metal. On the other hand the makers do not mention the presence of aluminium in the alloy.

The analysis of a tube gave the following figures:—

	I.	II.	Mean.
Copper .....	61.42	61.50	61.46
Lead .....	0.85	0.87	0.86
Iron .....	0.92	0.90	0.91
Manganese .....	0.78	0.74	0.76
Zinc .....	35.98	..	35.98

showing that it consisted of an alloy of brass and lead, to which, on melting, ferro-manganese had been added as a reducing agent.—C. O. W.

#### PATENTS.

*Separating and Purifying Metals.* J. A. Mays, Hampstead. Eng. Pat. 8964, May 4, 1893.

THE claim is limited to the separation of metals from molten lead by the use of molten zinc or an alloy of zinc. The molten lead containing gold, silver, or other substances

to be separated is made to pass in minute particles through the molten zinc or zinc alloy, when the lead is purified, and the gold, &c., are retained by the zinc. The reactions may be effected either by the aid of gravity or by that of centrifugal force.—J. H. C.

*Improvements in the Manufacture of Metallic Alloys or Compounds.* R. Howarth, Wolverhampton. Eng. Pat. 15,117, Aug. 8, 1893.

FOR the purpose of thoroughly alloying iron, copper, tungsten, or other metals with zinc, without loss of the latter metal, the mixture is heated to and maintained at a red heat in a flanged crucible, which is capped with an iron collar, so arranged that by means of a collar a lid may be firmly pressed into contact with the crucible. The cover is luted on, and the crucible, which should be nearly filled, is made of plumbago. The time required for heating must be determined by experiment. When the alloy is required for further mixture the metal may be allowed to solidify in the pot, and may be subsequently broken up.—W. G. M.

*Improvements in Processes of Coating Metals with Aluminium or its Diluent Alloys.* E. C. Broadwell, Philadelphia. Eng. Pat. 15,152, August 8, 1893.

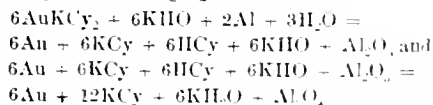
THE metals to be coated are first cleaned, then covered with a flux containing some halogen compound of an electro-negative metal, such as tin chloride, and then dipped in a bath of molten aluminium or alloy.—J. H. C.

*Improvements in Apparatus for the Manufacture of Cast Iron and Steel.* C. Allen and C. Davy, Sheffield. Eng. Pat. 15,875, August 22, 1893.

A STORING and mixing vessel for liquid metal is described, the cross section of which is rather more than a half-circle; it is provided above with a covered movable channel, which can be detached when not in use, for the feeding of the metal into the mixer from ladles brought alongside, and at one side is a tap-hole which may be brought into use by tilting the vessel and opening a protected plug. The whole vessel is mounted on rollers and fitted with hydraulic machinery adapted to communicating transverse oscillating motion to it, while along the centre of the bottom is a longitudinal ridge to aid in the mixture of the fluid contents. At either end are detachable flues, placed in the axis of oscillation, and connected with regenerators, which serve to maintain the fluidity of the contained metal when necessary.—W. G. M.

*A Process for Precipitating Precious Metals from Solutions.* C. Moldenhauer, Frankfort-on-Maine. Eng. Pat. 16,361, September 2, 1893.

ALUMINIUM is employed instead of zinc, and in the presence of free alkali or earthy alkali, for precipitating gold from a cyanide solution. This results in an important economy of cyanide of potassium which is continually being regenerated as shown by the following equations:—



—J. H. C.

*Improvements in the Method of Separating Metals from Ores and from other Mixtures of Insoluble Substances therewith.* E. R. Besenfelder, Grosse Mochbern, Silesia. Eng. Pat. 23,619, December 8, 1893.

THE dry ores are exposed to heat under pressure in contact with ammonium chloride or a similar salt. Any excess of reagent and the volatile products are distilled or sublimed and condensed in a suitable receiver and subsequently

utilised. The fixed residue is heated with a solvent such as water and the solution as well as the insoluble portion are subsequently dealt with. The heat and pressure may vary within considerable limits, thus, in treating the strontian residues from the de-saccharisation of molasses one part of the dried and pulverised residue is mixed with two parts of ammonium chloride, placed in a suitable rotating vessel and heated for one hour to 350° C. under a pressure of from 3 to 6 atmospheres. The volatile, soluble, and insoluble parts obtained as above are then separately treated and purified by known means.—J. H. C.

*An Improved Apparatus for Precipitating Gold and Silver from Cyanide Solutions.* A. H. Bell and C. Carter, Ithaca, Utah. Eng. Pat. 1472, January 23, 1894.

The solution is made to rise up against and between moveable and stationary zinc surfaces which are kept bright by friction against each other. The particles of zinc so removed, pass into the solution together with the precipitated gold and silver which are collected in a settling chamber and taken out at suitable intervals.—J. H. C.

*Improvements in Furnace and Apparatus for Working Gold, Silver, Copper, and other Ores.* C. A. Allison, London. From J. J. Storer, F. Martin, and G. O. Eaton, Helena, U.S.A. Eng. Pat. 6263, March 28, 1894.

In this specification, a vertical shaft with an inner lining has at its summit a series of fire-places with charging doors and with fire bridges leading into a central chamber, through which the finely divided ore passes as it falls from an upper feeding channel down the main axis of the shaft. The ore, stayed for a moment by the resistance of the gaseous currents, has time to become ignited, and in falling through the shaft the oxidation may become complete. If it contain insufficient sulphur to supply the necessary additional heat for complete roasting, the ore may be mixed with carbonaceous material. The fire-places are provided with separate flues to carry away smoke at the time of lighting; these are then closed, and an artificial draught produced by a fan, draws down the shaft the products of combustion, with the ore which is afterwards added. At the bottom the roasted ore may fall into a trough of water running through zig-zag condensing chambers, along which it is conveyed by a revolving screw to the exit door. Or it may fall upon a dry hearth, the gases passing through a water-spray condensing chamber for the recovery of dust. Or it may be received upon the hearth of a reverberatory furnace, provided with a separate grate, so that the roasting and smelting are accomplished in the same furnace, the gas here also being passed through depositing chambers. In whatever way the ore is received, and the dust is primarily recovered from the gases, the latter pass from the depositing chambers through the exhaust fan, in which they are again thoroughly admixed with spray, produced by the action of the fan upon water introduced for the purpose, to an escape shaft, the lower part of which is filled with lattice or cob-work of wood for the interception of the last traces of finely divided material which would otherwise escape.—W. G. M.

*Improvements in Melting Furnaces.* F. X. Dantzenberg, jr., Crefeld, Königshof, Germany. Eng. Pat. 6983, April 7, 1894.

A VESSEL lined with fireclay is suspended from pivots, so that it may be tipped up on a horizontal axis. On one side is a blast chamber communicating through tuyère holes with the interior of the vessel, and on the opposite side is a tap-hole on the bottom, connecting by a bent channel in the fireclay walls with an outer tap-hole half way up the side of the containing-vessel. The material to be melted may be placed (with fluxes) in alternate layers in this crucible, and when melted is poured off from below, free from admixture with slag or dross. When the substance to be fused should not come in contact with fuel, it may be contained in an

inner liner with apertures beneath, through which the liquid mass may find its way to the tap-hole. The tapping is effected by tilting the whole vessel in such wise that the tap-hole is on the under side.—W. G. M.

*Process for Treating Blast Furnace Gases for the Extraction of Alkaline and Ammonium Compounds, Iodides, and Iodine therefrom.* From O. Imray, London. P. Gredt, Esch-sur-Alzette, Belgium. Eng. Pat. 10,589, May 31, 1894.

FURNACE gases are passed through pipes of about 3 yards diameter and 25 yards length, set in pairs in basins wherein either added or condensed water furnishes a water seal, this water being pumped up from time to time into a receiver constructed in the upper part of the pipes and provided with a perforated bottom, from which the liquid falls as a fine rain, carrying the dust with it, and soluble matters in solution. There is another connected set of pipes placed horizontally, through which the partially purified gases pass. The lye drawn off is evaporated in basins, "heated by the same purified gases," and when concentrated, "containing on average 0.75 per cent. of iodine," is run into an alembic suspended in a stove in which refractory bricks are set with open spaces forming channels, to which gas and air are admitted through a number of openings, "in order by their combustion to heat the bricks and alembic to a white heat." The vapours and gases formed, containing ammonium compounds and cyanides, are passed into sulphuric acid, and the gases which escape from the receiver are taken to a Glover tower, where hydrochloric acid and cyanides are recovered. The mass remaining in the alembic is dissolved in water, and the solution is fractionally crystallised to separate calcium chloride, potassium chloride, and potassium iodide, from which latter iodine is obtained by known processes.—E. S.

*Improvements in the Manufacture of Steel.* W. P. Thompson, Liverpool. From E. Bertraud and O. Thiel, Kladno, Bohemia. Eng. Pat. 10,923, June 5, 1894.

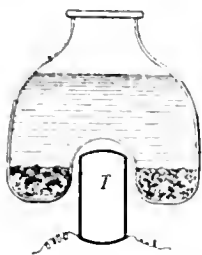
Two or more open-hearth furnaces are placed in cascade position on different levels. They may contain acid or basic linings, according to circumstances. In the uppermost furnace the silicon and some of the carbon are removed; the contents are next tapped into the middle furnace (being thus separated from the acid slag first produced) and here the metal is dephosphorised. It is lastly run, freed from the phosphoretic slag, into the lowest hearth to receive the recarbonising charge prior to tapping finally. Two furnaces on the same level may be used to treat different materials simultaneously, and the contents of the two mixed on the hearth of a third and lower one, for the addition of spiegel and ferro-manganese.—W. G. M.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

*Electrical Heating by Induced Currents.* Zeits. f. Elektro-techn. u. Electrochem. 1894, 93.

A VESSEL of suitable heat-resisting, and preferably non-conducting, material is constructed with a deep indentation in the bottom. Into this indentation is inserted the single primary coil of a coreless transformer T. Within the vessel is a ring of lead, or, as in the figure, of carbon

fragments, which becomes heated by the induced currents flowing in them, when a suitable current is passed through



the primary coil. If the vessel be made of metal it will itself act as a secondary conductor, and no additional ring of lead or carbon will be needed.—W. G. M.

*The Aim of Electro-Chemical Investigations.* F. Oettel. *Zeits. f. Elektrotech. u. Electrochem.* 1894, 35—57.

RECALLING the fact that the simple application of Faraday's law can seldom be made, except when the period of electrolysis is very short, owing to secondary reactions, such as the formation of per-sulphuric acid in decomposing copper sulphate, or of that of acid and ozone in the water voltameter, or as the decomposition of nitric acid in the electrolysis of copper nitrate, the author urges that a new era will commence for electro-chemistry when electro-chemical reactions are studied with the aid of the ampère-meter and the balance, as a new epoch began for chemistry with the quantitative examination of chemical phenomena. Since physical conditions, such as concentration and temperature of solution, current density, and rest or motion of the electrolyte, have so great an influence on the reactions of the bath that they may sometimes be made oxidising, reducing, or neutral by a variation of these conditions, it is necessary to record complete details in describing a research if the full value of the work is to be assured. In studying electro-chemical reactions, the current should be measured, and from this the theoretical result of the operation should be calculated according to Faraday's law; if then by weighing or titration this result is reached, it may be concluded that no secondary reactions have occurred. But if there be a difference beyond the range of experimental error, and this difference is found to recur on repeating the experiment, a further examination must be made into the nature, conditions, and extent of the by-reaction. Such secondary reaction may result from electrolysis of new products formed, or by their chemical interaction. For technical purposes, the conditions yielding the highest return for current expenditure must be determined. For small installations a copper voltameter may be used, made by immersing weighed copper plates or concentric cylinders in a beaker containing a solution of 15 grms.  $\text{CuSO}_4 + 5$  grms. of concentrated  $\text{H}_2\text{SO}_4$  and 5 grms. of alcohol in 100 cc. of water, the liquid being agitated by a gentle stream of hydrogen. The current to be recorded by this voltameter may range from 0.06 to 2.0 amperes per sq. dm., the result being 1.181 grms. of copper deposited per ampère hour. For larger operations an ammeter should be employed.—W. G. M.

*A Problem in Electro-Chemical Economy.* A. W. *Zeits. f. Elektrotech. u. Electrochem.* 1894, 95—96.

THE perfecting of electro-chemical processes is likely to result in the transference of many manufactures to mountainous countries where water-power is available. The actual cost of 1 h.p. as produced by water and by steam-power is with difficulty determined with accuracy, owing to the uncertainty of many factors; but for large installations it is certainly greatly in favour of the water-power. Houston and Kennedy, allowing only 2½ per cent. for depreciation, arrive at a cost of 12 m. per h.p. per annum for uninterrupted work at the Niagara 120,000-h.p. installation. For steam plant of this

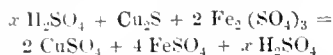
size a far larger sum would have to be written off for repairs and depreciation, and the cost would amount to from 20 to 30 times the sum named. But even such installations as would be demanded by chemical works could be run more cheaply by water than by steam. As a case in point, potassium chlorate has recently grown into an important product, owing to its use in the manufacture of safety lucifer matches. This salt was first made exclusively in England, then German manufacturers entered the field, and now about 600,000 kilos, out of the 1½—2 million kilos, required annually are made by the *Société d'Electrochimie* (of Paris) at their works in Vallorbes (Jura), where an absorption of 2,000 h.p. of water-power produces the body, by the electrolysis of potassium chloride, at a selling price of 115—120 marks per 100 kilos. An estimate of the cost of production shows that this must yield a handsome profit, and that therefore the price might be lowered to a degree which would exclude competition from German manufacturers. Meanwhile a larger installation with water-power equal to 6,500 h.p. has been made at Mansboe in Dalecarlia (Sweden); and here the promoters (*Superphosphat Aktien gesellschaft*) estimate the cost per h.p. at one-tenth of that required in England. The transference of this one small industry is of itself unimportant; but it behoves chemical manufacturers, in countries where water-power is not available, to watch the progress of electro-chemical methods, and to be prepared to meet the competition of countries in which inaccessibility and other disadvantages is far more than counterbalanced by the natural advantages of a high available water-pressure.—W. G. M.

*Electrolytic Copper Deposition in the Stolbury Lead Works.* E. Cohen. *Zeits. f. Elektrotech. u. Electrochem.* 1894, 50—55.

THE ore to be treated contained 7—8 per cent. Cu, 10 per cent. Pb, and 0.05 per cent. Ag; besides this, a matte was produced containing 15—20 per cent. Cu., 5 per cent. Pb., and 0.04 per cent. Ag. The Marchese process was first tried, in which the matte, cast into thin slabs and slowly cooled, served as anode in a bath of copper and ferrous sulphates, the ferric sulphate formed being afterwards reduced by causing it to attack fresh regulus (this *Journal*, 1885, 600). Two Siemens-Halske dynamos, of the type CF 17, gave a current of 430 amperes and 35 volts at 700—800 revolutions; the current-values per bath being 30 amperes per square metre, and 1 volt. In calculating the profit to be expected from the installation, the adverse effect of the large stock of copper locked up in the baths was considered to be far more than neutralised by the increased value of the pure copper deposited (*vide Trans. Inst. C.E., 82, 446*). At first the process worked satisfactorily, but in a few days the pressure requirements of the bath slowly increased (in one case, up to 5 volts), owing to the separation of sulphur on the anode preventing contact of the liquid with the next layer; next, the honeycombed anodes crumbled and, falling to the bottom of the baths, produced short circuits. Polarisation was at first combated, but without success, by intermittent working; then, as it was ascribed to the formation of  $\text{PbO}_2$ , the copper percentage in the anode was increased and that of the lead reduced; but neither this, nor an alteration of the iron percentage, was effectual. The deposited copper also contained traces of Sb, Bi, Pb, Fe, Zn, and S. Lead anodes were next substituted for matte, using the same electrolyte, which lasted longer, owing to the non-extraction of iron from the anodes, but polarisation was produced by the peroxidation of the lead, so that the initial voltage of 1.7 had to be raised to 2.15, while only 60 per cent. of the theoretical yield of copper was obtained. To prevent this peroxidation, sulphur dioxide, formed by burning sulphur, was blown with air into the bath. The resistance was unaffected, but the yield of copper was increased, and the metal was of high quality (99.984 per cent. Cu). One objection to this process was the excessive quantity of sulphuric acid formed, which dissolved too large a proportion of the anode, and caused crystallisation to take place in the vats. The products of roasting copper sulphide ores was too dilute, but before

erecting muffle-furnaces to obtain a more concentrated sulphur dioxide gas, the Siemens-Halske process was brought out, and received a trial.

The installation of this process (see this Journal, 1892, 534) was entrusted to Dr. Höpfner in 1888. Carbon anodes were used, and the anodes were in separate compartments with parchment-paper walls, so that the anode and cathode liquids were kept distinct. Thus, while copper was deposited on the cathode, the ferrous salts in the solution were peroxidised at the anode by the  $\text{SO}_4$  liberated; so, when the copper had been deposited from the cathode liquor, this was transferred to the anode cells, where the iron was peroxidised. It was next passed over copper matte, where it acted as follows:—



and was then available for the cathode compartments. The regeneration was effected either by mixing the ore and liquid in rotating vessels, and allowing the fine deposit to settle before use, or by running the liquid over the ore in vats with several superposed false bottoms. The baths first employed were lead-lined wooden vessels, 2·2 m.  $\times$  1 m.  $\times$  1 m., and had each 15 anodes consisting of rows of carbon rods 1 cm. thick and 45 cm. long, and 16 cathodes (80 cm.  $\times$  80 cm.  $\times$  1 mm.).

At Höpfner's recommendation, the anodes were joined up, by passing the ends, to the distance of 3 or 4 cm., through holes in a perforated copper strip, and then coating the projecting ends and the strip with copper electrolytically. The vats were placed terrace-wise, and the cathode liquid circulated from end to end, the anode liquids being transferred through the series with the aid of glass siphons regulated by screw clips. Air was blown into the anode cells, to equalise the oxidising action of the current. The siphons were not found to act well, any failure on their part causing an overflow from the anode to the cathode cell. The parchment became swollen and very tender after a few days' use, and the carbons simultaneously became very fragile, so that at the slightest provocation they crumbled, and the accumulated fragments at the bottom of the cell broke the parchment membranes. Thus resistance increased, and in nine days the voltage required rose from 0·98 to 1·75. At this time Siemens and Halske had introduced a new bath (see Höpfner's patent, this Journal, 1891, 839), in which 16 cells, separated by parchment paper supported by a wooden frame, contained alternately anodes and cathodes. Side channels permitted communication between like cells, so that the two circulations could be kept up. Air was blown into the anode compartments, and the anodes were carbon rods, as before. The result of a three-months' trial showed no improvement in working; but when a membrane was broken, the whole apparatus had to be taken to pieces, in order to replace it; while the rupture of membranes and crumbling of carbons frequently required this to be done. Possibly the breaking of the carbon may be due to the use of a voltage of 1·8 where only 1 volt was recommended, owing to the evolution of gas within the pores of the carbon. The Stolberg Company have now finished experimenting in this direction.—W. G. M.

*Electric Heating.* Zeits. f. Elektrotech. u. Elektrochem. 1894, 8—10.

See under II., page 789.

*On the Efficiency of Heating by Electricity as compared with Heating by Combustion.* A. Wilke. Zeits. für Elektrotech. u. Elektrochem. 1894, 2.

See under II., page 790.

## PATENTS.

*Improvements in Secondary Electric Batteries.* The Lithanode and General Electric Company, Limited, and J. T. Niblet, both of London. Eng. Pat. 13,217, July 6, 1893.

THE invention relates especially to "solid secondary electric batteries," such as that described in Eng. Pat. 14,411 of 1890 (this Journal, 1891, 839). In this case, lithanode materials are placed in the spaces for active material, and the formation of lithanode proceeded with. Crystals of magnesium sulphate may be added, and afterwards dissolved out, so as to leave pores or interstices.

Or the cell may be constructed with alternating electrodes of spongy lead and lithanode with diaphragms or separators between of porous clay or asbestos, or of inert perforated material.—E. T.

*Improvements in or connected with Apparatus for the Manufacture of Chlorate of Potash by Electrolysis.* F. Hurter, Liverpool. Eng. Pat. 15,396, August 12, 1893.

See under VII., page 810.

*Improvements in Apparatus for the Electrolytical Decomposition of Brine and other Liquids.* F. G. Baily and M. Guthrie, Liverpool. Eng. Pat. 15,610, August 17, 1893.

THE apparatus is designed to prevent the mingling of the substances formed at the two electrodes by the supply of fresh electrolyte and the removal of the decomposed or modified liquid. The electrolyte is fed into the cell continuously at points lying between the electrodes, and overflows are arranged near to or behind them. To assist in preventing the admixture of the products formed at the electrodes, the electrolyte around each is confined by a porous or perforated screen, through which the electrolyte flows from the centre towards the electrodes, the rate of flow being such as to prevent diffusion.—J. H. C.

*Improvements in the methods of effecting the Electrolysis of Saline Solutions.* L. A. P. and H. E. A. Liéard, Paris. Eng. Pat. 6046, September 22, 1893. (Under International Convention.)

THE electrodes are placed horizontally, the anode uppermost, a porous partition being placed between them. This partition is of peculiar construction, and specially devised so as to afford the greatest possible obstacle to re-combination and solidification, and, at the same time, the greatest possible electric conductivity.—J. H. C.

*Improvements in Primary Galvanic Batteries, and in the Liquids used therein.* W. H. Longsdorf, London.—From G. Hewitt, New York, U.S.A. Eng. Pat. 3674, February 20, 1894.

A DOUBLE fluid-cell, in which the central electrode consists of several carbon plates clamped together at the top by an aluminium bolt and nut, and furnished with a binding screw. The bolt passes also through separating pieces of aluminium sheet, which cause the immersed part of each carbon plate to be separated from its neighbours by about  $\frac{1}{16}$  in. The depolarising solution is made as follows:—Seven parts of sulphuric acid are added to eight of a saturated solution of crude sodium nitrate. The crystals of sodium sulphate formed are removed, and to the liquid remaining one-seventh part of water is added. In the zinc or zinc cell the sodium sulphate separated as already described may be used, or in its place common salt. In either case a small quantity of bisulphate of mercury is added, and, if found better, a little sulphuric acid.—E. T.

*Improvements in or in Connection with Apparatus for the Electro-Deposition of Metals.* H. Alexander, Berlin. Eng. Pat. 8917, May 1th, 1894.

The poles of the battery are arranged so as to allow the articles to be treated, to rest directly upon the cathode, or on a wire netting supported directly by it, instead of being suspended from it by separate wires as is usually done. The points of contact are changed by shifting or turning as may be required. The arrangement can be readily carried out in the form of stages at different levels in deep baths.

—J. H. C.

*Method of Bleaching Paper Pulp or other Flocky Substances.* J. W. Abom, Stockholm. Eng. Pat. 8964, May 5, 1894.

See under XIX., page 834.

*Improvements in Electrolytic Apparatus.* T. Craney, Bay, U.S.A. Eng. Pat. 9761, May 18, 1894.

A LONG tank constructed preferably of boiler-plate, is divided by a longitudinal partition into two compartments communicating at the ends. A screw is provided to insure continual circulation of the liquor. In each compartment is a row of cylindrical vessels perforated through all but the upper portion, the perforated part being over-wound with asbestos sheet to form a porous diaphragm; and that again with a "wire screen" to act as cathode. Inside this vessel is a second one of much smaller diameter, also perforated, and containing carbon to act as anode. The outer cell is closed in round the anode, and provided with an inlet pipe reaching nearly to the bottom, and an outlet pipe projecting from the imperforated portion, and passing through a stuffing box in the wall of the tank to communicate with a drain pipe outside. This outlet-pipe serves both to carry away any overflow, and to lead away the gases formed. The whole is carried on an iron cage, and is easily removable. The action is continuous, any cell becoming faulty can be removed without disturbing the rest, and be replaced by a good one. The apparatus is designed more especially for the electrolysis of common salt.—E. T.

*Improvements in Electrolytic Apparatus.* F. Craney, Bay, U.S.A. Eng. Pat. 9949, May 22, 1894.

THIS apparatus also is for the electrolysis, more especially of common salt. The outer vessel is a long shallow tank constructed preferably of boiler-plate. Into this dips a dish or tray of iron wire, the middle portion being most deeply immersed, and the sides sloping gradually upwards till they are above the surface of the liquid. This is supported from the sides of the tank by numerous suitably shaped iron bars and is covered by a layer of sand, cotton, fibrous asbestos or other material, which, while very suitable for use as a porous diaphragm, requires mechanical support. An earthenware cover fitting over the dish, converts the latter into a closed second compartment. A layer of carbon granules over the porous material forms the anode, and makes electrical connection with the dynamo by numerous earthenware tubes, forming part of the cover, which contain gas carbon electrodes. Gases are drawn from the anode compartment by means of a fan, and arrangements are made for keeping up a continuous circulation of the liquid. The iron wire tray forms the cathode.—E. T.

*Improvements in Plates for Secondary Electric Batteries.* Georges René Blot, Paris, France. Eng. Pat. 10,169, May 25, 1894.

AN improved construction of accumulator batteries of the Planté type, wherein two narrow ribbons of lead, transformed in advance, are wound together lengthwise on a stout strip of antimonial lead, of the same width as core. Both ribbons are of embossed or grooved lead, and one in addition is furnished with corrugations to allow free penetration of the electrolyte. A number of these flat bobbins

are mounted in a lead frame to form a plate, the bobbins being slightly apart to allow of swelling, and the electrical attachment being made by lugs on the core, which are burned on to the frame.

In another construction a number of strips of "transformed" lead are laid together and a supporting frame made by casting lead round them. The strips may in this case be alternately grooved "transformed" lead, and smooth hard antimonial lead, which latter will lead the current into the active material when all the lead of the first set has been practically eaten through.—E. T.

*Improvements in Electrical Accumulators or Storage Batteries.* J. Y. Johnson, London. From La Société, "L'Accumulateur Fulmen," Paris, France. Eng. Pat. 10,853, June 4, 1894.

To prevent the cases or sheaths of perforated celluloid, or such material, as that in which accumulator plates are sometimes enclosed, from swelling and bulging out at the sides, these latter are at places connected by strips or blocks of celluloid, passing through the electrode and cemented to them. In some cases small split keys are put through case and electrode, and their projecting ends expanded or separated, so as to hold the sides of the case in, and prevent their bulging.—E. T.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*Pea-Nuts and Pea-Nut Oil.* U.S. Consular Reports, July 1894, Vol. 45 [166] 378—389.

A REPORT on pea-nuts and pea-nut oil, as regards occurrence, modes of extraction, exports, prices and methods of production, and utilisation of residues and refuse, in the following countries:—Algiers, Bordeaux, Marseilles, India, Mozambique, and Senegal. (This Journal, 1894, 530—531.)—W. S.

*Pea-Nut Oil.* U.S. Consular Reports, through "Chemist and Druggist," August 18, 1894.

THE report of the American Consul at Marseilles contains some further facts concerning the manufacture of pea-nut oil, an oil which is largely coming into use for various economic purposes (see also this Journal, 1894, 530). Extraction of oil from pea-nuts is rapidly increasing, no fewer than 17 factories being at present engaged in the industry, and the quantity of nuts imported at Marseilles for this purpose during 1893 exceeding by 314,000 metric quintals (69,224,400 lb.) the importation for 1892. The general method of producing the oil is as follows:—On arriving at the factory the pea-nuts are first placed in a machine of the nature of a "winnowing," in which all outside dirt and other foreign substances are removed. Having been thus superficially cleansed, the nuts are conveyed by an Archimedes screw to the shelling-machine, where they are deprived of the shells. Thence the nuts fall into the first triturating-machine, consisting of a pair of cast-iron rollers, where they are coarsely ground, and at the same time any foreign bodies, stones, &c., are by an ingenious arrangement rejected. From this machine the meal passes to another, where it is again ground finer, and thence into a long hexagonal case forming a sieve, through which the fine meal passes, while the coarse is sent back to the rollers again. The meal is then pressed in "scourtins" made of horsehair, a pressure of 2,850 lb. to the square inch being exerted and left on for an hour, which is sufficient to extract all that can be obtained in the first yield. The meal is then removed from the "scourtins," ground a second time, heated to a temperature of about 70° C.

(158 lb.), and a second pressing is effected. If oil of a very fine quality is required the nuts are crushed only once, partially-ground nuts yielding a smaller but finer product. The yield varies according to the quality of the nuts. Mozambique nuts produce about 50 per cent. in the first pressing, and the value is from 70 f. to 95 f. per 100 kilos.; the second pressing yields about 12 per cent., the value of which is from 45 f. to 50 f. per 100 kilos.

The oil is largely devoted to the manufacture of white soap, for which it is highly prized. It is also used as salad oil and in the composition of margarin. Large quantities are also sold as olive oil, principally in the United States. A smaller amount is used for illuminating purposes. The cake left after pressing is particularly rich in nitrogen and forms an excellent cattle food, and commands a price of 13 f. per 100 kilos, if made from shelled nuts.

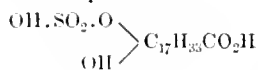
#### *The Constitution of Turkey-Red Oils.* P. Juillard.

Bull. Soc. Chim. 11, 1894, 280—286.

THE different products formed by the action of sulphuric acid on ricinoleic acid are as follows:—

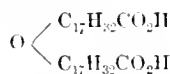
1. *Ricinoleic sulphuric acid*,  $\text{OH} \cdot \text{SO}_2 \cdot \text{O} \cdot \text{C}_{17}\text{H}_{33}\text{CO}_2\text{H}$ , is a dibasic acid, stable only in dilute solution, concentrated solutions readily decomposing into sulphuric and ricinoleic acids. It has the characteristic property of forming acid salts when treated with solutions of almost any salt, e.g., on agitating its solution with potassium chloride, acid potassium "ricino-sulphate" and HCl result. The corresponding sodium salt was formerly erroneously described as diricinolein-sulphuric anhydride.

#### 2. *Dihydroxystearo sulphuric acid*—



almost always accompanies (1) and possesses similar properties

#### 3. *Dibasic diricinoleic acid*—

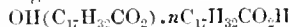


A thick liquid soluble in alcohol. Its methyl and ethyl salts, however, are almost insoluble in alcohol, and on this property is based a method of separation of this from other acids, particularly from isoricinoleic acid. It is very stable, and is not saponified by boiling alkalis.

#### 4. *Monobasic diricinoleic acid*—



This is the first member of the series of polyricinoleic acids having the general formula—

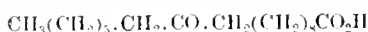


When castor oil is treated with sulphuric acid, in the proportions of one part acid (98 per cent.) to eight parts of castor oil, the product consists almost entirely of these polyricinoleic acids, while with a larger proportion of sulphuric acid, isoricinoleic and dibasic diricinoleic acids chiefly result.

5. *Dioxystearic acid*,  $\text{C}_{15}\text{H}_{26}\text{O}_4$ , is found in small quantity in crude isoricinoleic acid, it melts at  $66^\circ$ — $68^\circ\text{C}$ ., and is very soluble in alcohol and ether.

6. A solid acid which melts at  $70^\circ\text{C}$ ., and is soluble in alcohol and ether, answers to the formula  $\text{C}_{36}\text{H}_{70}\text{O}_7$ , and appears to be a molecular compound of dihydroxystearic and ricinoleic acid.

7. *Isoricinoleic acid*,  $\text{C}_{15}\text{H}_{34}\text{O}_5$ .—An oily liquid soluble in alcohol and ether. It is distinguished from ricinoleic acid by its solubility in petroleum ether; and appears to be a ketonic acid with formula—



or, perhaps—



Experiments made with the Turkey-red oils resulting from the action of sulphuric acid on oleic acid and olein, have shown that, starting from oleic acid, the first product is hydroxystearosulphuric acid  $\text{OH} \cdot \text{SO}_2 \cdot \text{OC}_{17}\text{H}_{33}\text{CO}_2\text{H}$ , from which is formed hydroxystearic acid  $\text{OH} \cdot \text{C}_{17}\text{H}_{33}\text{CO}_2\text{H}$ . In an analogous manner to ricinoleic acid, this acid condenses to form polyhydroxystearic acids.

Olein treated in the cold with sulphuric acid yields two acids, one mono-, the other dibasic, which appear to be addition products of sulphuric acid and olein. They are soluble in water, and readily form sodium and potassium salts.—R. B. B.

*The Analytical Constants of Seal Oil.* A. C. Chapman and J. R. Rolfe. Chem. News, 1894, 70, 1—2

See under XXIII., page 843.

*Margarin Compared with Natural Butter as regards Digestibility and Nutritive Value.* A. Jolles. Monatsh. Chem. 15, 147—163.

See under XVIII. A., page 827.

*New Method for Estimating Pychnometrically the Densities of Soft Fats.* Zdzislaw Zawalkiewicz. Monatsh. Chem. 15, 132—138.

See under XXIII., page 839.

### PATENTS.

*Improvements in the Treatment of Cotton-Seed for the Removal of Fibrous Matters therefrom and in Apparatus therefor.* W. P. Thompson, Liverpool. From W. H. Stead, Geneva. Eng. Pat. 11,361, June 9, 1893.

THIS is described as an improvement on previous patents, 13,831, 1884 (this Journal, 1885, 585), and 10,992, 1887 (this Journal, 1888, 433). The improvement in the process is stated to consist in the treatment of the cotton-seed, for the removal of fibrous materials, with hydrochloric acid in a heated kettle, with constant stirring, adding oil or grease when desired, and separating the dust by friction and sieving with or without aspiration. The improvement in the apparatus consists in a stationary jacketed pan with revolving stirrer in combination with a sieving device. Also in a series of pans one below the other, having several stirrers and means for introducing acid at or near the top, and alkali or oil in the lower parts of the single pan or the lower portion of the series of pans, whereby the lint can first be destroyed by the acid and then the acid neutralised prior to its arrival at the sieving arrangement.—E. G. P. T.

*Improved Inspissated Oil for Electrical Insulating Purposes, and Method of producing same.* A. Geuttsch, Vienna, Austria. Eng. Pat. 16,597, September 4, 1893.

THIS is said to be a new article of manufacture, prepared by letting water containing salts in solution (such as carbonate of lime, carbonate of magnesia, magnesium hydroxide, sulphate of lime, oxide of iron) drip from a suitable height into the oil or fat, placed in a suitable boiler, and retained at a temperature of more than  $100^\circ\text{C}$ ., until the desired consistency is obtained for such inspissated or "concentrated" oil.—J. C. C.

*An Improved Process for Removing the Green Colour from Paraffin, Vaseline, and Lubricating Oils, which have had that Colour Imparted to them by Bodies containing Oxygen.* A. Wendtland, Berlin, Germany. Eng. Pat. 9179, May 9, 1894.

To illustrate the process, paraffin, vaseline, and lubricating oils, which have undergone a preliminary purification by



sulphuric acid, are taken as an example. These residues are coloured green. To remove the colour the mass is thoroughly mixed with soap solution in a finely-divided condition, in a manner described in detail. This soap solution is then separated by settling or by centrifugal force, and the purified products washed with hot water and a weak solution of barium chloride. The soap solution contains the colouring matter; the fatty acids, being separated by dilute mineral acid, may be used again. In connection with this treatment, a method of bleaching the paraffin or vaselines, &c., which have assumed a green colour through being treated with bone-black, is described, "by means of a treatment with methyl or ethyl alcohol, aldehydes, ketones, &c., either before or after the treatment with bone-black."

—J. C. C.

### XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

#### (A).—PIGMENTS, PAINTS.

##### PATENT.

*Improvements in the Manufacture of Plumbago or Graphite, commonly called Black-Lead.* P. F. Johnson, Liverpool. Eng. Pat. 10,424, May 29, 1894.

GRAPHITE crushed and passed through a sieve of from 120 to 150 meshes per inch, is stirred into a saturated solution of alum or aluminium sulphate at 212° F.; stearite is then added, and more water if required. After mixing, excess of water is evaporated until a consistency suited to grinding in a chilled steel or other mixer is obtained. More graphite may here be added; then, after thorough grinding, the material may be compressed into cakes for household use, or is ready for the manufacture of pencils or crucibles. The average formula for the mixture is: graphite, 80; stearite, soapstone, or talc, 14; alum, 6; but this varies with the purpose to which the material is to be applied. When several different kinds of graphite have to be employed, the richest in carbon is first mixed into the alum solution. By this process graphites previously regarded as incapable of being compacted are utilisable, and are improved in polishing power; for pencils, the material may be hard without being brittle, and black without being soft; while crucibles made from the treated graphite are at once harder, more durable, and lighter.—W. G. M.

#### (B).—RESINS, VARNISHES.

*An Investigation of the Resins: The Gum Benzoin of Siam.* F. Ludy. Arch. d. Pharm. 231, 461.

THIS substance, whose exact botanical source is unknown, contains, according to various authors, benzoic and cinnamic acids, styrolene, and vanillin; while the present investigator finds in it two alcohols—benzoresinol and siarresinotannol—combined with benzoic acid, traces of a very aromatic benzoic ether, vanillin (0.15 per cent.), and benzoic acid.

*Siarresinotannol* ( $C_{15}H_{14}O_4$ ) is obtained, as an amorphous brown mass, by dissolving the gum benzoin in dilute soda, precipitating the sodium derivative of benzoresinol by ether, and evaporating the filtrate. It forms a potassium derivative ( $C_{15}H_{13}O_4K + H_2O$ ), and an acetyl derivative ( $C_{15}H_{13}O_4 \cdot COCH_3$ ), both amorphous. Nitric acid yields with it picric acid when concentrated, and when dilute phlophaphene.

By precipitating the ethereal extract of this gum-benzoin with petroleum ether, the benzoates of benzoresinol and siarresinotannol are readily obtained, and of the following composition:—Benzoic acid, 38.20 per cent.; benzoresinol, 5.1 per cent.; siarresinotannol, 56.7 per cent.

Siam benzoin therefore differs, as to its composition, from Sumatra benzoin.—E. R. B.

##### PATENT.

*Improved Insulating Material for Electrical Purposes, and Method of Producing same.* A. Gentsch, Vienna. Eng. Pat. 16,851, September 7, 1893.

ACCORDING to this invention mineral resins, more especially ozokerite, asphalt, and amber, are subjected to distillation in a closed still to a temperature of 100°. After the mass has been heated until gases, vapour, or oils cease to escape, it is allowed to cool. In that condition it is of pliable consistency, and may either be used alone or incorporated with other substances for insulating cables, such as resins, fats, or oils, the proportions of these being from 30 to 70 per cent. By this method the inventor proposes to remove those substances present in fossil resins, which prejudice or deteriorate the insulating properties.—D. B.

### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

*Leather and Glove-making Industry in Spain.* J. Soc. Arts, 42, 1894, 766—767.

SKINS used for the manufacture of gloves in Spain are kid for the first quality and lamb for the second quality. The United States Consul at Cadiz says that the skins are placed in a lime-water bath for four or five days, after which they are taken out and extended upon a bench called *caballete*, and with a curved knife that has no cutting edge, known as the *descarnador*, are depilated, then turned over, and by means of another knife of dull edge the flesh is cleared from them. After this the skins are worked with another unsharpened knife, then stamped with a rammer and rasped again. They are then put into a bran-water bath until they float on the surface, when they are taken out and the bran removed by means of a slightly-sharpened knife, which is passed over the skin from side to side. When thus cleansed they are placed in a large earthen vessel containing beaten yolks of eggs and wheat-flour, and agitated for a short time, then alum solution is poured into this bath in the proportion of three pounds for every 24 skins. In this wash they are macerated with the feet for two or three hours, after which they are taken out, washed, pressed to remove the water, and placed to dry. The skins are prepared for colouring by rasping and softening them, placing them upon a lead table, and saturating them with urine or potash. Aniline dyes are employed to produce the colours, and the brilliancy is obtained by means of polishers. Russia-leather is sometimes used for making gloves. Its preparation commences like that of the common leather, by removing the grease and depilating the skins with lime. They are afterwards macerated in a bath of fermented rye-flour dissolved in water, and they are kept in this bath for two days. They are then cleaned and washed in running water. The tanning is performed by employing the bark of the birch, pine, or willow trees, in which decoction the skins are submerged and agitated twice a day for a fortnight and sometimes for as long as six weeks. After this they are rubbed and scraped upon boards, and once cleaned, extended on a table, and impregnated on the flesh side with the oil of birch-bark tar, and then immediately stretched, in order to give them the necessary flexibility. Once saturated with this oil, the hair side is bathed with a solution of alum, then dried, and a fluted cylinder passed over it. It is now ready for colouring. The red colour of Russia-leather is obtained by boiling red sandalwood and logwood in lime-water, containing a small quantity of carbonate of potash and oxide of sodium, which infusion is



poured into sacks, formed by two skins sewn with the hair side inwards. The liquid is kept inside, until it penetrates the skin. The colour can also be applied with a brush, giving the skin continual coats of the infusion named above. When the skins are coloured, the ordinary methods are proceeded with, to soften and polish. Knapp-leather is prepared by two methods: the first consists in submerging the skin in a bath containing 5 per cent. of lime, where it is kept until well impregnated, then taken out and left to drip: it is afterwards placed in another bath, composed of 3 to 5 per cent. of potash soap, in which it is tanned. The second method, by which better results are obtained, consists in mixing an alum solution with the soap, thus obtaining a compound, in which the skin is kneaded; after this it is washed and dried. The Knapp-leather obtained by means of the alum soap has a white surface, and is soft and agreeable to the touch. White leather is prepared from kid, goat, and sheep skins, by submitting them to the preparatory operations of tanning, lime being employed for cleaning and depilation. The skins are well washed, to remove the grease, and, with the object of effecting this and of swelling them, they are placed, after being cleansed, in a bath of oat-water, where they are kept for two or three days; they are then taken out, washed, and wrung out by twisting. They are then immediately put into the tanning bath, which is composed of 750 grms. of alum, 30 grms. of marine salt, and about 22 litres of water for every 10 skins. To operate with it, a hot mixture is placed in a tub, and when it begins to cool, a skin is submerged in it once or twice until it is well soaked; it is then taken out and the others treated in the same way. They are then placed, without drying, one over the other, and are left for two or three days. They are afterwards twisted and hung up to dry on long poles, called *pertigos*. During this drying, the skins become wrinkled and very rigid. In order to lengthen them and to remove the wrinkles, they are rubbed longitudinally against the convex part of a curved bar. The preparation of glove-leather requires especial tanning, every operation being performed in an exceedingly careful manner, so that the skins may be completely cleaned, softened, and given that elasticity necessary to adapt them to the form of the hand, and to always preserve their grain intact. The preliminary operations of preparing this leather are the same as those indicated for white leather, the tanning being different. When prepared, they are introduced and macerated with the feet in a bath composed of wheat-flour, yolk of eggs, alum, marine salt, and water in a sufficient quantity to form a sort of a light paste, which acts in the following way. The flour swells the skin by the combination of its glutinous parts with the alum absorbed by it, the yolk of the eggs acts not only by its albumin, but also by the oil which it contains, and gives to the skin the softness and resistance necessary for leather intended for gloves. Once well soaked in this paste, the skins are taken out, and, after drying, slightly moistened and stretched as rapidly as possible. They are then placed by dozens inside a cloth, and pressed by the feet, to soften them. They are then submitted to the polisher, by whom they are stretched and polished. This operator removes the last vestige of flour which may remain. They are then whipped and beaten to give them softness. When the leather is intended for the manufacture of embroidered gloves, it is polished with a glass polisher on the hair side, or covered with a light bath of albumin, Senegal gum, or soap. A dozen tanned kid-skins cost from about 20s. to 25s., and these will make from 20 to 21 pairs of gloves. A dozen tanned lamb-skins cost about the same, but will make from 44 to 48 pairs of gloves.

#### PATENT.

*Improvements in the Art or Process of Tanning Hides or Skins.* W. Zahn, Newark, U.S.A. Eng. Pat. 24,463, December 19, 1893.

To produce a waterproof, soft, and elastic leather, in quality superior to those produced by other systems of tanning,

the skins prepared as usual for tanning are left for from one to three hours or more, according to their thickness, in a weak ( $\frac{1}{2}$  per cent.) solution of chromic acid, and are then drained. They are now transferred to a bath of chromo alum (or other chromic oxide salt) containing 70 or 80 lb. of the salt per 1,000 lb. of skin and sufficient water to cover the latter. Here, with the aid of a paddle-wheel, the sulphide of an alkali or alkaline earth is mixed in with the solution, with the result that the chrome alum is converted into a basic salt; and the chromic oxide reduced from the chromic acid in the skin, forms chromium hydrate. In from 10 to 15 hours the hides may be removed from this bath, washed, and finished in the ordinary manner.

—W. G. M.

#### XV.—MANURES, Etc.

*The Volumetric Estimation of the Soluble Phosphoric Acid in Superphosphates.* W. Kalmann and K. Meissels. Mitt. d. k. k. tech. Gew. Museums, 4, 1894 (4,556), 174.

See under XXIII., page 842.

*A Problem in Electrochemical Economy.* A. W. Zeits. f. Elektrotech. u. Elektrochem. 1894, 95.

See under XI., page 817.

*The Determination of Phosphoric Acid.* W. J. Williams, F. Bergami, and B. Terne. J. Franklin Inst. 137, 126, 129, and 138.

See under XXIII., page 842.

#### PATENTS.

*Improvements in the Treatment of Sewage Sludge.* R. C. Tanner, Chester. Eng. Pat. 13,305, July 8, 1893.

See under XVIII. B., page 832.

*A Fish Extract or Essener, and in Utilising the Residual or Waste Products Resulting from the Production of the same.* W. Knebel and F. E. Sahlfeld, both of Hannover, Germany. Eng. Pat. 15,595, August 17, 1893.

See under XVIII. A., page 829.

#### XVI.—SUGAR, STARCH, GUM, Etc.

*Phoma Betae, the Beetroot-destroying Organism.* Frank. Zeits. Ver. Rübenzuck.-ind. 44, 264.

The author contends that this pest, which was observed during 1893 in almost every part of Germany, is not identical with "*Sporidesmium putrefaciens*" or "*Peronospera Schachtii*," which Kühn recognised 20 or 30 years ago as the cause of the core-rot (compare, however, *Hollrung*; this Journal, 1894, 264).

It is now positively established that the organism exists in the seeds, and may be the cause in some instances of mildew in the young beetroots. The threatened infection by *Phoma Betae* is only to be avoided by disinfection, a fact likewise recognised by Hellriegel and Klarson. As observed by Hellriegel, carbolic acid is effective as a disinfectant, but it weakens the germinative power of the

seeds. The use of the following mixture is recommended.—Copper sulphate (4 kilos.) is dissolved in water (100 litres), and into the solution is stirred the paste resulting from 4 kilos. of freshly-slaked lime. With this liquid the seeds are thoroughly moistened and allowed to remain therewith for 18 hours, when they are washed; thorough washing prior to sowing is unnecessary. It is well in the case of fields infected with *Phoma* to sprinkle the soil with the above-described mixture, which does not harm the roots in any way; if any diseased roots are discovered it is best to remove them.—A. R. L.

*Estimation of Sugar in Beetroots.* Kroecker. Zeits. Ver. Rübenzuck.-ind. 44, 322.

THE author finds that both cold and warm digestion of the roots, as also when sufficient basic lead acetate was added to the juice to produce alkaline reaction, leads to results which are too high. Cold alcoholic digestion yields too low results; warm alcoholic extraction, however, gives the same values that are obtained by Scheibler's extraction process.—A. R. L.

*Galvanised Iron Sugar-Moulds with an Internal Coating.* E. Donath. Zeits. angew. Chem. 1894, 232.

THE drawbacks attending the use of janned and galvanised sugar-moulds have been successfully overcome in a new mould, which is made of galvanised iron provided with an internal coating. This coating consists of a strong solution of sodium silicate mixed with china clay and either finely-ground dolomite or a mixture of chalk and magnesite. The moulds are coated with this mixture and dried at about 60° C. In using these moulds for the first time they should be filled with erude sugar-liquor (*massecuite*); subsequently they can be used for refined liquor. It appears that the small quantity of lime contained in the former renders the coating resistant to the action of water. This coating of the moulds is renewed from time to time.—C. O. W.

*Evaporation and Boiling of Sugar-Liquor (Beetroot-Juice).* J. Lexa. Zeit. Zuckerind. Böhm. 18, 258—275.

THE author criticises adversely several portions of the latest edition of Stohmann's *Handbuch der Zucker-fabrikation*. Stohmann is said to have erroneously described the Rillieux multiple-effect evaporating apparatus first constructed in 1848. The author further asserts that Peelet, Otto, Walkhoff, and Stammer have all completely misunderstood Rillieux' drawings and his description.—A. R. L.

*The So-called Scum Fermentation in the Javanese Sugar Factories.* Prinsen Gurlings. Zeits. Ver. Rübenzuck.-ind. 44, 297.

THE above-named phenomenon consists of a decomposition of the degradation products resulting by the action of lime at the boiling temperature on invert sugar; it must therefore specially relate to the decomposition of glucinic acid, which latter in the free state or in hot acid solution decomposes into acetic, formic, and humic acids together with carbonic anhydride. The glucinic acid can be destroyed by rendering the thick liquor or green syrup faintly acid with sulphurous or phosphoric acid and boiling, the scum being subsequently skimmed off. It is, however, preferable to prevent the formation of glucinic acid, and this can be accomplished by defeccating the cane-juice with 2—3 per cent. of lime at a temperature not exceeding 60°, and saturating with cold carbonic anhydride, whereby the saccharine and likewise the invert sugar is decomposed. Practical experiments on a large scale showed, however, that when either alternative is adopted scum fermentation no longer occurs.—A. R. L.

*The Invertive Action of Glycerol.* E. Donath. J. prakt. Chem. [2], 49, 546—548.

REFINED sugar was dissolved in ten times its weight of pure commercial glycerol of sp. gr. 1.2556 at 17.5° (thus

containing about 97 per cent. of glycerol), and the solution heated in an open dish for half an hour at 130°. The liquid after dilution now showed, on being heated with Fehling's solution, cupric reduction equivalent to 4.2 per cent. of sugar inverted. The experiment was then carried out using the same glycerol diluted with 10 per cent. of water; the quantity of inverted saccharose indicated by the copper test was in this case 7.3 per cent. In a third experiment, in which the glycerol was diluted with 20 per cent. of water, 15.3 per cent. of inverted saccharose was indicated. When the sugar was dissolved in the last-mentioned glycerol containing 20 per cent. of added water, and the solution was heated in a Lintner's pressure flask at 120—130°, it appeared that more than 60 per cent. of the sugar was inverted. By heating a solution of sugar in aqueous glycerol contained in a Lintner's flask at 160° for several hours, the product was lavo-rotatory and of a dark colour, showing that considerable decomposition had taken place. The levulose had probably undergone reversion to some extent.

When heated with glycerol to which 10 per cent. of water has been added, "dextrin" does not appear to be converted into maltose, but a partial conversion of maltose into dextrose is indicated by the increase in the reducing power and decrease in the rotatory power; the inversion of milk-sugar also attains a higher limit than that of maltose, but is nevertheless far from complete. Raffinose is scarcely altered by aqueous glycerol at ordinary pressure, but when heated with it in a Lintner's flask at 125° considerable inversion takes place. Taking into account the fact revealed by these experiments, viz., that glycerol containing 10 per cent. of added water has a greater hydrolytic power than the original substance containing only a slight amount of water, and that an addition of 20 per cent. of water still further enhances the hydrolytic power; also the well-known fact that glycerol combines with water at the ordinary temperature, and again parts with it when heated, the author suggests that it is perhaps this water of hydration in "*statu nascendi*" which brings about the hydrolysis of the sugars.—A. R. L.

*Comparative Defecation Experiments with Beetroot-Juice.* A. Herzfeld. Zeits. Ver. Rübenzuck.-ind. 44, 278.

ACCORDING to the author the beetroot-juices employed in 1893 were better than those of 1892, and when the former were heated at 90° C. for a long time, only a slight amount of invert sugar was formed. The juices were treated at 90° C. in suitable apparatus provided with mechanical agitators, with slaked lime or with caustic lime, and then saturated. The experiments show that the use of milk of lime of 20° Beaumé, which is the plan adopted in practice, is free from reproach, whilst defeccation with dried slaked lime may render the saturation difficult; it is, however, shown that dry defeccation proceeds quicker, better, and more completely, and also renders possible a quicker saturation, probably because in consequence of the formation of calcium saccharate more lime is dissolved. These results are not, however, to be regarded as final, but will be confirmed by comparison with other methods of moist defeccation. Experiments in which the defeccation was conducted in the cold yielded very good liquors free from invert sugar, but the nature of the mud obtained in these cases increases the difficulty of filtration to such an extent as to render the process an impossible one.—A. R. L.

*Purification and Decolorising of Beetroot-Juice and Sugar Solutions.* Steffen and Drucker. *Sucr. indigène*, 43, 501.

THE liquor from the filter-presses is cooled to 30—35° C. and treated with 80, until it contains 1—2.5 grms. per litre; the cold solution is then filtered over 1—2 per cent. of animal charcoal, the filtrate neutralised with lime or other bases, and after mechanical filtration the liquor is boiled. Even when animal charcoal is entirely dispensed with, the liquor is said to be so pure and colourless that "refined *massecuite*" can be directly obtained from it. The green syrup from the latter, moreover, possesses the

ordinary quality of that obtained in the refinery. The same results can be obtained by similarly treating refinery syrups or solutions of raw sugars by the above-described process.

—A. R. L.

*Tables showing the Amounts of Coal and Steam required in the Manufacture of Sugar.* Claassen. Zeits. Ver. Rübenzuck.-ind. 44, 173.

THESE tables, which the author has calculated for the coal and steam required for the evaporation, boiling, and warming of the juice (not for the entire need of the manufacture), show at a glance the efficiency of the various evaporating and heating plants, and are therefore of great value for comparative calculations. The values are not, of course, of universal application, but generally by means of a calculation the amounts of steam and coal required for any description of apparatus, as well as the cost of the same, may be approximately arrived at. The important fact revealed by the tables is that in multiple-effect plant an increase in the number of members beyond 4, or at most 5, is not attended with profit, and that such evaporating plant as that of Rillieux and of Lexa may be classed among the most efficient.—A. R. L.

*Fermentation of Molasses.* J. Effront. Monit. Scient. 8, 161.

THE author arrives at the result that the non-fermentation of the whole of the sugar present in molasses is to be traced to the presence of certain bacteria. The action of these can be arrested by treating the molasses with a sufficient quantity of antiseptic agents or acids, which must not, however, be also enough to arrest the fermentation of the yeast.

For this purpose, tannin (25 grms.) is added to molasses (100 kilos. diluted to 18° Balling°); after subsiding, the clear liquid gave the normal yield of alcohol. Pieric acid (25–30 grms.) to molasses (100 kilos.) also gave good results; it must, however, be used with great caution on account of the poison which it imparts to the residues. The simplest and most convenient substance to remove these bacteria appears to be egg-albumin; the quantity obtained from three to four eggs is sufficient for 100 kilos. of molasses. Alumina and also hydrofluoric acid serve the same purpose.—A. R. L.

*Gravimetric Methods for Estimating Sugars by their Cupric Reducing Power.* L. Grünhut. Chem. Zeit. 18, 447.

See under XXIII., page 845.

#### PATENTS.

*Improvements in the Method of Separating Metals from Ores and from other Mixtures of Insoluble Substances therewith.* E. R. Besemfelter, Grosse, Mochbern, Silesia. Eng. Pat. 23,619, December 8, 1893.

See under X., page 815.

*Process for Purifying and Decolorising Saccharine Juices and Solutions.* B. Lach and H. Benies, Vienna, Austria. Eng. Pat. 8463, April 28, 1894.

THE process consists in treating the saccharine juices and solutions (1) with the products obtained by heating to incandescence, with exclusion of air, animal refuse (blood, flesh, hide, horn, hair, &c.), mixed with 5 per cent. of carbonate of potash, carbonate of soda, caustic potash, or caustic soda; or (2) with the waste products resulting from the manufacture of ferrocyanide of potash.—A. R. L.

\* In the Balling areometer  $d = \frac{200}{200 - n}$  where  $d$  = specific gravity and  $n$  = weight of water displaced when the instrument sinks to a certain division of the scale. In cases of liquids lighter than water the formula becomes  $d = \frac{200}{200 + n}$ .

*Improvements in the Manufacture of White Sugar and the like from Molasses, and in Centrifugal Apparatus for use therein.* E. Hübner, Domaglice, Bohemia, Austria. Eng. Pat. 9436, May 12, 1894.

THE patentee claims a process for the manufacture of white sugar and the like from molasses in centrifugal machines fitted with plates disposed radially, or in parallel positions with perforated cylinders, these plates being fitted with flanges to regulate the dimensions of the cells thus formed. The drum of the centrifugal is charged with molasses, and into it is led a jet of steam from a pipe. The general construction and combination of the several parts of the apparatus employed is also claimed.—A. R. L.

*A Process for Deodorising and Bleaching Dissolved or Fused Amylaceous Materials.* C. D. Abel, London. From L. König, Berlin, Germany. Eng. Pat. 9674, May 17, 1894.

OZONE or ozonised air, prepared by passing oxygen or air through apparatus in which there is a silent electrical discharge produced by alternating high-tension currents, is blown from fine apertures through the liquid (solutions of soluble starch, dextrin, or gum); or the latter is passed in fine streams through a space charged with ozone. The quantity of ozone employed is such that, after acting once or several times on the liquid, it is mostly absorbed; the action is continued until the desired effect is produced.

—A. R. L.

*Improvements in the Production of Dextrins and Leiocomes.* C. D. Abel, London. From C. Pieper, Berlin, Germany. Eng. Pat. 9675, May 17, 1894.

THIS invention relates to a process of obtaining dextrins and leiocomes from starch by the action of ozone or of ozonised air. The starch (purified amyllum, fecula, &c.) is impregnated in the usual manner with acid—preferably hydrochloric acid—and allowed to dry. It is then heated in suitable roasting apparatus provided with inlet and outlet tubes. While thus heating the starch to the usual temperature to convert it into dextrin, a current of dry or moistened ozone is passed into the cylinder through the said inlet or inlets, leiocome being produced. The principal reaction takes place while the material is giving off aqueous vapour. Dextrins and leiocomes prepared by the above process are said to be clearer and brighter than those at present in the market; besides which they have, when dissolved, a pleasant odour and taste instead of the well-known repugnant odour given off under like circumstances by the ordinary commercial products.—A. R. L.

#### XVII.—BREWING, WINES, SPIRITS, Etc.

*The Specific Character of the Fermentative Functions of Yeast Cells.* A. J. Brown. Proc. Chem. Soc. 1894 [141], 159.

THE usually accepted view as to the cause of the exhibition of the fermentation functions of yeast cells is that it is a starvation phenomenon brought about by want of free oxygen during the life of the cells in a fermentable liquid; or, more briefly, that it is a phenomenon of "life without air." This view was originated by Pasteur, a full account of the experimental results which led him to formulate this theory being given in Chapter VI. of his "Études de la Bière."

In a paper on the influence of oxygen on alcoholic fermentation (this Journal, 1892, 237), attention was called by the author to the fact that some of the experimental results described appeared to be contradictory to Pasteur's

theory. Further work on this subject has led him to think that a review of the experiments on which Pasteur's theory rests, in conjunction with the results of some of his own, will not be out of place at the present time.

Pasteur's experiments may be divided into two classes, one containing experiments which appear to have suggested his theory, the other containing experiments undertaken for the purpose of proving its truth. It is not proposed to discuss the experiments in the first class, as, although they demonstrate most important facts regarding the life and growth of yeast, they afford no proof that fermentation is a consequence of "life without air."

The attempted proof of this theory lies in those experiments by which Pasteur determines, under varying conditions of aëration, the proportion of the weight of the yeast formed to the weight of sugar fermented. This ratio, or proportion, of yeast to sugar is, Pasteur considers, an expression of fermentative power; and when considering the ratios established by different experiments, he treats them as comparable with each other. By thus comparing fermentative powers of yeast cells under varying conditions of aëration, he arrives at the conclusion that when aëration is perfect, fermentative power ceases, and when aëration is reduced, fermentative power increases; the proof of Pasteur's theory, indeed, rests upon this conclusion.

As so much turns on Pasteur's interpretation of the experimental results by which he determines fermentative power, it is desirable to test it as thoroughly as possible. The ratio of yeast formed to sugar fermented in any experiment is an expression of fermentative power; but if it is found desirable to compare fermentative powers determined from the results of two or more experiments, it becomes necessary to make sure that either the total fermentative power, or some known fraction of it, has been measured in each case, otherwise no true comparison can be made.

If the amount of yeast formed during fermentation were in direct proportion to the sugar fermented, the ratio of yeast to sugar would remain constant, however much or little sugar were available; but experiments made by the author show conclusively that such is not the case. The amount of sugar available during fermentation is a factor that has but little influence on yeast increase; *there is, in fact, no direct proportion between weight of yeast formed and sugar fermented.* In order to show that the total fermentative power of yeast has not been measured in Pasteur's experiments, a fermentation was carried on under aerobic conditions until the sugar originally present was decomposed. Afterwards, using the principle of overcrowding as a means of preventing reproduction, the crowded cells were fed with more sugar. Feeding was carried on at intervals until the yeast had fermented three times the amount of sugar originally present at the beginning of the experiment, but no increase in the weight of yeast took place. Thus the fermentative power of the yeast was found to be at least threefold what it would have been if determined in the manner adopted by Pasteur.

Apart from the fact that Pasteur fails to prove the truth of his theory by comparing fermentative powers, his hypothesis is further weakened by the fact that none of the results of his experiments are contradictory to a hypothesis opposed to his. The aerobic and anaerobic life-history of yeast, as demonstrated by Pasteur, can be accepted without the truth of his hypothesis regarding fermentation being admitted. There is no *prima facie* reason why the fermentation functions of yeast cells should not be exercised independently of the cells' environment, so far as the presence or absence of free oxygen is concerned; and nothing in Pasteur's experiments contradicts this.

#### Utilisation of Waste Carbonic Acid Gas in Breweries. A. Marcet, Trans. Inst. Brewing 7, 199—223.

This paper gives a historical sketch of the development of the plan (first carried out at Guinness' Brewery in 1890) of collecting and compressing the carbonic acid produced in breweries during fermentation and a description of the apparatus and method employed. (See also XXIII., page 844.)

The quantity of carbonic acid evolved during fermentation is approximately equal in weight to the alcohol produced. The rapid evolution of carbonic acid begins in breweries about 20 hours, in distilleries about 6 to 8 hours after "pitching," and for some time after that the evolution is rapid and regular enough to allow the gas to be drawn off by the compression pumps without fear of drawing in air. If air is drawn into the compression pump it can be got rid of by a special release valve. For collecting the gas the ordinary parachute or skimmer is used with raising and lowering gear, allowing it to be kept just above the head of the yeast. The level of the carbonic acid in the vat is ascertained by an ordinary collodion air balloon which floats on the surface of the carbonic acid. The collection is stopped as soon as the evolution of gas becomes slow.

The compressor used is of a three stage type, the gas being purified after the first stage (15 lb. compression) by passing through water, strong sulphuric acid, and a weak solution of permanganate of potash. For purification 100 lb. of carbonic acid require  $5\frac{1}{2}$  lb. of sulphuric acid,  $\frac{3}{4}$  oz. of potassium permanganate and  $\frac{1}{2}$  oz. of sodium carbonate. The pressure in the third stage reaches about 60 atmospheres, and from this the gas passes to the condensing coils, cooled by water and is liquefied. The intermediate collecting vessel and the receivers are suspended on Salter's balances so that the attendant can at once see when they are full. Obtained in this way the gas has a purity of 99.5 per cent., and is odourless. The receivers or cylinders are made of mild steel and are either solid drawn or lap welded. They are tested to 3,000 lb. on the square inch, the normal filling pressure at ordinary temperatures being about 750—800 lb. In some tests made a year or two ago by the Scotch and Irish Oxygen Co. of Glasgow, vessels of this type filled with oxygen at 1,800 lb. to the square inch were allowed to fall 35 ft. on to iron blocks and otherwise severely treated without damage further than bending and slight loss of shape.

The cost of the liquid gas (inclusive of labour and interest on capital) is about 3*l.* 10*s.* and its selling price at present about 18*l.* per ton.

The liquefied carbonic acid is now being used for raising beer, in place of the ordinary pumping-engine. A reducing valve is attached to the cylinder, reducing the pressure to about 4 lb. per square inch, or whatever pressure is required to raise the beer from the cellar to the tap. An air-tight connection is made to the top of the barrel and the pressure not only sends the beer up in good condition but is said to improve it as the level gets lower in the barrel. The antiseptic qualities of the gas and its freedom from germs also make its use preferable to that of the ordinary beer-engine. Liquid carbonic acid has been found advantageous in bottling beer, beer thus bottled maturing in three days in place of three weeks. It is also used for a variety of purposes from aerating water and bread to hardening armour plates.

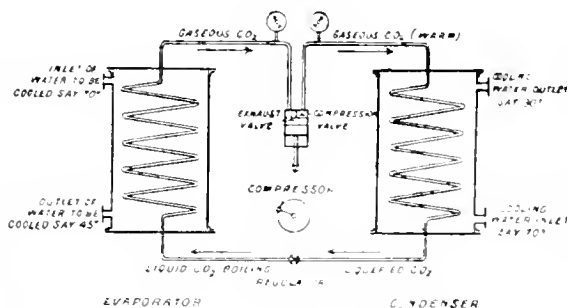


DIAGRAM OF CYCLE OF REFRIGERATING MACHINE.

The author considers, however, that one of its most important uses will be in refrigerating machines. The accompanying diagram will explain the character of the apparatus and cycle of operations. The principle of the machine is

of course, the same as that of the ammonia or ether refrigerating machines, and the degree of cooling can be regulated by the pressure under which the liquefied gas is evaporated, and it is equally easy to cool water, say from 70—30 F., or to maintain brine at say +10° F. or -10 F. Under 35 atmospheres pressure, for instance, liquid carbonic acid boils at 30° F. The surfaces of the evaporator coils are so proportioned that all the liquid which enters the lower end of the coil is evaporated by the time it reaches the top end, and thus the maximum efficiency is obtained. It has been said that as carbonic acid will not liquefy at temperatures above 87° F. (its critical point) its possible duty as a refrigerant in the tropics would be very low. Experience with machines of considerable power using condensing water at 90—95° has, however, shown the loss of efficiency to be very slight, and 40 or 50 carbonic acid refrigerating machines are working satisfactorily in the tropics. The carbonic acid refrigerating machine has the advantage over the ammonia or ether machines that a leak is not so unpleasant or dangerous.

In the discussion T. W. Thorpe stated that in his brewery about 1 ton 2 cwt. of liquid carbonic acid was obtained from 600 barrels of wort, and that the actual cost of collecting and compressing was  $\frac{1}{2}$ d. per lb., a second  $\frac{1}{2}$ d. per lb. being paid as royalty. In their case glass coverings over the tuns had been introduced without any deterioration of vigour of fermentation, and with increased freedom from germs. The Aerated Bread Company used their liquefied carbonic acid for raising their bread. Sir A. S. Haslam was not prepared to admit that there was no particular falling off in efficiency in the carbonic acid gas machine at temperatures at or above its critical point. The critical point of carbonic acid is 31° C. (88° F.), and of ammonia 117° C. (or 232° F.). At 68° F. the latent heat of vaporisation of ammonia is 540 and of carbonic acid 63°, whilst at 86° that of ammonia is 524, and of carbonic acid only 19°. He considered therefore that, at least at high temperatures, the ammonia machines must be more economical than the carbonic acid machines. The ammonia machine too, only required a pressure of 90 to 150 lb. (according to the temperature of the condensing water) while the carbonic acid machines would require from 900 to 1200 lb. pressure. He believed 1 ton of coal with an ammonia machine would do as much work as  $1\frac{1}{2}$  tons with a carbonic acid machine. Dr. Sykes believed the relative economy of working of the ammonia and carbon dioxide machines to be about equal.

In replying to the discussion the author showed a curve the pressure produced in the cylinders by increased temperature. At 113° F. the pressure was only about half that at which the cylinder was tested. In the tropics, where in the sun a temperature of 160° F. might possibly be reached, the pressure was of course considerably increased, but experience showed that even then there was practically no danger. Referring to the loss of efficiency above the critical point of the liquefied gas he said that a large machine to make four tons of ice per day had been run for a week with the cooling water at 100° F. There was of course a loss of efficiency (as was the case whatever material was used), but it was far less than they had been led to expect. These machines were working satisfactorily in the tropics, in some places side by side with ammonia machines, so that any excessive loss of efficiency would have been at once detected.—L. T. T.

*Hansen's System of Pure Yeast Culture in English Top Fermentation.* A. Jørgensen. Trans. Inst. Brewing, 7, 227—254.

In this paper the author upholds Hansen's system of employing "pure yeast" from one cell against Van Laar's "composite yeast" or yeast composed of two selected types. The one type of pure yeast has now been proved to be successful both in extended experimental brewings, and also in practice in many breweries on the Continent and in England. Secondary fermentation takes place regularly and normally, and does not need the presence of a second type of yeast. Experiments made in

Jørgensen's laboratory and also by A. K. Miller at Ardwick Brewery, Manchester, with absolutely pure cultures proved that one single species can carry through both the primary and secondary fermentation and give excellent beer. W. R. Wilson's practical experience at Coombe's Brewery fully confirmed these results. On the other hand, careful experiments were conducted by the author in conjunction with Holm and F. Petersen, in his own laboratory and in breweries, with Van Laar's composite yeast (obtained from Van Laar's Burton Company) and with mixtures of culture-yeast types which had already been tried singly. These experiments showed that in worts pitched with these composite yeasts the ratio of the two types altered as the fermentation proceeded, one type gradually increasing with the gradual suppression of the other and weaker type. Sometimes this suppression was almost complete in one fermentation, in others it was but partial, but continued to total extinction when the yeast from one fermentation was used to start succeeding ones. The author concludes that "Van Laar's composite yeast is not able to preserve the constancy of ratio between the species of which it is composed, but has to be renewed continually if wanted to be kept unaltered." His experiments show that in some cases if type *a* and type *b* of yeast each give satisfactory results in a brewery, a mixture of those types, in the same brewery and under, as far as can be seen, identical conditions, gives unsatisfactory results. Thus the "one species pure yeast" system both gives thoroughly satisfactory results, and is much more certain, regular, and reliable than the "composite yeast" system advocated by Van Laar.

The author also upholds Hansen's claim to be the first to cultivate and investigate really pure yeast cultures containing only a single species.

In the discussion on the paper, Tanqueray said his own experience showed that where a large quantity of pure one-species yeast was used in pitching with the ordinary yeast of the brewery it would crowd out the old yeast, and if, after a short time, a second charge of the same pure yeast were introduced, it would entirely replace the old yeast. This both confirmed and extended Jørgensen's results. G. H. Morris disclaimed any desire on the part of English brewers to depreciate Hansen's work. He would like to see more attention paid to the chemical composition of the wort at different stages and under different conditions, and especially to their carbohydrate composition. His own experience was that each variety of pure yeast had one definite limit-attenuation, below which no further fermentation took place with that yeast in a reasonable time. He would like to see more definite figures brought forward to prove that the single-cell pure yeast gave a true secondary or after fermentation. Dr. Sykes said since the meeting of the Institute in January last, his own actual observation had compelled him to reverse the views he then expressed and admit that single-cell pure yeast could be successfully used and did produce beer satisfactory as regards both flavour and after fermentation. Fellowes did not consider the evidence regarding secondary fermentation was satisfactory. Whilst Wilson was obtaining good results with single-cell yeast, others were equally satisfied with the results from composite yeast. In replying, the author pointed out that the results described by Tanqueray of the crowding out of the old yeast by pitching with pure single-cell yeast may prove useful in all cases where a period of transition from the old to the new yeast is desired.—L. T. T.

*Affinity-Constants of Weak Acids.* R. W. Wood. Amer. Chem. J. 16, 313—325. (Compare this Journal, 1894, 515.)

THE author has continued his experiments on the action of various substances in retarding the fermentation of starch by diastase. By employing a new constant-temperature bath, several possible sources of error are eliminated. He has investigated the effects of changes of temperature on diastatic action, and finds that 55° C. is the most favourable temperature. The retarding influence of acids is due to the presence of hydrogen ions and from his measurements

on weak acids he has been able to calculate their affinity-constants, which agree fairly well with those obtained by Ostwald from the measurement of electrical conductivity.

The neutral sodium salts of the weak acids have also a strong inhibitory action on diastase, probably due to the presence of hydroxyl ions formed by the hydrolytic dissociation of the salt. The author's method does not apply to the salts of exceedingly weak acids, and consequently no satisfactory comparison can be made with the numbers obtained by Shields. The action of salts on the corresponding acids obeys the law of mass action. In conclusion the author points out that the method which he has adopted may be applied to the determination of the relative affinities of the organic bases.—J. S.

*Composition and Analysis of Yeast.* P. Guichard. Bull. Soc. Chim. 1894, 11, 230.

See under XXIII., page 844.

### PATENTS.

*Improvements in the Process of and Apparatus for Drying Distillers' Washes and the like, and for Removing the Acids therefrom.* Actien Maschinenbauanstalt, vorm. Vemulth and Ellenberger, Darmstadt, Germany. Eng. Pat. 11,153, June 7, 1893.

THE production of feeding-cake from distillers' wash, &c., usually involves the evaporation of large quantities of water. The object of this invention is to do away with that expensive operation as far as possible, by making use of a process of precipitation and filtration. For this purpose the wash is raised to boiling, treated with alum (1 lb. to 5,000 galls.) to precipitate the dissolved albuminoids, and filtered through an improved filter-press. The latter consists, as usual, of a row of cells and intermediate pairs of filter-plates separated by ribbed frames. The wash is supplied by a common main-pipe simultaneously and independently to all the cells; and, moreover, each filtering partition is fitted with a separate discharge-cock. In consequence of this arrangement, filtration is rapid and uniform at all points of the press; and the cells may be made much wider, and therefore the press-cakes thicker, than usual. When the cells are filled with insoluble matter a further portion of water is expressed from the same, by admitting air at high pressure to every alternate filtering partition, and the press-cakes from the frames are finally completely dried in any convenient manner. The water thus mechanically removed from the wash, carries with it the bulk of the organic and inorganic acids, and other valueless substances originally present. The cake being deprived of these bodies, becomes correspondingly richer in albuminoids, &c., and compares very favourably with cakes produced in the usual way.—H. T. P.

*Improvements in the Manufacture of Yeast and the Distillation of Spirits.* L. Lederer, Dalston. Eng. Pat. 12,837, June 30, 1893.

THE special points of the process are: (1) The use of green malt for saccharifying purposes; (2) the employment of a previously-prepared "fermenting medium"; (3) the carrying out of the whole of the operations, fermentation and distillation included, without removal of the grains. The fermenting medium is prepared by making a strong mash of equal parts of green malt and rye at  $62\frac{1}{2}$ — $65^{\circ}$  C., allowing the mixture to acidify for 20—24 hours at  $48^{\circ}$  C., and finally pitching it at  $20^{\circ}$ — $24^{\circ}$  C. with fresh distillers' yeast. In about 10—12 hours the temperature rises to  $34^{\circ}$ — $35^{\circ}$  C., and the yeast is ready for use. The main mash is made in the usual way in an apparatus consisting essentially of an ordinary mash-tub, into which a framework carrying a rotatory stirring arrangement, and heating and cooling coils, can be lowered. Raw grain (maize, &c.), if used, is soaked and boiled prior to its admixture with the cold mash of green malt, &c., and saccharification is allowed to proceed at  $65^{\circ}$  C. for 15—30 minutes. The mash is then cooled to  $35^{\circ}$  C., mixed with the above fermenting medium, further

cooled to  $28$ — $30^{\circ}$  C., and run into fermenting vats. In the latter, the liquid is diluted until it measures 75—85 gallons per 112 lb. of grain used, and acidified with sulphuric acid (2—2½ per cent.).

The yeast which forms is skimmed off, washed twice by decantation with water, and is then ready to be pressed. The fermented mash is distilled in the usual way for the production of spirit. It is claimed that this process is more economical and rapid (the whole operation, distillation included, requires 60 hours) than the older ones; and that a much higher yield of whiskey, rectified spirit, &c., of excellent quality may be obtained by a single distillation, without having recourse to rectification.—H. T. P.

*Improvements in the Clarification and Preservation of Syrups, Extracts, Wines, Beers, Mineral Waters, and other Liquids.* R. E. Evans, Stratford-on-Avon. Eng. Pat. 11,373, July 26, 1893.

THE inventor's claim is for an improved clarification effected by using aluminium hydrate, either alone or in conjunction with gelatin, isinglass, algin, albumin, or other materials.

The liquid to be treated is mixed with a suitable quantity freshly prepared aluminium hydrate; or alum is first added and then a judicious quantity of carbonate of potash to precipitate the alumina. The clarification may be materially assisted by the addition of gelatin, isinglass, &c.—about one part for every part of alumina.—L. de K.

*Improvements in Apparatus for the Preparation of Malt.* R. R. P. Schmiedeecke and E. O. Gade, Berlin, Germany. Eng. Pat. 1717, January 26, 1894.

AN improved system of pneumatic malting. The germinating grain is contained in a horizontal cylinder, free to rotate on a fixed tubular axis, through which the necessary air is supplied. The central tube is provided with air-outlet holes only on one half of its circumference, and is surrounded by a second tube, from which radiate, like the spokes of a wheel, a series of perforated air-conveying pipes. The outer tube, &c., rotates with the cylinder and serves as a species of slide-valve, air being admitted to the pipes only during their passage over the apertures in the fixed axis. Normally, the said apertures face downwards, resulting in air being supplied to the pipes as they revolve below the axis (and consequently are submerged in the malt); but the latter may be set at any angle, according to the degree of aeration desired. A throttle-valve, mounted inside the axis, and capable of being slid to any desired point, serves to divert the bulk of the air-current to any particular section of the cylinder which may happen to become overheated. H. T. P.

*Improvements in Carbonic Acid Pressure Apparatus for Vessels from which Liquids are Retailed.* N. Browne, London. From P. Beck, Augsburg, Bavaria, Germany. Eng. Pat. 1926, January 29, 1894.

See under I., page 788.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY AND WATER PURIFICATION, DISINFECTANTS.

### (A).—CHEMISTRY OF FOODS.

*Margarin compared with Natural Butter as regards Digestibility and Nutritive Value.* A. Jolles. Monatsh. Chem. 15, 147—163.

VARIOUS statements have been made as to the relative values of margarin and genuine butter as foodstuffs, the



general outcome of which is, that whilst there is not much to choose between the two as regards digestibility and nutritive value, butter has a slight advantage over margarin in these respects. The author has carried out a long series of observations with a dog fed during four consecutive periods with butter and margarin alternately; the urine and faeces being collected and examined for fatty matter, nitrogenous constituents, &c., so as to obtain the data for determining how much fatty matter passed unassimilated through the animal under each set of conditions as to feeding. In the first and second periods more fat and less carbohydrates were given; in the third and fourth, less fatty matter and more carbohydrates; the fatty matters being butter in periods 1 and 3, and pure margarin in periods 2 and 4. The various articles of food (wheatmeal, sugar, &c.) were carefully analysed and made up into dog-biscuits, so that the amounts of the different kinds of food constituents consumed during each period were accurately known. In this way it was possible to trace out during each period the proportion of proteids, fat, non-nitrogenous matters (starch, &c.), and mineral constituents (ash), which were either digested and assimilated, or passed out undigested in the faeces. So far as fatty matters were concerned, 97 to 98 per cent. was uniformly digested, whether butter or margarin; the figures obtained during the four periods respectively were as follows (in grms.):—

	Period.			
	I.	II.	III.	IV.
Contained in food.....	52.12	42.14	36.84	37.36
"    faeces.....	0.79	0.86	1.05	1.22
Digested.....	51.33	41.28	35.79	36.14
Percentage digested.....	98.1	97.9	97.1	97.3

Hence the conclusion is drawn that under similar conditions of feeding, butter and margarin have practically identical coefficients of digestibility and nutritive value.

—C. R. A. W.

#### *The Proteids of the Kidney-Bean (Phaseolus vulgaris).*

T. B. Osborne. Seventeenth Annual Report of the Connecticut Agricultural Experiment Station, 1893, 186—210.

"THE kidney-bean contains two globulins characterised by great solubility in very dilute saline solutions, and by yielding precipitates with acids, soluble in sodium chloride solutions. One of these globulins, *phaseolin*, probably forms about 20 per cent. of the seed, and has the following composition:—C 52.58 per cent., H 6.84 per cent., N 16.47 per cent., S 0.56 per cent., O 23.55 per cent. The other proteid, *phaseelin*, is much more soluble, remaining in solution after the phaseolin has separated. It is slowly coagulated by heat at temperatures varying with the amount of salts present and the rapidity of heating. It is precipitated by acids, on prolonged dialysis yields insoluble or albuminate modifications, and has more nearly the properties of a globulin than of any other recognised class of proteids. Its elementary composition is: C 51.6 per cent., H 7.02 per cent., N 14.65 per cent., S 0.49 per cent., O 26.24 per cent. In addition to these globulins, the extracts were found to contain an extremely small amount of *protease*."—H. T. P.

#### *The Proteids of Cotton-Seed.* T. B. Osborne and C. G. Voorhees. Seventeenth Annual Report of the Connecticut Agricultural Experiment Station, 1893, 211—217.

CONSIDERABLE practical difficulties were encountered in this investigation, and the results are by no means complete. So far the authors have found that the proteids of cotton-seed comprise:—1. A very small proportion of a substance resembling *protease*. 2. About 16 per cent. of a globulin (styled *edestin*) similar in composition and probably identical with the *vitellin* existing in the seeds of wheat, maize, hemp, flax, &c. *Edestin* is soluble in saline solutions

and has the following composition:—C 51.71 per cent., H 6.86 per cent., N 18.64 per cent., S 0.62 per cent., O 22.17 per cent. 3. A proteid soluble in weak potash, but not in salt solutions. 4. A nitrogenous substance insoluble in either of the above solvents. The total nitrogen of cotton-seed is distributed among these various proteids in the following proportions:—*Protease*, 2 per cent.; *edestin*, 42.3 per cent.; alkali-soluble proteid, 44.3 per cent.; insoluble proteid, 11.4 per cent.—H. T. P.

#### *Normal Chlorine in Spring Waters.* R. Haines.

J. Franklin Inst. 137, 134—139.

IT is probable that the amount of "normal chlorine" naturally existing in spring waters, as distinguished from that due to contaminating influences arising from the life and occupations of man, will be found to be fairly constant in the waters of any particular locality, as has been found in Massachusetts, where the matter has been under investigation for several years.

It has been shown that the amount of normal chlorine decreases regularly from the sea-coast inland. Waters, free from all contamination, near the coast contain about 0.65 part of chlorine to 100,000, and in the western part of the State about 0.10 part to 100,000.

Lines, called "isochlors," have been plotted, passing through places of equal amount of chlorine in normal waters, and as these preserve a general parallelism to the coast-line, it is reasonable to attribute the decrease of normal chlorine to the diminishing effect of the sea-air. From recent investigations it is probable that a similar set of lines can be found for New Jersey and Eastern Pennsylvania. In the present paper the vicinity of Philadelphia, east of the Delaware River, is alone under consideration. Ten suitable sources of water were found, from which 21 samples were taken and analysed. It is probable from these that the normal chlorine of this district approximates closely to 0.20 part to 100,000. The chlorine was determined by the usual volumetric method with silver nitrate and potassium chromate, but with the improvements and corrections described by Hazen (Am. Chem. J. 11, 409), which are re-described.—R. B. P.

#### PATENTS.

##### *Improvements in or relating to the Manufacture or Preservation of Condensed Milk.* C. Ekin and W. H. Thew, both of London. Eng. Pat. 14,894, August 3, 1893.

ACCORDING to this invention, levulose or maltose, or a mixture of both, is used instead of cane-sugar in the manufacture of condensed milk or condensed peptonised milk.

The inventors claim the preparation of a product which does not cause dyspepsia. In other respects the manufacture of the peptonised article is carried out as set forth in the specification of W. H. Thew, Eng. Pat. 1313 of 1884.

—L. de K.

##### *Improvement in the Manufacture of Condensed Milk Preparations.* H. B. Briggs, London. Eng. Pat. 15,434, August 14, 1893.

ACCORDING to this invention, levulose is substituted for cane-sugar in the manufacture of condensed milk, and used to the extent of about 25 per cent.

The inventor claims that thus a more soluble and easily digestible product is obtained.—L. de K.

##### *Improvement in the Manufacture of Condensed Milk Preparations, Invalids' and Children's Foods.* H. B. Briggs, London. Eng. Pat. 15,435, August 14, 1893.

ACCORDING to this invention, invert sugar is substituted for cane-sugar in the manufacture of condensed milk and farinaceous food preparations.

The inventor claims the preparation in this wise of articles of easier digestibility.—L. de K.





TABLE I.—*cont.*

Description of Effluent.	Organic Matter in Grms. in 1,000 cc.			Organic Matter removed in Grms. in 1,000 cc.			In Grms. of CaO in 1,000 cc.
	In Suspension.	In Solution.	Total.	In Suspension.	In Solution.	Total.	
Towns' effluent :							
12. { Unpurified .....	1,165	160	1,325	1,165	30	1,195	280
{ Purified .....	0	130	130				
13. { Unpurified .....	220	215	435	220	80	260	..
{ Purified .....	0	175	175				
Mixture of manufacturing and household effluents :							
14. { Unpurified .....	3,020	575	3,595	3,020	190	3,210	308
{ Purified .....	0	385	385				
15. { Unpurified .....	550	1,050	1,600	550	305	855	..
{ Purified .....	0	745	745				
16. { Unpurified .....	270	985	1,255	270	160	430	358
{ Purified .....	0	825	825				

TABLE II.

Description of Effluent.	a.		b.		c.		Difference between b and c.	
	Loss on Ignition.	Degrees absorbed.	Loss on Ignition.	Degrees absorbed.	Loss on Ignition.	Degrees absorbed.	Loss on Ignition.	Degrees absorbed.
1. Starch-works' effluent .....	950	289	835	284	785	267	- 50	- 17
2. " " .....	710	243	540	217	495	211	- 45	- 6
3. " " .....	520	185	425	180	415	..	- 10	..
4. " " .....	555	249	525	224	515	224	+ 20	+ 0
5. Cardboard-works' effluent .....	615	269	510	214	..	217	..	+ 3
6. " " .....	240	58	112	36	87	35	- 25	- 1
7. " " .....	435	179	280	137	275	118	- 5	- 19
8. " " .....	310	118	235	96	240	97	+ 5	+ 1
9. Mixture of effluents .....	505	270	535	262	525	249	- 10	- 13
10. " " .....	340	269	295	169	233	117	- 62	- 52
12. Towns' effluent .....	160	..	130	..	115	..	- 15	..
13. " " .....	215	70	175	45	170	38	- 5	- 7
14. Mixture of effluents .....	575	221	385	118	430	119	+ 45	+ 1
15. " " .....	1,050	378	715	172	685	157	- 60	- 15
16. " " .....	985	435	825	308	747	314	- 78	+ 6

These results show that in the direct purification an effluent is obtained containing almost invariably a greater amount of organic matter in solution, as is the case when the purification has been preceded by filtration. This shows that lime to some extent does dissolve suspended organic matter, but the statement that by the lime treatment the total quantity of organic matter in solution is increased, is without foundation.—C. O. W.

*The Removal of Pathogenic Bacteria from Drinking-Water by Sand-Filtration.* G. W. Fuller. Tech. Quart. 1893, 6 [4], 273—285.

THAT sand-filters properly worked are effective in removing bacteria from drinking-water is shown by the investigations

of P. Frankland, who found an average removal of 97.6 per cent. of the bacteria in unfiltered Thames water, after filtration. At the experiment station at Lawrence, Massachusetts, intermittent filtration through sand was found to effect the removal of 97.3 per cent. of the bacteria in the original water taken from the city supply. When river-water containing more than ten times as many bacteria as did the city supply, was filtered experimentally in the same manner, a filtrate containing as low a number of bacteria as in the former experiment was obtained. Three of the species of bacteria most prominent in the river-water were not present in the filtered water, *B. coli communis* being one of these. That the water was not always completely sterile appears to be due to the fact that bacteria found access to it after its filtration, through

the outlet-pipes and under-drains. Intermittent filtration, according to the method thus experimentally employed, would be too costly for practical purposes, as the amount of water filtered per acre did not exceed 250,000 gallons daily.

An elaborate series of experiments was carried out to ascertain whether ordinary sand-filters carefully worked were competent to remove bacteria applied in large numbers as pure cultivations. An example of the method of experiment ultimately adopted, may be quoted. A pure cultivation of a bacterium was prepared of such strength that 1 cc. contained on an average 20,000,000 bacteria. This solution was applied to the filters in the proportion of one part to 3,000 parts of water at intervals of one or two hours, according to the rate of filtration. Every cubic centimetre of water which entered the filter contained on an average 6,000 bacteria in addition to the water-bacteria normally present. The amount of organic matter in the water was increased as little as possible, so that direct comparison could be made with the water in its un inoculated condition. Using *B. prodigiosus* for the purposes of this investigation, the following results were obtained:—

Depth of Filter in Feet.	Rate of Filtration (Galls. per Acre daily).	Average Number of Bacteria applied.	Per Cent. removed.
5	1,300,000	3,000	99.81
5	2,400,000	6,220	99.96
2	1,800,000	1,700	99.86
1	1,000,000	6,230	99.83

With regard to the efficiency of the surface layer of a filter (considered in Berlin to be the most important portion) it is found that the number of bacteria in the filtered water is but slightly influenced by the removal of the surface layer during the scrapings of the filter which are periodically necessary to prevent choking. There is certainly a slight increase in the number of bacteria in the filtered water after scraping, but it only amounts to a small fraction of 1 per cent.

Practice differs concerning the selection of sand for the construction of filters. In Germany it is usual to wash out the finer particles, but this is not done at the Lawrence experiment station now under consideration. Samuelson, the chief engineer of the Hamburg Waterworks, states that the best filtering sand is one consisting of particles uniformly approximating to a diameter of 1 mm. Experience at Lawrence shows however, that finer particles than this are useful in filling up interstices, and the following canon has been arrived at:—"The effective size of a sand is considered as the maximum diameter of the finest 10 per cent. of the grains." The size of the sand grains from the best filters at Lawrence lies between 0.2 and 0.3 mm. That it is the upper part of the filter which does the greater part of the work of purification, is shown by the following table, containing the figures given by a filter that had been in operation for about 6 months.

Depth from Surface (in inches).	Bacteria per Grain of Sand.
0-1	1,100,000
1	320,000
1	110,000
2	21,000
4	4,000
6	1,600

As a practical outcome of the experiments carried out at Lawrence, a filter  $2\frac{1}{2}$  acres in area, and having a capacity of 5 million gallons per day has been constructed, which is remarkable from the fact that it is worked intermittently,

the oxidation thus induced aiding the removal of the organic matter, which accumulates at the surface of the sand. It has also been demonstrated that it is practicable to construct filters which will economically remove 99 per cent. of the bacteria in the unfiltered water.—B. R.

## PATENTS.

*Improvements in the Treatment of Sewage or Analogous Foul Waters or Matters for Effecting the Disinfection and Purification thereof.* A. P. Hope, Leicester. Eng. Pat. 10,458, May 27, 1893.

Air or oxygen, together with anti-septics or deodorisers such as creosote, cresolite, carbolic emulsion, is injected into the sewage by means of steam. After this treatment, the sewage may be treated by any precipitation process.

—J. C. C.

*Improvements relating to the Purification of Water, and Apparatus therefor.* L. and C. Maiche, Bois Colombes, France. Eng. Pat. 12,941, July 1, 1893.

THE inventors lay claim to a process for sterilising water which consists in destroying the germs and microbes by the employment of lime, then rendering the water drinkable by precipitating the lime with carbonic acid. An apparatus for mixing the lime water and water to be purified, is also described and claimed.—J. C. C.

*Improvements in the Purification of Sewage and other like Waste Liquids, and in Apparatus employed therefor.* W. E. Adeney, Monkstown, co. Dublin, and W. R. Parry, Kingstown, co. Dublin. Eng. Pat. 13,154, July 5, 1893.

THE patentees here extend and improve upon their previous inventions, see patents 3312 and 18,983 of 1890, and 10,929 of 1891 (this Journal 1891, 1021 and 1892, 630). They now treat the effluent of settled sewage, successively, with sodium manganate (2—5 grains to the 1 gallon), aluminium sulphate (about 7 grains to the gallon), and sodium nitrate (2 to 3 grains to the gallon), the effluent being then subjected to the action of bacteria and other micro-organisms.

The plant employed is quadruplicate, each unit comprising (1) a deep tank, narrowing at the bottom to (2) an axially located sump hole, to the bottom of which reaches (3) a pipe connected with (4) a chamber in which a partial vacuum can be produced. The foregoing arrangement permits of the collection, in the respective four chambers, of the solid sewage, of the three precipitates produced by the three reagents employed, and of the deposit produced by the bacterial action. Near the top of each tank is (5) an overflow, leading to (6) a wide pipe which in its turn extends nearly to the bottom of the next tank; this allows of the flow of liquid from the first to the second tank, from the second to the third, and so on, the reagents enter the stream in the conduit between two tanks. An equal distribution of the upward flow of liquor in the tanks is secured by means of spreading arms or plates placed horizontally near the end of the affluent pipe.

The products obtained are (from tank No. 1) 1. solid sewage settlings, which are drained and dried, being sometimes mixed with a little manganese oxyhydrate to prevent putrefaction during the process; 2 (from tank No. 2), manganese oxyhydrate which is dried and used in sodium manganate manufacture; 3 (from tank No. 3), aluminium hydrate and basic sulphate, which is recovered as sulphate by treatment with sulphuric acid. This precipitate gradually gets richer in manganese (carried over from tank 2) and is then fused with sodium hydrate and nitrate to obtain sodium manganate and aluminate.

The authors have further found that the sulphates of iron and manganese, as obtained by treating manganiferous iron ore with sulphuric acid, may be substituted for sulphate of alumina in tank 3 and the precipitated basic hydrates and carbonates recovered by subsequent treatment with sulphuric acid.

When sewage contains much or very carbonaceous organic matter, it is well to add the aluminium sulphate (or its iron and manganese equivalent) before the sodium manganate, increasing its amount to 10–20 grains per gallon. Waters containing much acid or alkaline trade effluent require to be preliminarily neutralised by chalk or by mineral acid, according to circumstances.

A further claim in this patent relates to an automatic machine for supplying regularly definite amounts of reagent, whether liquid or solid.—E. R. B.

*Improvements in the Treatment of Sewage Sludge.* R. C. Tanner, Chester. Eng. Pat. 13,305, July 8, 1893.

THESE improvements consist in breaking up pressed sludge cakes in a pug mill, to the under side of which is attached a perforated cone, through which, by the rotation of a conical screw, the material is forced to fall down in a vermicular condition on to a travelling belt which carries it into a hot chamber, where it is dried and is fit for immediate use as a manure, or for carbonisation to obtain ammoniacal liquor and other by-products.

The machinery which the inventor proposes to employ is described in Eng. Pat. 2218, 1859.—J. C. C.

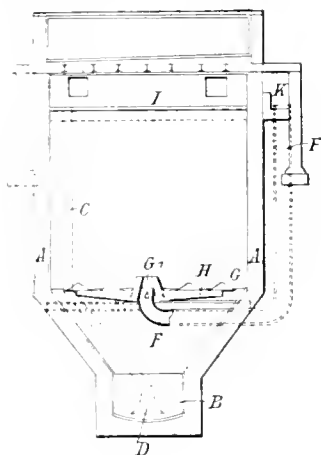
*Improvements in the Treatment of Sewage and other Foul Waters.* R. C. Tanner, Chester. Eng. Pat. 14,642, July 31, 1893.

A SOLUTION of persulphates of iron or a mixture of proto- and persulphates of iron is evaporated to dryness and mixed with bleaching powder. The sewage is treated with this mixture after having been made alkaline with lime.

—J. C. C.

*Improvements in the Construction of Tanks or Receptacles for the Precipitation of Sewage, and for like Purposes.* E. C. Ives, Derby. Eng. Pat. 13,411, July 11, 1893.

IN the figure, A is the tank, terminating at its lower extremity in an inverted cone, with or without the sump B. C is the sludge suction-pipe. The sewage enters the tank through the pipe F, directly over the sump B (finally flowing



TANK FOR THE PRECIPITATION OF SEWAGE, &c.

upwards, by means of the cover G, which is connected to distributing arms H, designed to distribute the sewage over the whole surface of the tank when running into the same, and to ensure the rising of this sewage uniformly to the overflow arms at I. At or about the top are overflow arms at I, spread out to assist the uniform upward action of the sewage, the overflow arms being connected with an outlet K to carry the effluent away.—J. C. C.

*Improvements in and in the Production of Compositions adapted to be used in the Treatment of Sewage and other Foul Waters.* C. A. Burghardt, Manchester. Eng. Pat. 14,801, August 2, 1893.

THE patentee produces a new composition or cake containing ferric sulphate, ferric chloride, aluminium sulphate, and, in some cases, sodium sulphate, for the treatment of sewage and other foul waters. The new product principally differs from that claimed in his patent, Eng. Pat. 13,316 of 1892 (this Journal, 1893, 778), by the presence in it of aluminium sulphate and by the fact that no storing process is now necessary.

Two methods of preparation are described: either finely-powdered bauxite, or other aluminiferous material, ferric oxide, and salt are mixed with sulphuric acid, or to an aqueous solution of aluminium sulphate concentrated solution of ferric chloride, mixed with sodium sulphate, is added. In either case evaporation yields the mass or cake claimed.

Whether the aluminium or the iron is allowed to predominate in the cake is determined by the special purpose to which it is to be put: for "general domestic sewage" the cake should contain about 11 per cent. of iron as ferric sulphate or chloride, and 2.5 per cent. of aluminium as sulphate.—E. R. B.

*Improvements in and Relating to the Softening and Purifying of Water and Apparatus therefor.* H. L. Doulton and A. W. Manger, both of Lambeth Potteries, Surrey. Eng. Pat. 15,169, August 8, 1893.

THE apparatus is intended to soften and purify water by firstly, removing the temporary hardness; secondly, removing the permanent hardness; and thirdly, for removing the alkalinity when required. To remove the temporary hardness the water is treated with lime in a mixing trough and afterwards passed through settling and filtering tanks. To eliminate the permanent hardness the water is then treated with soda or other suitable reagents, in a similar set of tanks, and finally, to remove alkalinity a perforated tank containing suitable reagents, such as boracic acid, aluminium phosphate, or the like, is arranged between the baffles of the settling tanks in such a manner that the water is subjected to the action of such anti-alkaline reagents as are adopted.—E. G. C.

*Improvements in the Treatment of Sewage and Waste Waters.* G. Jarman, Huddersfield. Eng. Pat. 16,522, September 2, 1893.

THE inventor treats charged sewage sludge, the result of precipitation, with crude hydrochloric acid to dissolve out the iron or other mineral matter contained in the sludge. This solution, after being partially neutralised, can be used again as a precipitant for other sewage. The animal charcoal after the extraction is used as a filtering medium.

—J. C. C.

*Improvements in or Relating to the Disinfection of House and other Refuse, and Apparatus therefor.* A. T. Boulton, Middlesex. From F. S. Salberg, Brussels, and M. Neumaun, Vienna. Eng. Pat. 4169, February 27, 1894.

IN this patent is described a general process, automatic in its action, for the disinfecting and rendering harmless or refuse and offal of all kinds, at the time of their being deposited in all the various kinds of receptacles allotted to their storage. The several modifications of these receptacles (dust bins, scavengers' carts, water-closets, with and without water supply, urinals, lavatories, &c.) rendered necessary by the process are also described, figured, and claimed at considerable length.

The "fundamental idea" of the process "consists in the utilisation of the force developed, for instance, by the discharge into a scavenger's cart of refuse from a bin, for working an apparatus intercepting the dust on the one hand, and on the other distributing a disinfectant."

In a modification which is generally applicable, refuse falling into a receptacle loads the vanes of a shaft disposed across the cavity of the receptacle, the shaft then rotates, and by a simple mechanism allows a certain quantity of a disinfectant to fall into the receptacle. Either solid or liquid disinfectants may be employed.—E. R. B.

### (C.)—DISINFECTANTS.

#### PATENTS.

*Improvements in Fumigants.* I. S. McDougall and J. T. McDougall, London. Eng. Pat. 14,411, July 26, 1893.

To facilitate the combustion of sulphur and of carbon, when either of these substances is burned as a fumigant, the inventors propose to add suitable nitrates, such as those of potassium, sodium, ammonium, or barium, in proportions, however, not so high as to cause explosions. In practice, they find "sulphur, 75 parts; nitrate of potassium or other nitrate or mixtures of them, 25 parts"; and "carbon, 70 parts; nitrates, 30 parts"; to be useful proportions. The percentage of nitrate must not exceed 60 per cent. according to the inventors.—E. R. B.

*Improvements in the Production of Insecticides.* I. S. McDougall and J. T. McDougall, London. Eng. Pat. 14,412, July 26, 1893.

In the preparations, by means of boiling water, of tobacco extract, to be used as an insecticide, hitherto much of the nicotine has been lost by volatilisation; the patentees therefore propose to extract with very dilute acid, e.g., with 16 oz. of concentrated sulphuric acid to 100 lb. of tobacco. The acid employed may be sulphuric, hydrochloric, nitric, acetic, phosphoric, or a suitable mixture of them.—E. R. B.

*A New Method of Treating Cotton Wool, Lint, Gauze, Wood Fibre, Lint, Bandages, Flannel, Sponges, and similar Substances used for Medical and Surgical Purposes, in order to render them Antiseptic.* M. C. Clutterbuck, Barton-on-Trent. Eng. Pat. 14,812, August 2, 1893.

The substance to be treated is placed in a chamber and alternately subjected to the action of sulphur dioxide gas and ammonia, or a mixture may be introduced by volatilising ammonium sulphite. The process is completed by driving out the excess of ammonia, if any, by a current of dry air.

—J. C. C.

*Improvements in Sulphur Candles or Fumigators.* C. T. Kingzett, Chislehurst, Kent. Eng. Pat. 14,903, August 3, 1893.

The object of this invention is to provide a means whereby the block of sulphur, forming a "sulphur candle" or fumigator, may be readily ignited. This "lighter" is made by steeping any open or porous material (such as muslin, cotton wool, paper, straws, &c.) in melted sulphur so that it becomes coated with it. Combustible material is naturally preferable for the supporting material, but thin asbestos cloth for example may be employed, providing the coating layer of sulphur be sufficiently thin.

To use the "lighters" these may be either wrapped round the top of the candle projecting above the end, or the strip may be fused on, or simply lie loosely on the top of the candle.

In any case the thin layer of sulphur on the "lighter" is readily set on fire, and running down on to the top surface of the candle, fires it also.

The lighting material may itself be used as a fumigator, and is then conveniently made in a ribbon like form which can be rolled up as a candle.—E. R. B.

*Improved Method and Means for Preserving Food and other Substances and for supplying Sterilised Air to Rooms and other Enclosed Spaces.* J. Eilfont, Brussels, Belgium. Eng. Pat. 15,757, August 19, 1893.

The principle recognised in the process is that a layer of air passing over a moist surface will leave on this surface all the microbes and bacteria it contains, so that if the place where air is admitted into a suitably closed vessel be provided with an apparatus forcing such air to circulate over hygroscopic surfaces, or zig-zags, before it obtains access to the vessel, the latter will only contain absolutely sterile air, not altering the food or other substances stored or placed in this vessel. It is proposed to mix with the water or liquids used for moistening the hygroscopic or moist surfaces or zig-zags,—hydrofluoric acid or fluorides, or other antiseptics.

—L. de K.

*New or Improved Sanitary Paper, Felt, or Analogous Material.* S. Guillian, Liverpool. Eng. Pat. 15,914, August 23, 1893.

See under XIX., page 834.

*Scab Wash or Medicinal Lotion for the Cure of Scab in Sheep.* A. Robertson, Oban, N.B. Eng. Pat. 3500, February 19, 1894.

This is a paste which when mixed with water forms a medicinal lotion. The following are the constituents of the paste.

Arsenious acid, 320 grains; sulphur, 540 grains; tar distillate (25 per cent.), 1 oz. 5 drams; carbolic acid (95 per cent.), 6 drams; water, 5 drams; caustic soda, 184 grains; wool fat, 240 grains; resin, 220 grains. The fat having been partially saponified by heating with part of the soda and water, the remainder of the soda and water together with the arsenious acid previously dissolved in the solution of soda, is added while all are still hot, and the other ingredients stirred in.—J. C. C.

*Improvements in Insecticides.* A. J. Boulton, London. From F. Haase, Dresden, Saxony. Eng. Pat. 10,188, May 25, 1894.

Two parts of finely ground quassia-wood are mixed intimately with one part of sugar; the mixture is then triturated with a slight addition of some suitable substance, which will tempt insects from some distance, and at the same time conceal the matter contained in the quassia-wood which repels them; such substances being valerician or butyric acids, &c. The mixture is then ground with a solution of gum-tragacanth and made into tablets, pastilles, &c.—J. C. C.

## XIX.—PAPER, PASTEBOARD, Etc.

*Nitrocellulose in Filter-Paper.* E. Cramer. Zeits. angew. Chem. 1894, 269.

The incineration of filter-papers is a tedious operation which may be obviated when employing papers consisting wholly or partially of nitrocellulose. Such papers offer the additional advantage of filtering very rapidly, and of not being quite as hygroscopic as ordinary filter-papers.

—H. A.

## PATENTS.

*Process for Utilising the Lyes of Sulphite Cellulose Products Obtained in such Process, and Apparatus Employed for that Purpose.* A. Mitscherlich, Friburg, Germany. Eng. Pat. 12,927, July 1, 1893.

THE inventor claims a separation of the constituents of the lye of sulphite cellulose by osmose in a special apparatus, and utilises such constituents for a tanning material and a cementitious material. He also obtains alcohol by fermenting a certain portion of the liquor, and further obtains a food for animals.—J. C. C.

*New or Improved Sanitary Paper, Felt, or Analogous Material.* S. Quilliam, Liverpool. Eng. Pat. 15,914, August 23, 1892.

A FELTED paper is produced by any ordinary process out of wool, wool and cotton, or sponge. When made, or nearly made, it is perforated, and then thoroughly indurated with a disinfectant, such as "Izal," carbolic acid, and the like.

—J. C. C.

*Improvements relating to the Treatment of Paper for Receiving Printed Impressions.* P. M. Berger, R. Waldbaur, and E. F. Worlitzer, Leipzig-Schönefeld, Germany. Eng. Pat. 16,358, August 30, 1893.

IN this process barium sulphate, hydrate of alumina, or calcium sulphate, are precipitated in the paper itself, and it is claimed that paper treated in this way is particularly suited for block printing, as it takes the ink equally all over.—J. C. C.

*Method of Bleaching Paper Pulp or other Flocky Substances.* J. W. Abom, Stockholm. Eng. Pat. 8964, May 5, 1891.

THE material to be bleached is placed in a receptacle of wood, stone, or other non-conducting material which is not acted on by chlorine, and which is provided with a stirring device. A chloride solution, preferably of an alkali or an alkaline earth, is poured into the receptacle, and an electric current is made to traverse it, preferably so that one pole will be at the circumference of the vessel and the other near the centre. The poles consist of carbon plates. When the current has been in action for 20 or 40 minutes the contents of the receptacle are discharged into another suitable vessel where the after bleaching is carried on during 10 to 20 hours. During the bleaching operation the temperature must not rise to 60° C., or the hypochlorite formed by the current will be transformed into chlorate which is inert as a bleaching agent.—J. H. C.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

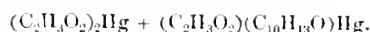
*Sodium Boro-Salicylate.* P. Adam. Bull. Soc. Chim. 1894, 11, 204.

BOSE and Schwartz have demonstrated that addition of borax increases the solubility of salicylic acid. Such compound solutions have been recommended by various authors. However, the question whether these solutions contain a compound of borax and salicylic acid has never been investigated, although the existence of tartroborates renders such an assumption very probable. It was found that by boiling 62 grms. (1 mol.) of boric acid with 160 grms. (1 mol.) of sodium salicylate in 350 cc. of water, a syrupy liquid was obtained. If boric acid is used in excess the excess separates out on cooling. The mixture, in the above proportions, of boric acid and sodium salicylate, reddens litmus and

turns turmeric paper brown, but the syrupy solution obtained on boiling the mixture has no action on these indicators. On evaporating this solution white opaque masses of sodium boro-salicylate are obtained. This substance is very easily soluble in water, and the solutions produce no precipitate with the ordinary reagents for boric acid.—C. O. W.

*The Constitution of the Mercurial Compound of Thymol Acetate.* Arch. Pharm. 231, 123.

THE compound possesses the formula—



The substance contains 55 per cent. of mercury.

—C. O. W.

*Oil (Otto) of Roses.* J. Bertram and E. Gildermeister. J. prakt. Chem. [2], 49, 185—196.

ECKART showed (this Journal, 1892, 265) that the chief constituent of the so-called "otto of roses" is an alcohol,  $C_{10}H_{18}O$ , to which he assigned the name rhodinol. Markownikoff and Reformatzky (this Journal, 1894, 272) believe that rhodinol has the formula  $C_{10}H_{20}O$ ; Barbier, however (Comptes rend. 117, 177), confirms Eckart's formula.

The present authors were induced to make the following experiments on account of the statement of Monnet and Barbier (this Journal, 1894, 658) that Oil of Pelargoniums contains a considerable quantity of rhodinol. They have therefore treated the various geranium oils of commerce with alcoholic potash, and, after driving over the oil with steam, fractionally distilled it *in vacuo*. In no case was a compound isolated having the properties of Eckart's rhodinol, but the fractions which are said to contain rhodinol consist chiefly of geraniol.

The authors draw attention to the conflicting statements regarding the physical properties of rhodinol. They have isolated Geraniol from Bulgarian and German oil of roses by means of its compound with chloride of calcium (Jacobson, Annalen, 157, 231), from "palmrosa oil" (*Andropogon Schoenanthus* L.), African geranium oil, and citronella oil. They confirm Bouchardat's observation that Barbier's licanthol from linalool is identical with geraniol. The difference in specific gravity of the geraniol from various sources (0.8834—0.8801) may possibly be attributed to the ready oxidisibility of the compound when exposed to the air.

The chief constituent of rhodinol, and consequently also of otto of roses, may therefore be regarded as geraniol, but the nature of the minute impurity which imparts the perfume of roses to the geraniol is unknown.

In opposition to the views of Markownikoff and Reformatzky (*loc. cit.*), the authors consider that the stearoptene of otto of roses is a mixture of hydrocarbons.—A. R. L.

*Champacol.* E. Merck. Arch. Pharm. 231, 123.

CHAMPACA-wood contains an aromatic substance which is obtained by distillation with steam. It is a new camphor of the formula  $C_{17}H_{26}O$ , which, when recrystallised from dilute alcohol, melts at from 86 to 88° C. It forms white needles soluble in alcohol and ether, less soluble in water. The pure substance is odourless and can be sublimed.

—C. O. W.

*The Preparation of Pure Digitonine.* H. Kiliani. Arch. der Pharm. 231, 460.

DIGITONINE crystallises from 85 per cent. alcohol with the composition  $C_{57}H_{100}O_{13}$ , 511.0. To obtain large prisms, it is necessary to keep the saturated solution at 45° for about eight hours; at lower temperatures, minute lamellae are obtained, and these are less pure.—E. R. B.

*Note on the Preparation of Free Hydroxylamine.* J. W. Bruhl. Ber. 27, 1347.

The author, in opposition to Lobry de Bruyn, states that he can prepare several hundred grms. of free hydroxylamine at one operation, by means of his vacuum fractionating apparatus. (This Journal, 1893, 1054. *Oximides*.)—J. W.

*The Investigation of Saccharin.* R. Hefelmann. Pharm. Central H. 1894, 105.

1. *Para-Acid Determination.*—10 grms. of saccharin and 100 cc. of sulphuric acid of 1.31 Tw. are immersed for three hours in a briskly boiling water-bath, with occasional agitation. The decomposition is completed when a drop, on considerable dilution with water, is free from a sweet taste. The acid solution is diluted with an equal volume of water, allowed to cool, and let stand over night. No precipitate will be formed under these conditions from pure saccharin, but in the presence of the para-acid the whole of it separates out on standing for 12 hours up to three days, or on addition of a small crystal of the substance. The precipitate is filtered off, washed with a little water, and transferred from the parchmented filter-paper into a beaker and from this on to a fresh filter-paper for freeing it from all the sulphuric acid. It is dried at 100°, separated from the filter, and weighed. The para-acid ought not to taste sweet, but acid; no sulphuric acid reaction ought, however, to be obtained. Its melting-point is between 270°–280°. The washing and weighing of the precipitate may be performed in a Gooch crucible.

2. *Saccharin - Nitrogen Determination.*—The filtrate from the para-acid is made up to 500 cc., 50 cc., equal to 1 gm., are supersaturated with caustic magnesia, and the ammonia distilled off without cooling, into 20 cc. of half-normal sulphuric acid, titrating back with one quarter normal caustic potash, using methyl-orange or rosolic acid as indicator. The acid consumed is equivalent to the nitrogen decomposed. One hundred per cent. saccharin contains 7.67 per cent. of nitrogen. One per cent. of nitrogen corresponds to 13.04 per cent. of saccharin.

3. *Determination of the Total Nitrogen according to Kjeldahl.*—One gm. of saccharin is weighed off into a thin-walled vessel and introduced into Kjeldahl's decomposing flask, where it is boiled for 2 hours with 25 cc. of concentrated sulphuric acid and 0.5 gm. of mercury. On cooling, the solution is diluted with water, introduced into a flask of 1 litre capacity, and further diluted to about 250 cc. Excess of potassium or sodium hydrate is now added along with 3 grms. of well-washed zinc-dust free from nitrogen (or, if desired, excess of potassium sulphide solution), and the ammonia distilled off by boiling for one hour as described under 2).

Commercial saccharins are sufficiently characterised when proceeding as under 2 and 3, and a gravimetric para-acid determination is thus obviated. As an illustration the following results were obtained:—

	Saccharins, 300 times sweeter than Sugar.	
	Heyden.	Fahlberg.
Moisture .....	0.28	0.23
Ash, without addition of sulphuric acid.	0.82	0.50
Dried substance, less ash .....	98.92	99.47
Benzoyl- <i>p</i> -sulphonic imide .....	32.47	36.37
Nitrogen of the filtrate from the <i>p</i> -acid.	5.19	1.06
Total nitrogen.....	7.37	7.15
Saccharin, calculated from the nitrogen of the filtrate from the <i>p</i> -acid.	67.66	69.75

—H. A.

*Coniine and its Compounds.* J. Schorm. Zeits. angew. Chem. 1894, 266–267.

When treating henlock seed with sodium carbonate solution and distilling with superheated steam, the base is found in the distillate, partly as the carbonate, soluble in water. This circumstance may be made use of for producing the pure base. To this end the distillate is neutralised with hydrochloric acid, the aqueous solution boiled down, and any oily impurities present decomposed by prolonged heating on a sand-bath until the residue is odourless. On dissolving in water and filtering, a clear solution ought to be obtained, but if tinged, it may be purified by treatment of the cold neutral solution with commercial 3 per cent. hydrogen peroxide solution. Beautiful and clear coniine crystals are formed on boiling down the solution, and further crops may be obtained by repeated evaporation. The final mother-liquor contains conhydrine and a new base, which may be separated by dilution with water, rendering slightly alkaline with caustic soda, and shaking with ether, which dissolves conhydrine, leaving the new base in the residue.

For the preparation of its salts an aqueous solution of coniine forms the starting-point. Such a solution may be produced from coniine chloride by dissolving in five parts of water, treating with potassium carbonate as long as turbidity is produced, pouring off the clear liquor from the potassium chloride, and distilling. The aqueous distillate is a 10 per cent. coniine solution, and floating on this is the pure base in the form of the hydrate. The specific gravity of the latter is 0.9 at 12 C., and on heating above 100° 20 per cent. of water separates out, and anhydrous coniine remains. This distils at 168° (760 mm.), and has a specific gravity of 0.87.

Purification with hydrogen peroxide, as described, may be made use of with advantage for obtaining other alkaloids. —H. A.

*Mercuric Phenolates and Certain of their Derivatives.* E. Desesquelle. Bull. Soc. Chim. 11, 1894, 263–269.

*Naphthol compounds.*—By the action of mercuric chloride on potassium  $\beta$ -naphthol in aqueous solution, a compound has been prepared answering to the formula  $\text{HgCl}(\text{OC}_{10}\text{H}_7)$ . This substance, which may be called mixed chloride and naphtholate of mercury, and for which the author suggests also the name  $\beta$ -sublimo-naphthol, is soluble in alcohol, but very slightly soluble in water. Its alcoholic solution on cooling deposits colourless prismatic crystals, which, after a time, become coloured.

On pouring a solution of mercuric chloride (1 mol.) into an aqueous solution of potassium- or sodium- $\beta$ -naphthol (2 mols.)  $\beta$ -naphtholate of mercury is precipitated. The precipitate, at first yellowish-white, becomes colourless after 24 hours. The compound is insoluble in the ordinary solvents, but soluble in phenol or in a boiling solution of phenol. Its formula is  $\text{Hg}(\text{OC}_{10}\text{H}_7)_2$ . It is attacked by acetic acid with the formation of a mixed acetate and  $\beta$ -naphtholate  $\text{HgOOC}_2\text{H}_5(\text{OC}_{10}\text{H}_7)$ , consisting of colourless crystals, soluble in alcohol, but very slightly soluble in water.

*Phenol compounds.*—The corresponding phenol compounds are not prepared with the same ease. The mercuric phenols hitherto prepared are amorphous precipitates consisting of mixtures of several substances and of various colours. Many such are sold the composition of which does not in the least correspond with the formula on the label.

The precipitate obtained on pouring potassium phenol (1 mol.) into mercuric chloride (1 mol.) is brick red, and contains chlorine; it answers to no definite formula. If the solutions are heated before adding them together, the brick-red precipitate becomes colourless after standing for 48 hours. On purification, by crystallising from alcohol, it is obtained in the form of colourless crystals, which melt with decomposition at 210 C.; the crystals are soluble in phenol and possess the formula  $\text{HgCl}(\text{OC}_6\text{H}_5)$ . This may be called mixed chloride and phenolate of mercury or sublimo phenol.

If, at a low temperature, a solution of mercuric chloride (1 mol.) be poured into an aqueous solution of potassium

phenol (2 mols.), the white precipitate so formed contains 64.12 per cent. of mercury and 9.62 per cent. of chlorine. With 4 mols. of potassium phenol the precipitate contains 59.57 per cent. of mercury and 3.42 per cent. of chlorine.

If mercuric chloride solution is poured into potassium phenol until the precipitate which at first redissolves becomes persistent, it is found that at this moment one molecule of mercuric chloride has been used for eight of the phenol. On filtering, the filtrate deposits on cooling faintly pink crystals, which on recrystallising from phenol are colourless, and answer to the formula  $\text{HgO} \cdot \text{OC}_6\text{H}_5(\text{OC}_6\text{H}_5)_3$ , that is, they consist of hydroxyphenolate of mercury. This product may be also obtained, but in a coloured form, by the action of 1 mol. of mercuric chloride on 4 mols. of potassium phenol at the boiling temperature.

Acetic acid acts on the hydroxyphenolate so as to yield a mixed acetate and phenolate,  $\text{HgO} \cdot \text{OC}_2\text{H}_3(\text{OC}_6\text{H}_5)_3$ , consisting of colourless prismatic crystals, very slightly soluble in water or alcohol, fusible with decomposition at an elevated temperature.

Up to the present it has not been found possible by the above methods to prepare the phenolate of the formula  $\text{Hg}(\text{OC}_6\text{H}_5)_2$ .—R. B. B.

*The Alkaloids of the Papaveraceæ.* C. and G. Kænig and W. Tietz. Arch. d. Pharm. **231**, 136—184.

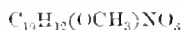
A. ALKALOIDS CONTAINED IN THE ROOT OF *Sanguinaria Canadensis*.—

(1.) *Chelerythrine*,  $\text{C}_{21}\text{H}_{17}\text{NO}_4 + \text{C}_2\text{H}_6\text{O}$ , exists also in the root of *Chelidonium majus* and of *Glaucium luteum*. It crystallises in small rhombohedra, which melt at 203° C., is soluble in chloroform, with a blue fluorescence, but less soluble in alcohol. Acids colour it an intense yellow; ammonia reprecipitates the white base. With Erdmann's reagent, a yellow coloration; with Fröhde's reagent, yellow colour becoming dirty green; with vanadyl-sulphuric acid, a red-violet colour. This alkaloid contains two methoxy-groups, and its formula may be written—



It forms a crystalline hydrochloride and other salts.

(2.) *Sanguinarine*,  $\text{C}_{20}\text{H}_{15}\text{NO}_4 + \text{H}_2\text{O}$ .—Crystalline needles, melting at 213° C., soluble in chloroform, alcohol, and acetone. Its solutions exhibit violet fluorescence, and its salts are blood red. The base itself acquires a pink colour when exposed to air. With sulphuric acid, yellowish-red colour; nitric acid, brownish; Erdmann's reagent, orange becoming scarlet; Fröhde's reagent, earmine-red becoming dirty brown; vanadyl-sulphuric acid, dark green becoming violet, and finally brown. Its formula is—



It forms crystalline salts.

(3.) *γ-Homochelidonine*,  $\text{C}_{21}\text{H}_{17}\text{NO}_3$ , is identical with the *β*-homochelidonine extracted by Selle from *Chelidonium majus*. Crystallises in small needles or in large plates, which melt at 169° C. Sulphuric acid gives a violet coloration becoming brownish-yellow; nitric acid, a yellow; Erdmann's reagent, light yellow, then violet, and finally yellow again; Fröhde's reagent, brown, passing to violet, blue, green, and yellow; vanadyl-sulphuric acid, violet becoming greenish-blue, and finally light brown. This alkaloid contains two methoxy-groups.

(4.) *β-Homochelidonine*,  $\text{C}_{21}\text{H}_{17}\text{NO}_3$ .—Crystalline needles melting at 159° C.

(5.) *Protopine*,  $\text{C}_{20}\text{H}_{15}\text{O}_3$ , is also found in the roots of *Chelidonium majus*, *maclaya cordata*, and *escholtzia californica*. It crystallises in two forms: from chloroform, in glassy crystals; from ether or acetone, in fine needles. Both forms melt at 207° C., and are insoluble in water. Coloration with sulphuric acid violet, becoming dirty green; nitric acid, yellow; Erdmann's reagent, in turn yellow, violet, green, and yellow; Fröhde's reagent, bluish-green; vanadyl-sulphuric acid, red violet, passing to dark blue.

B. THE PROTOPINE OF *Chelidonium majus*.—This only differs from that just described in a few minute details.

C. THE CHELERYTHRINE OF *Chelidonium majus* differs only from the chelerythrine of *Sanguinaria* in forming salts which are bright yellow in colour instead of orange.

To extract this Chelerythrine, the root is pulverised and then exhausted with boiling alcohol of 96 per cent., after having first separated the other alkaloids. The filtered liquid, after treatment with hydrochloric acid, on standing, deposits a mass of brilliant needles, more or less coloured, and mixed with short prisms—indeed, a mixture of hydrochlorides. These are decomposed by ammonia. The bases dissolve in alcohol and chloroform, and there subsequently separate rose-tinted rhombohedra with a small quantity of white needles, melting at 206°, and very unstable in the air. Chelerythrine melts at 203°, and dissolves in acetic ether with blue fluorescence.—R. B. B.

*Preparation of Quinoline.* J. Walter. J. Prakt. Chem., [2], **49**, 549.

See under IV., page 798.

*The Decomposition of Tartaric and Citric Acids by Sunlight.* W. Seekamp. Annalen, **278**, 373.

See under VII., page 810.

## PATENTS.

*Improvements in or connected with the Production of Oxygen from Atmospheric Air, and Apparatus to be Employed therein.* Brin's Oxygen Co. and K. S. Murray, London. Eng. Pat. 14,918, August 3, 1893.

IN the manufacture of oxygen from an absorbent material under reduced pressure the oxygen begins to come off before the whole of the residual air has been removed from the retorts, &c., and thus the first portions of oxygen become contaminated with nitrogen and must be wasted. With the view of minimising this loss it is proposed, immediately pumping out is commenced, to connect the retorts, preferably at their lower ends, with the oxygen holder, thus admitting oxygen. It is claimed that this oxygen displaces the air and enables it to be drawn off with less loss of oxygen than is at present suffered. Automatic gear is described, which opens connection with the oxygen directly the retorts are put under vacuum and closes it again at such time (regulatable at will) as it is considered the air should have been withdrawn.—L. T. T.

*Manufacture of Caffeine Sulphonates.* O. Imray. From "The Farbwerke vormals Meister, Lucius, and Brüning," Hoechst-on-the-Main, Germany. Eng. Pat. 16,949, September 8, 1893.

A SALT of caffeine sulphonic acid results when bromo- or preferably chloro-caffeine is heated with a solution of a neutral sulphate. In practice 100 parts of chloro-caffeine and 75 parts of sodium sulphide (anhydrous) are dissolved in 1,000 parts of water and the solution heated, preferably, in a digester, to 150° C. for eight hours. On cooling the sulphonated salt separates out almost entirely. When purified sodium caffeine sulphonate forms a white powder, easily soluble in water, but insoluble in alcohol.—H. T. P.

*An Improved Iodine Liniment.* J. J. Anning, Leeds. Eng. Pat. 17,209, September 13, 1893.

A SOLUTION of iodine in oleic acid. It is claimed that it will not stain, and is readily absorbed by the skin.

—H. T. P.

*A New Improved Process for the Manufacture of Anhydro-ortho-sulphamine Benzoic Acid (Ortho-benzoic Sulphinide) or of its Salts from Commercial Saccharin.* A. R. Ling, Wimbledon. Eng. Pat. 21,417, November 10, 1893.

COMMERCIAL saccharin consists of a mixture of anhydro-ortho-sulphamine benzoic and para-sulphamine benzoic

acids. The new process for separating them is based on the fact that the ortho-acid readily decomposes the neutral salts of certain weak acids (acetic, citric, &c.), and passes into solution, whilst the para-acid is unaffected and remains undissolved. In practice, sodium acetate may be used at the rate of 1 part of acetate dissolved in 10 parts of water to 2 parts of 60 per cent. saccharin, the reaction being allowed to proceed in the cold, or assisted by heat. In either case the cold liquid is filtered from the insoluble para-acid. The filtrate contains the pure sodium salt of the ortho-acid, together with an equivalent amount of acetic acid. The latter may be recovered by distillation, and the ortho-acid finally isolated by any suitable method.

—H. T. P.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*The Annual Report (Eighteenth) of H. M. Inspectors of Explosives for 1893.*

In this report attention is called to the retirement of Col. Cundill from his position as inspector on account of ill-health. His place has been filled by Capt. J. H. Thomson, R.A.

The only modification made in the law during the year is the authorising of the packing of smokeless powders containing nitroglycerin in tin canisters, so as to put them on the same footing as powders composed essentially of nitro-cellulose.

The total number of factories licensed to manufacture explosives is now 127, one new factory having been licensed and three old ones having ceased to exist since the previous year. The inspectors say that "A more intimate acquaintance with the properties of nitro-cotton, which has been brought about in a large measure from its employment in the manufacture of the various smokeless powders, has brought to light the fact that its explosiveness does not depend, as hitherto supposed, on the degree of its insolubility. All nitro-cotton, however soluble, is now admitted to be 'explosive.' The question, therefore, whether collodion cotton, which is largely used for the manufacture of collodion, should be ranked as an explosive has again come under our consideration, and we have decided that for the present there is no necessity to bring it within the scope of the Act, so long as it is either in solution (in alcohol and ether) or wet, or saturated with methylated spirit and inclosed in air-tight cases, in any one of which states it is available for conversion into collodion." The amount of foreign nitroglycerin compounds suitable for blasting purposes imported during the year was 576,950 lb., being a large decrease on 1892, when the total was 850,000 lb.

Dr. Dupré, in his report to the inspectors, again calls attention to the liability to oxidation so often found in the samples of gelatin dynamite examined, and insists on the necessity of great care being used in this manufacture. Two new smokeless powders, "Westfalite," consisting of ammonium nitrate and gum lac, and "Von Forster's Powder," consisting of gelatinised nitrocellulose with a small proportion of chalk were favourably reported on, and a "Chlorate" powder, "Schnebelite," passed the preliminary tests and is still under examination. In consequence of an explosion caused by sodium peroxide a series of experiments was undertaken to ascertain how such an explosion could have originated. "These experiments clearly demonstrated that peroxide of sodium by itself is a perfectly stable body, not liable to spontaneous decomposition, and not liable to explode by percussion, friction, heat, or by the addition of water, although in the latter case much heat is evolved and steam produced. When, however, the peroxide is mixed with, or even simply in contact with any combustible substance, it becomes a highly dangerous material, inasmuch as, under these circumstances, the

access of water to the mixture causes the almost instantaneous outbreak of fire, or causes an explosion according to the greater or less intimacy of the mixture. The precautions suggested by this explosion, fortunately unattended with loss of life or serious damage to property, are obvious. Peroxide of sodium should always be packed in a strong metallic case not liable to injury during transport, and all contact, or possibility of contact, with any combustible matters carefully avoided." The number of deaths from accidents in factories is six, as against two in 1892; the average for the past ten years being 5.9.

Owing to the repeated accidents which have been caused by mixtures of potassium chlorate and sulphur, steps are being taken to prohibit the manufacture or use of fireworks containing these ingredients, except under approved conditions. The chief explosions which occurred at home and abroad are described and discussed. An interesting case is the burning without explosion of 4.212 lb. of gun-cotton at Waltham Abbey, and details gathered in regard to the terrible explosion of about 30 tons of dynamite at Sandaer show that structural damage did not extend beyond 650 yards, a distance only one-third of that which has to be kept clear as a proper safety zone round magazines containing comparable quantities of explosive under the Explosives Act.—W. M.

*The Inventor of Phosphorus Matches.* E. Jensch. Zeits. angew. Chem., 1894, 268-269.

RÖMER, Preschel, and Irinyi are variously named as inventors of phosphorus matches. From the testimony of a still living college friend, it appears that the real inventor is the Hungarian, János Irinyi. It was in 1835 when the latter, then 19 years old and a student at the Polytechnic School in Vienna, attended Professor Mössner's lectures on chemistry. He became greatly impressed by a demonstration of the reaction produced on rubbing together peroxide of lead and sulphur. It struck him straightway that the reaction may be greatly intensified when substituting phosphorus for sulphur. Irinyi was not to be seen at the college for the next few days. His friend wishing to see him called at his rooms but found the door locked, and on giving his name, received the unmistakable answer, "Geh' weg Schwab, ich mach' eine Erfindung." On joining his friends Irinyi had his pockets full of matches which he struck on the walls, all of them taking fire. He prepared them by melting phosphorus in a concentrated solution of glue, and shaking until the mass became cold and all the phosphorus assumed a finely divided state. This emulsion was mixed with brown peroxide of lead, and sticks previously dipped in molten sulphur, were immersed in the mixture. He sold his invention to a merchant named Römer for about 700*fl.* Irinyi is said to be still living in the south of Hungary.—H. A.

ERRATUM.—This Journal, July number, p. 730, col. 1, the abstract "An Improved Still or Retort," &c., should have appeared under XXIII., on page 761.

## XXIII.—ANALYTICAL CHEMISTRY.

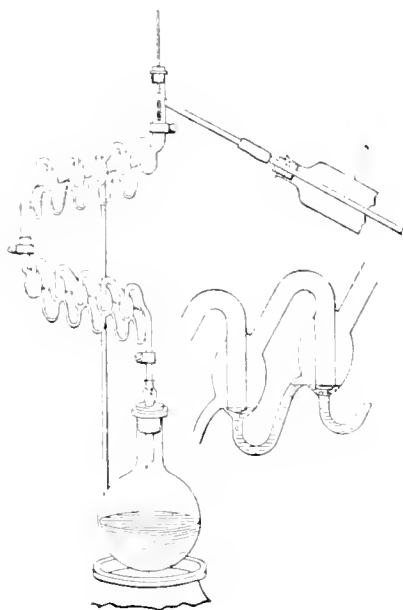
### APPARATUS, ETC.

*Apparatus for Fractional Distillation.* M. Otto. Bull. Soc. Chim. 1894, 11, 197.

The apparatus consists of a series of egg-shaped bulbs connected with each other on the top by wide tubes, and by narrow tubes on the bottom, in the manner shown in the diagram.



Experiments with mixtures of benzene and toluene, and methyl alcohol and acetone, show this apparatus to be vastly



superior to fractionating tubes of the well-known Le Bel-Henninger type.—C. O. W.

*The Prevention of Bumping in Boiling or Distilling Liquids.* V. Gernhardt, Ber. 27, 964–965.

In working out the method for determining the molecular weights of dissolved substances from the lowering of vapour pressure of their solutions, Beckmann introduced a boiling apparatus the function of which was to promote a steady ebullition in the liquid by disturbing the thermal equilibrium at one point by partial superheating, and thus preventing superheating throughout the whole mass of liquid. To attain this end Beckmann sealed a short piece of stout platinum wire through the bottom of the boiling-vessel. As platinum is a much better conductor of heat than glass, the liquid immediately surrounding the platinum wire becomes partially superheated, and a steady evolution of vapour bubbles takes place in the neighbourhood of the sealed-in wire. Vessels of this description, however, are very apt to crack, owing to the unequal expansion of platinum and glass. In a later form of the apparatus the platinum wire is sealed in by means of the red Jena enamel, and the vessel is thus capable of standing abrupt changes in temperature. Whilst using an apparatus of this kind the author noticed that the boiling took place, not from the platinum rod itself, but from the red enamel. Numerous experiments have shown that the expensive platinum may be replaced by the enamel alone, and a large number of such flasks made by Kachler and Martini, of Berlin, for the author, have given entire satisfaction.—J. S.

*Apparatus for Weighing out Liquids.* H. Schweitzer and E. Lungwitz. Chem. Zeit. 18, 529.

The weighing out of liquids which readily volatilise, of fats and oils, of tanning acids, &c., is a lengthy and inconvenient operation. The process is, however, much simplified by the use of the pipette shown in the figure. The exhaustion, which terminates in a capillary, which is bent within the bulb of the pipette towards the upper portions of its walls; the opposite side of the bulb is flattened in order that the pipette may be placed on the pan of the balance; the mouth-piece may be drawn out to a capillary, and the same applies

to the discharge-tube. A sufficient quantity of the liquid to be weighed is sucked up into the bulb so that the end of the capillary within the bulb projects beyond the surface of the liquid when the instrument is laid down on the flattened

Fig. 1.

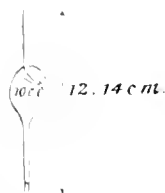
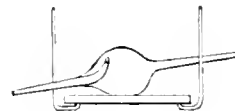


Fig. 2.

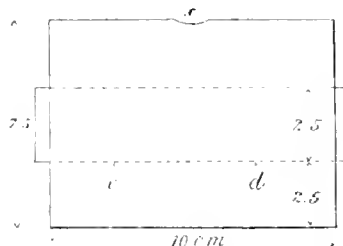


portion. After wiping the end of the tube, the pipette and its contents are weighed; the necessary quantity of liquid is then run out, and the weight redetermined. For weighing solid fats the instrument may be warmed; the error introduced by weighing a slightly warm liquid in a small pipette being for practical purposes quite negligible.—A. R. L.

*Muffle for Sugar Assay.* H. Schweitzer and E. Lungwitz. Chem. Zeit. 18, 529.

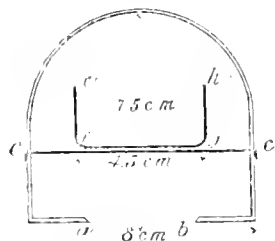
For the determination of the ash in sugar by incineration, Scheibler recommends the use of a small muffle of platinum-foil which may be raised to a red heat. F. G. Wiechmann's sheet-iron muffles have only found limited application because they render the determination too slow. The disadvantages of the platinum muffle are its costliness—a large sugar laboratory requiring four or five,—

Fig. 1.



and the tendency to cause the fusion of the ash by superheating, the consequences of which are well known to every sugar-chemist. The muffle devised by the authors is much cheaper than the platinum muffle, permits of as rapid, and, if possible, more rapid work, and has none of its disadvantages. A strip is cut out longitudinally, as at *a* and *b* (Fig. 2), from a fireclay muffle. On the two ends

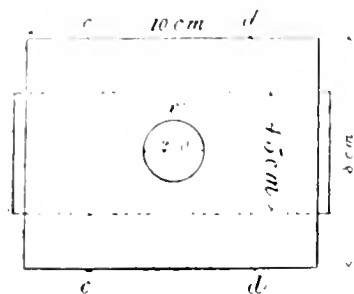
Fig. 2.



of the muffle *c d* (Fig. 1) platinum wires are stretched, *c c'* and *d d'* (Fig. 3), on which the platinum-foil *e f g h* (Fig. 2) is fastened. On the upper portion of the muffle a hole *x* (Fig. 3) is cut out. The platinum dishes are heated

on the sheet *e f g h*, which may also be of sheet iron. A good draught is generally obtained with a muffle of this construction, so that rapid incineration of the sugar is rendered possible. In spite of the large flame of the

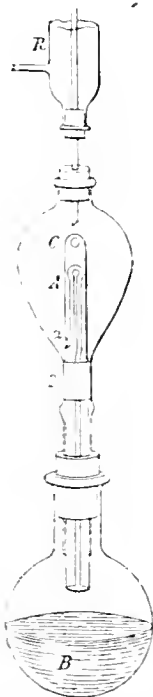
Fig. 3.



so-called "Acme burner," the authors have never observed fusion of the ash. The use of an iron plate renders the assay more lengthy than when platinum is employed, but incineration is just as complete. A great saving of time and also of gas, in comparison to the amount of the latter consumed with fireclay muffles, is effected by employing the new form of muffle.—A. R. L.

**A New Extraction Apparatus.** L. Etai. Bull. Soc. Chim. 11, 1894, 259—260.

THE apparatus consists of an adapter (A), and ground so as to fit into this (2) a large tube, the lower end of which is several centimetres below that of the adapter, and the upper end of which opens laterally (C) into the upper part of the



adapter. The tube contains within it a smaller tube, curved to form a siphon, its shorter branch communicating with the interior of the adapter, its longer branch descending freely to the bottom of the large tube. To make use of the apparatus the tube is withdrawn from

the adapter, a ring of glass-wool introduced at the level of the opening of the siphon (*ca*), and a little glass wool also placed in the lower part of the adapter. The substance to be extracted is put into A, up to the level of the top of the siphon.

For extraction in the cold arrange the apparatus above some kind of reservoir, and below a dropping funnel, so that the solvent falls in drops on to the substance; the solvent accumulating finally covers the substance, and the level of the liquid being now above the curve of the siphon, the latter completely empties the adapter.

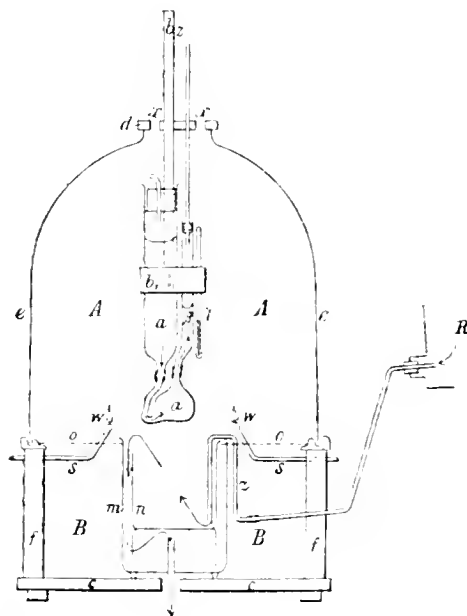
For a hot extraction, arrange the adapter above a flask (B), and connect it with an upright condenser (R). Introduce into the flask a volume of solvent at least equal to the total capacity of the adapter. On boiling the solvent, the vapour liquefies in the condenser, and falls back on to the substance, the further action being as before, with the addition that the vapour in passing upwards serves to keep at a boiling temperature the liquid covering the substance.

The apparatus is made by the firm of Alvergnyat, 10, Rue de la Sorbonne, Paris.—R. B. B.

**New Method of Estimating Pyknometrically the Densities of Soft Fats.** Zdzislaw Ziwalkiewicz. Monath. Chem. 15, 132—138.

In order to determine the relative density at the ordinary temperature, of a fat, &c., semi-solid at that temperature, a pyknometer is used with two orifices, Fig. 1: these are connected by means of india-rubber tubing with two glass tubes serving as reservoirs, *a* and *B*, Fig. 2; a brass ring *b*, supports these, together with a thermometer, *γ*, inside a bell-jar, A A, the interior of which can be heated as required by means of a series of small flames, *w w*. The bell-jar

Fig. 1.



rests on an annular support, B B, containing in its centre a vessel kept full of water, passing through from the outside reservoir B, as indicated by the arrows. To make an observation, the supporting-rod *b*<sub>2</sub> is raised, the larger reservoir *a* filled with the substance to be examined, and the flames *w w* lit; the material gradually melts, and descends into the pyknometer, ultimately filling it and rising up in the smaller reservoir *B*, until an equality of level subsists; in this way the pyknometer is completely

filled with the melted substance without air-bubbles; the temperature should not be more than some 20° above the melting point of the substance. The supporting-rod  $b_2$  is then lowered, so that the pyknometer is immersed in the water some 3 mm. deep. The cooling operation generally requires 1½ to 3 hours, the pyknometer being kept full from the reservoirs, as the warmer material contracts in

Fig. 2.



volume during cooling. Finally the india-rubber tubes, &c. are disconnected, and the pyknometer wiped and weighed. If  $P$  be the weight of the empty pyknometer,  $p$  that of pyknometer full of water, and  $p_2$  that of pyknometer full of fat, the specific gravity of the fat relatively to water is  $d = \frac{p - P}{p_2 - P}$ . Thus, samples of lanolin, yellow vaselin, lard, and butter-fat gave respectively the numbers 0.95178, 0.88273, 0.94083, and 0.93175 at 16°. On examining the density of lanolin at temperatures above and below its melting point, it was found that the rate of expansion was perceptibly greater in the semi-solid condition than at higher temperatures when completely melted; the average diminution in density per 12 rise of temperature being about 0.016 in the first case, and 0.007 in the second.—C. R. A. W.

#### PATENT.

*An Improvement in Hydrometers, Saccharometers, and such like Instruments.* H. S. Keating, Kensington, London. Eng. Pat. 24,266, December 16, 1893.

The graduated stem of a hydrometer or the like, consists of a tube, open at the top, and with a lateral opening below, so that when immersed in a liquid, the latter has free access to the interior of the graduated tube, and the displacement of the liquid is therefore due to the walls of the tube only. Of course, the air-bulb below the graduated tube is closed as usual. This form enables the graduated stem to be made much stouter and stronger, with a scale of great delicacy.—E. G. C.

#### INORGANIC CHEMISTRY.—QUALITATIVE.

*Qualitative Detection of Aluminium.* G. Neumann. Monatsh. Chem. 15, 53—54.

THE methods usually recommended for the qualitative detection of aluminium in presence of iron or chromium, are not entirely satisfactory. The treatment of the precipitate with caustic soda or potash to dissolve out alumina, involves either the use of absolutely pure alkali, or a check-experiment with the reagent as regards alumina and silica therein contained; and thus renders the detection of traces uncertain. Fusion with sodium nitrate, alone or together with sodium carbonate, requires a platinum vessel, porcelain being inadmissible. Classen recommends that the solution in hydrochloric acid should be boiled with excess of baryta-water and filtered; the alumina is precipitated from the filtrate by further boiling with ammonium chloride, noting that traces of barium carbonate may be thus thrown down, owing to absorption of  $\text{CO}_2$  from the air. The following mode of manipulation answers well: the mixed precipitate containing aluminium, iron, and chromium, obtained by means of barium carbonate, is boiled with baryta-water; the filtrate is acidulated with hydrochloric acid, and precipitated hot with dilute sulphuric acid; the filtrate from the barium sulphate thus formed is then treated with ammonia, which precipitates aluminium, if present.

—C. R. A. W.

#### The Qualitative Reactions of Gases.

*Researches upon the Phenomena of Oxidation and Chemical Properties of Gases. Parts I. and II.* F. C. Phillips. Amer. Chem. Journal 16, 163—187, and 16, 255—277. Parts I. and II.

IN the course of an exhaustive research into the nature, analysis, and origin of natural gas (see page 790), the author was impressed by the comparatively small amount of attention which has been hitherto paid to the qualitative analysis of gases generally, and has therefore collected together "the more important reactions of the commonly occurring gases." These reactions are classified as follows:—

1. Oxidation phenomena of hydrogen, carbon monoxide and hydrocarbons in the presence of certain finely-divided metals, and other oxidising agents.

2. Reactions between gases and various compounds in solution and (at high temperatures) when dry.

1. From the experimental investigation of the oxidation phenomena, which usually consisted in passing mixtures of air with hydrogen (10 per cent.), hydrocarbons (3 per cent.), or carbon monoxide (10 per cent.) over palladium, the following conclusions were drawn:—

The temperature of oxidation for mixtures of gaseous hydrocarbons with air is mainly dependent upon the solid bodies with which the gas is in contact.

Two phases may generally be observed in the oxidation process. At first only minute traces of  $\text{CO}_2$  are produced, but the amount gradually increases during the rise of the temperature "through 20, 30, or even more degrees," when suddenly an intense reaction takes place.

Oxidation, mixture and environment remaining constant, does not always take place at the same temperature.

The oxidation temperature is not materially influenced by variations in the proportion of hydrocarbon to air in the mixture. Passing the air and gas mixtures over heated palladium, the order of stability (resistance to oxidation) is: paraffin, acetylene, carbonic oxide, olefines. Hydrogen alone, among combustible gases, is oxidised in the cold.

The lower members of a given homologous series of hydrocarbons are more stable than the upper. "In all cases where air is in excess, oxidation is complete (i.e., yielding only carbon dioxide and water), even though a considerable portion of the hydrocarbon may escape unchanged." With an insufficient amount of air some carbon monoxide may be formed. In oxidation,  $\text{CO}_2$  and  $\text{H}_2\text{O}$  are simultaneously formed.

As regards activity of oxidising-power, the order of the metals is as follows:—Osmium, palladium, platinum, ruthenium, iridium, rhodium, gold. This oxidising-power is apparently not dependent upon atomic weight. The cause of the oxidising-power possessed by these metals is still obscure, for although the majority of them have a tendency to produce an unstable oxide which readily gives up its oxygen to the hydrocarbon, yet platinum, which is nearly as powerful as palladium in inducing oxidation, does not produce an oxide when heated in air. At a bright red heat and with excess of air, palladium asbestos is as efficient as ignited copper oxide, for the oxidation of hydrocarbons. Palladium asbestos containing 2 per cent. of the metal is nearly as efficient as that containing 30 per cent. The statements of Berliner (Ann. der Phys. Wied. n. F. [35], 791) that, in the reaction  $\text{H}_2 + \text{O} = \text{H}_2\text{O}$ , the catalytic action of Pt, Cu, Zn and Al begins at a fixed temperature (which is for these metals 270°, 280°, 350°, and above 440° respectively) and increases with rise of temperature, and that, at constant temperatures, constant quantities of  $\text{H}_2\text{O}$  are formed, are not confirmed by the experiments of the author.

Krause and Meyer state that, in the presence of Hg, hydrogen begins to oxidise at 305°, increasing temperature accelerating the reaction. In contact with glass only, hydrogen burns between the limits 605° and 730°.

2. Qualitative Reaction of Gases.—The recognition of any gas in a complex mixture is still often difficult; there

are but few "groups," like those of Fresenius in the case of metals, and the members of these greatly resemble one another.

The following groups are proposed:—

Group 1. Hydrogen.

Group 2. Carbon monoxide.

Group 3. Methane, ethane, propane, &c.

Group 4. Ethylene, propylene, trimethylene, &c.

Group 5. Acetylene, allylene, &c.

Group 6. Sulphur compounds: hydrogen sulphide, methyl sulphides, carbon oxysulphide (COS), carbon bisulphide.

Group 7. Carbon dioxide.

Ungrouped. Nitrogen, oxygen.

There must be taken into account, as well as the gases permanent under ordinary conditions, the vapours of liquids, such as those of benzene, the lower paraffins, olefines, &c.

It is found convenient to recognise, by their relative intensities, three classes of gas reactions, viz.: (1) those where the reaction is prompt and of quantitative value; e.g., between  $\text{CO}_2$  and  $\text{KHO}$ ; (2) where the reaction is slow, but, time being allowed, no less complete; e.g., the reduction of  $\text{PtCl}_4$  solution by  $\text{H}_2$ ; (3) where it is extremely slow, the products of the reaction being, even then, only present in traces; e.g., the reduction of  $\text{Fe}_2\text{Cl}_6$  solution by  $\text{H}_2$ .

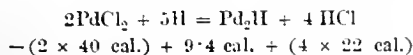
For each gas the author describes two sets of reactions.

(1.) *Reactions in Solution.*—Solutions of palladium chloride, platinum chloride, gold chloride, silver nitrate, ammoniacal silver nitrate, iridium chloride, rhodium chloride, potassium ruthenate, cerium dioxide in dilute sulphuric acid, potassium permanganate (in solutions—neutral, alkaline, and acidulated with sulphuric acid), potassium bichromate acidulated with sulphuric acid, mercuric chloride, osmic acid, ferric chloride, potassium ferrieyanide, ruthenium chloride and fuming nitric acid were usually employed as reagents, though occasionally potassium permanganate crystals in concentrated sulphuric acid, chromic acid, bromine water, hydrogen peroxide, mercurous nitrate, calcium hypobromite in excess of lime-water, sulphuric acid 1·8 sp. gr., iodine in potassium iodide solution, and gold chloride in excess of potassium hydroxide were also employed.

(2.) *Reactions at High Temperatures.*—Here the temperature at which the gas effects the reduction of ruthenium, gold, rhodium, platinum, palladium and silver chlorides, silver bromide, silver iodide, and mercuric iodide, when slowly passed over each of these substances in a heated glass tube, is noted. For hydrogen these temperatures are  $190^\circ$ ,  $150^\circ$ ,  $200^\circ$ , below  $80^\circ$ , in the cold,  $270^\circ$ – $280^\circ$ ,  $330^\circ$ – $360^\circ$ ,  $350^\circ$ – $370^\circ$  respectively, while, as to mercuric iodide, this substance volatilises without reduction. In each case the test is conveniently carried out by passing the resulting gas ( $\text{HCl}$ , &c.) through a dilute solution of a silver salt, the furnace temperature being recorded as soon as precipitation occurs.

With regard to the action of hydrogen upon silver nitrate solution the author corroborates Pellet's observation (see *Comptes rend.* [78], 1132), that hydrogen is unable to reduce a perfectly neutral silver nitrate solution, and that reaction only occurs when some silver oxide is present as well. Russell (*J. Chem. Soc.* [27], 3) states that silver nitrate solution is reduced.

The reduction of palladium chloride by hydrogen occurs at the ordinary temperature; it is an exothermic reaction: thus—



Hence the heat of the completed reaction =  $17 \cdot 4$  cal.

In point of delicacy this reaction, as a test for hydrogen, is superior to all others; the occlusion of hydrogen by the reduced palladium has, however, so far prevented its use in quantitative analysis.

Among the very numerous reactions detailed and commented upon by the author, the following seem to possess particular interest:—

*Methane* and all hydrocarbon gases yield, when passed through a mixture of crystals of potassium permanganate and strong sulphuric acid, carbonic acid. This is a very delicate reaction.

*Ethane*, which is practically identical with methane as to its other reactions, quickly reduces potassium ruthenate, whereas methane only slowly effects reduction.

#### *Distinction between Carbon Monoxide and Olefines.*

(1.) Palladium chloride yields: with  $\text{CO}$ ;  $\text{CO}_2$ : with  $\text{C}_2\text{H}_4$ ; no  $\text{CO}_2$ . Both reduce the  $\text{PdCl}_2$ .

(2.) Platinum chloride yields: with  $\text{CO}$ ;  $\text{CO}_2$ , but not immediately  $\text{Pt}$ , with  $\text{C}_2\text{H}_4$ , no reaction.

(3.) Ammoniacal silver nitrate yields: with  $\text{CO}$ ,  $\text{Ag}$  and  $\text{NH}_4\text{NO}_2$ ; with olefines containing not more than four carbon atoms, no reaction.

(4.) Rhodium chloride: with  $\text{CO}$ ; a slow reduction; with  $\text{C}_2\text{H}_4$ , no reaction.

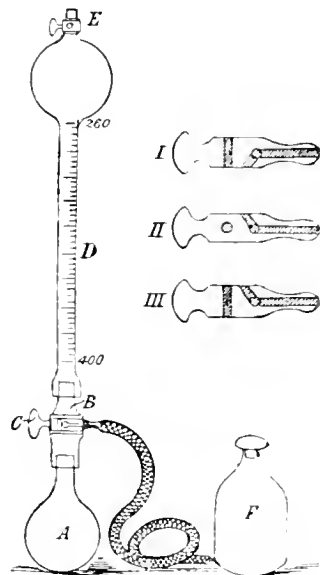
*Isobutylene* among olefines is distinguished by its reduction of cerium dioxide and its absorption of iodine from solutions of this element.

*Trimethylene*, a saturated hydrocarbon and therefore not properly included among the olefines, although isomeric with them, is distinguished from the olefines proper by giving no reaction with either osmic acid or neutral potassium permanganate.—E. R. B.

## INORGANIC CHEMISTRY.— QUANTITATIVE.

*Method and Apparatus for the Analysis of Zinc-Dust.*  
F. Meyer. *J. angew. Chem.* 1894, 231. (*This Journal*, 1894, 765.)

FRESENIUS' method of analysing zinc-dust by passing the hydrogen it evolves with dilute sulphuric acid over cupric oxide in a combustion tube and absorbing the water thus formed in suitable absorption-tubes, gives very accurate results, but is also very tedious. In the apparatus described below, the hydrogen is volumetrically estimated. The apparatus consists of the flask A, tap-joint B, three-way tap



C, burette D, with tap E, and bottle F. The tap-joint, by means of the three-way tap, can be connected either with the flask, which holds about 200 cc., or with the burette, which holds 400 cc., or by means of an india-rubber tube with the bottle, which has a capacity of 500 cc. The capacity of the burette is 250 cc. in the bulb, 140 cc. in the stem, and the latter is divided into  $\frac{1}{2}$  ccs. According to

these dimensions, the apparatus is adapted for the analysis of 1 gram. of samples of zinc-dust containing from 75 to 100 per cent. of metallic zinc, at a temperature of from 10° to 25° C., and a barometric pressure varying from 740 to 770 mm. Before using the apparatus all joints and taps are well greased. Then 1 gram. of zinc-dust is placed in the flask A, the latter connected with the tap-joint, and completely filled with water by placing the tap in the position 1. As soon as all the air is displaced from the flask, the tap is turned into position II., the tap-joint connected with the burette, and the latter filled with dilute sulphuric acid (1 : 3) contained in the bottle F, by lifting the bottle and opening tap E. The burette being full, tap E is closed and tap C brought into position III., when the acid begins to run into the flask. The hydrogen evolved rises into the burette, gradually displacing the dilute acid back into F. When the evolution of gas has ceased, temperature and pressure are noted, the gas in the burette brought under atmospheric pressure by lifting the bottle F, and the volume of the hydrogen observed. The percentage of metallic zinc in the sample is then ascertained by the equation—

$$\text{Percentage of Zn} = 100 \frac{V(b-f)}{760(1+0.00367t)} \times 0.00291135.$$

V = number of cc. of hydrogen, measured at the pressure *b* and temperature *t*; *f* = tension of aqueous vapour at *t*° C.  
—C. O. W.

*The Determination of Phosphoric Acid.* W. J. Williams, F. Bergami, and B. Terne. *J. Franklin Inst.* **137**, 126—128, 129—133, and 138—140.

THESE are three separate papers on the Pemberton method of determining phosphoric acid by titrating the yellow precipitate with standard alkali. (This Journal, 1894, 176). Mr. Williams considers the uranium-nitrate method to be sufficiently accurate for most purposes on an aqueous solution of phosphoric acid, but quite untrustworthy with phosphatic material dissolved in mineral acids, or even acetic acid. He describes a series of tests made to compare the Pemberton volumetric method with the ordinary gravimetric method, and concludes from them that, if Mr. Pemberton's instructions are faithfully carried out by a careful manipulator, the method is accurate and trustworthy, but that with careless or unskilful manipulation the possibilities of serious error are great. Mr. Bergami also describes a series of tests made to compare the Pemberton method with the official one, and is inclined to think the former the more accurate of the two, but he does not consider the number of tests he made great enough to prove the infallibility of it.

Dr. Terne, at whose request Mr. Bergami's tests were made, is of the opinion that the Pemberton method is as trustworthy as the official method, and, if anything, more accurate, whilst the saving of time it effects is a great advantage in a laboratory. He states that he has no hesitation in adopting it at the factory under his control (The Bagn and Sons Co.), though samples for shipment will, at course, have to be analysed according to the rules of the Association. He believes the method to be of sufficient interest to warrant an effort being made to get it adopted, at least as an alternative method, by the Association of Official Agricultural Chemists.—R. B. P.

*The Estimation of Ammonia by Means of the Colorimeter.* L. Hossay de Nagy Hossay. *Bull. Soc. Chim.* 1894, **11**, 216.

MINUTE quantities of ammonia can be most accurately estimated by means of Wolff's colorimeter and a Nessler's solution prepared by dissolving 2 grms. of potassium iodide in 5 cc. of water, adding to the hot solution 3 grms. of mercury iodide, letting the solution go perfectly cold, making another addition of 3 grms. of mercury iodide and 20 cc. of water. The precipitate thereby forming, is allowed to settle, the solution filtered, and to 20 cc. of the clear liquid, 30 cc. of 20 per cent. caustic potash solution

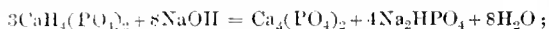
are added. This solution, which is ready for use when perfectly clear, is much more sensitive than the one indicated by Messrs. Hadow and Miller, and gives a distinct coloration with 0.02 mgrm. of ammonia in 110 cc. of water. 1 cc. of this solution is added to the 110 cc. of water contained in a tube of the Wolff colorimeter. The solutions to be examined should not contain more than 0.1 mgrm. of ammonia in 110 cc.

The colorimetric estimation of nitrous acid is best carried out according to Frommendorff's method by means of starch solution and potassium iodide. The solution to be tested should not contain more than from 0.01 to 0.04 mgrm. of nitrous acid in 100 cc. If the solution be too dilute, the reaction is a long time in appearing, and it is also necessary to protect the solution from the light. Preusse and Tiemann's metaphenylenediamine test gives bad results when carried out in the presence of free sulphuric acid. A further drawback is the rapid discoloration of the diamine solution on standing. Griess' test gives very satisfactory results if the solutions to be examined contain not more than 0.05 mgrm. of nitrous acid in 200 cc. The solutions of sulphuric acid and naphthylamine should be preserved separately. The results are equally satisfactory whether the reaction be carried out in the cold or accelerated by heating. The reagents are best employed in solution with acetic acid; sulphuric acid in excess is liable to retard the reaction considerably.

The colorimetric estimation of nitric acid is only possible by reducing this acid to ammonia. This is effected by adding to the solution to be tested from 5 to 6 grms. of reduced iron and sufficient sulphuric acid. Subsequently the solution is made alkaline with caustic potash, diluted with water, and distilled; two Peligot's tubes, each charged with 10 cc. of water and 1 cc. of standard sulphuric acid, serving as receivers. The results obtained may be as much as 5 per cent. too low.—C. O. W.

*The Volumetric Estimation of the Soluble Phosphoric Acid in Superphosphates.* W. Kalmann and K. Meissels. *Mitt. d. k. k. Techn. Gew. Museums*, **4**, 1894 (4, 5, and 6), 174—178.

THE method is based upon the difference of the behaviour of phosphates towards methyl-orange and phenolphthalein. In titrating phosphoric acid, using methyl-orange as an indicator, neutrality is reached when the salt  $\text{RH}_2\text{PO}_4$  has been formed, i.e., when one-third of the acid has been neutralised. With phenolphthalein, neutrality is obtained when the salt  $\text{R}_2\text{HPO}_4$  has been formed, i.e., when two-thirds of the acid have been neutralised. Thus, a solution of  $\text{CaH}_2(\text{PO}_4)_2$  appears neutral to methyl-orange, but acid to phenolphthalein. In titrating a solution of this salt in the presence of the last-named indicator the point of neutrality is reached by way of the reaction—



in other words, 8 mols. of NaOH are equivalent to 3 mols. of  $\text{P}_2\text{O}_5$ . These facts are applied in the analysis of superphosphates as follows:—Twenty grms. of the superphosphates are dissolved in 1,000 cc. of water. One hundred cc. of this solution are titrated with half-normal caustic soda, using methyl-orange as indicator. Then phenolphthalein is added and the titration continued until change of colour occurs, when the number of cc. used is noted (*a*). Another volume of 100 cc. of the original solution is then placed in a 250-cc. flask, a moderate excess of half-normal caustic soda added, the flask well shaken, filled up to the mark with water, and filtered. One hundred cc. of the filtrate are titrated with half-normal acid and phenolphthalein as an indicator; then methyl-orange is added, and the titration continued. The number of cc. used is multiplied by 2.5 (*b*). The mean of the sum of *a* and *b* multiplied by 0.0355 then shows the number of grms. of  $\text{P}_2\text{O}_5$  contained in 100 cc. of the original solution, i.e., in 2 grms. of the superphosphate. The following table shows the results obtained by this as against the citrate method.

Sample of Superphosphate.	a.	b.	$\frac{a+b}{2}$	Per Cent. P <sub>2</sub> O <sub>5</sub> .	
				Volumetrically.	By Titrate Method.
Origin unknown.....	13.4	5.75	9.575	16.74	16.95
" " .....	11.5	3.88	7.69	14.65	14.78
Potash superphosphate.....	7.7	4.31	6.21	16.66	9.98
Boneash superphosphate.....	11.15	3.75	7.45	13.49	13.60
Origin unknown.....	10.70	6.13	8.415	14.94	15.19
Prepared with waste acids from petroleum refining	12.65	6.13	9.39	16.67	16.23
Double superphosphate.....	26.60	19.25	22.925	19.60	19.19
Superphosphate from B. ....	12.70	4.80	8.75	15.54	15.41

—C. O. W.

*The Volumetric Estimation of Phosphoric Acid.*A. F. Holleman. *Rec. des Trav. Chim. des Pays-Bas*, **12**, 1.

This method is applicable to all those cases where the phosphoric acid is present in the free state, or in combination with the alkalis or alkaline earths. If the solutions contain alumina or iron, a correction is necessary. Any heavy metals present are first eliminated by means of sulphuretted hydrogen. The method is not generally applicable in the presence of metals of the third group. The solutions may contain sulphuric and nitric acid, but should not contain any hydrochloric acid.

The solution, if acid, is exactly neutralised by means of a dilute solution of sodium carbonate, and is precipitated by means of an excess of standard silver nitrate. The precipitation is rendered complete by the addition of sodium acetate. The liquid is then filtered, being kept protected from the light. Subsequently the uncombined silver is estimated volumetrically in the filtrate.—C. O. W.

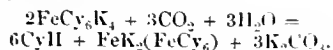
**ORGANIC CHEMISTRY.—QUALITATIVE.**

*Yellow Prussiate of Potash and the Detection of Hydrocyanic Acid in the Presence of Ferrocyanides.* W. Antenrieth. *Arch. Chem. Pharm.* **213**, 99.

CONTRARY to general notions, potassium ferrocyanide is partially decomposed at ordinary temperatures into hydrocyanic acid by dilute (0.1 per cent.) mineral acids, formic, acetic, butyric, lactic, tartaric and benzoic acid. Carbonic acid, sulphuretted hydrogen, the phenols, acetylacetic ether, and certain acid sulphones produce the same reaction at temperatures below 100° C. At the same time a very stable white ferrous ferrocyanide,  $\text{Fe}(\text{Cy})_6\text{FeK}_2$ , is formed, which, on exposure to the air, is not transformed into Prussian blue. The formation of Prussian blue in the preparation of hydrocyanic acid can be explained in two ways. Either the sulphuric acid decomposes the ferrous ferrocyanide, forming ferrous sulphate, which, in the presence of air, acts upon the hydrocyanic acid, or the sulphuric acid liberates some hydroferrocyanic acid, which is converted into Prussian blue according to the formula—



The action of the carbonic acid upon the yellow prussiate is accordingly—



The gastric juice decomposes the ferrocyanide at 40° C. The peptones and casein at 38° C. produce only traces of hydrocyanic acid, large quantities at a boiling temperature. Hydrocyanic acid not being retained by the tissues, it follows that yellow prussiate has no toxic action, as it is not absorbed simultaneously with acids. These observations

show that the usual method of detecting hydrocyanic acid and cyanides in the presence of ferrocyanides by distillation in a current of carbonic acid cannot legitimately be employed. Jacquemin proposed to distil the mixture with sodium bicarbonate, which does not decompose ferrocyanides, and in this manner 0.01 grm. of cyanide may be detected in a 10 per cent. solution of ferrocyanide. If the presence of mercurous cyanide be suspected, a few cc. of sulphuretted hydrogen water are added. Mercurous cyanide is very stable towards alkalis, alkaline carbonates, and dilute acids. To detect it in forensic investigations the substance is distilled with tartaric acid and a concentrated solution of sulphuretted hydrogen. Mercury cyanide is fairly soluble in ether, but cannot be extracted from aqueous solutions by means of that solvent.—C. O. W.

*Camphor as a Reagent for Sugar.* Neitzel. *Deutsch. Zuckerind.* **17**, 254.

The author recommends camphor instead of  $\alpha$ -naphthol as a reagent for sugar. Camphor is available for the detection of the smallest quantities of sugar met with in commercial investigations, but is not, like  $\alpha$ -naphthol, affected by the presence of small quantities of nitrates.—A. R. L.

**ORGANIC CHEMISTRY.—QUANTITATIVE.**

*The Analytical Constants of Seal Oil.* A. C. Chapman and J. R. Rolfe. *Chem. News*, 1894, **70**, 1—2.

THE usual analytical figures for oils have been scantily recorded in the case of seal oil, and the authors therefore give the following table for six samples obtained from reliable sources. They also quote the results obtained from a sample of seal oil blown for 45 hours at a temperature of 190° F.

A study of these figures leads to the following conclusions:—The specific gravity of the oil varies but little save in the case of the "brown seal," which is low in specific gravity, probably from the presence of much free fatty acid. The melting-points of insoluble fatty acids were determined by the capillary tube method, the point at which flow began being taken. The iodine absorption was carried out as usual with a large excess of iodine, the time adopted being five hours. Absorption continued on increasing the time, so that values of 140 to 151 were obtained after 12 hours. The bromine absorption was performed by the method prescribed by Mills and Snodgrass (*this Journal*, 1883, 435); 0.15 grm. of oil was dissolved in 25 cc. of dry, redistilled carbon disulphide, excess of a solution of bromine in the same solvent added, and the mixture allowed to stand in the dark for 15 minutes. Addition of potassium iodide and titration of the liberated iodine completed the

Oil.	Colour.	Sp. Gr. 15°-15°.	Insoluble Fatty Acids.	Melting- Point of Fatty Acids.	Iodine Absorp- tion.	Bromine Absorp- tion.	Total Acid Number.	Saponifi- cation Equivalent.	Free Fatty Acids (as Oleic).	Reichert's Test.
			Per Cent.	° C.						
Pale seal, No. 1	Very pale..	0.9258	93.8	22.0	136.4	77.2	19.6	286	1.13	0.22
Pale seal, No. 2	Very pale..	0.9249	93.6	22.0	133.0	77.6	19.4	289	1.15	..
Pale seal, No. 3	Very pale..	0.9255	94.2	23.0	141.0	79.8	19.04	294.6	0.98	0.07
Tinted seal...	Yellow.....	0.9263	92.8	23.0	137.4	80.0	19.6	286	1.41	..
Straw seal....	Light brown	0.9261	93.5	22.5	139.0	78.2	..	..	1.09	0.13
Brown seal....	Dark brown	0.9226	94.0	23.0	123.5	69.6	19.24	291.5	19.95	..
"Blown" seal	Very dark..	Sp. Gr. 20°-20°. 0.9845	73.4	23.0	78.2	55.5	22.1	253.8	16.5	0.45

operation. On increasing the time of absorption to one hour the bromine absorption increased, figures as high as 89 being obtained, but in this case substitution as well as addition of bromine took place, as was evident from the evolution of hydrobromic acid. The authors note that the results of the iodine absorption are usually more regular than those of the bromine absorption, the former being less influenced by slight variations of the conditions of experiment. The series of figures recorded under the head "total acid number" refers to the percentages of caustic potash needed for the saponification of the oils. The results given in the column headed "Reichert's test" express the weight of caustic potash in grams required to neutralise the volatile fatty acids obtained from 100 grms. of the oil—2.5 grms. being used in the experiments. The determination was made in order to ascertain what difference is produced when seal oil is blown. With regard to the blown seal examined it is mentioned that whereas the unblown oils had viscosities varying from 60 to 64 at 15° C. (rape oil at the same temperature being taken as 100), the blown oil was so viscous that at 50° C. it took 63 times as long to flow through a standard orifice as did the same volume of rape oil.—B. B.

*Acidimetric Estimation of Naphthalene, Acenaphthene,  $\alpha$ - and  $\beta$ -Naphthol, &c.* F. W. Küster. Ber. 27, 1101—1105.

THIS method of estimation applies only to such substances as form insoluble molecular compounds with picric acid. If naphthalene, acenaphthene, chrysene, or one of the naphthols be digested on the water-bath with a measured quantity of an aqueous solution of picric acid (nearly saturated at ordinary temperatures = about 0.05 normal) the original substance gradually disappears, and in its place an equivalent quantity of the molecular picric acid compound separates out quantitatively either at once or on cooling. The amount of picric acid in the original solution and in an aliquot portion of the filtrate from the molecular compound can easily be determined by titration with decinormal barium hydroxide (using lacmoid as indicator), and from these data it is easy to calculate the quantity of picric acid contained in the precipitate. Simple multiplication by the equivalent factor then gives the amount of naphthalene, &c., taken.

The digestion on the water-bath must take place in hermetically-closed vessels. For this purpose, the author recommends the following process:—The substance to be determined, together with the measured volume of picric acid of known strength in excess, is placed in a flask, which must be nearly filled with the solution. The flask is then closed with a good india-rubber stopper, through which passes a tube about 7 cm. long. The tube is sealed up at the lower end, and about 1.5 cm. above the sealed end there is blown a small hole in the side. When the side hole is just below the stopper the air in the flask may be pumped out; then, on withdrawing the tube until the sealed end is flush with the under side of the stopper, the

flask, having a vacuum in the upper part, may be disconnected from the pump and safely heated in the water-bath. The flask must be heated until the reaction is complete (2 hours—2 days), and frequently shaken during cooling, in order to wash down any naphthalene, &c. which may have sublimed into the upper part of the flask. After standing for a few hours, the picric acid in the filtrate is determined by titration.

The author gives several series of test analyses which show that the method is very exact, except in the case of  $\beta$ -naphthol, where a correction must be introduced owing to the solubility of the molecular compound. For every 100 cc. of picric acid solution, 0.0075 gm. of  $\beta$ -naphthol remains unprecipitated.—J. S.

*Composition and Analysis of Yeast.* T. Guichard. Bull. Soc. Chim. 1894, 11, 230.

THE moisture in yeast is estimated by drying the yeast cut into small shavings, at 120° C. Pure yeast contains 71 per cent. of moisture. It loses the whole of its moisture over calcium chloride, and all but 7 per cent. over sulphuric acid. With its moisture it also loses its fermenting capacity. The specific gravity of yeast (by suspension in a mixture of alcohol and chloroform) was found to be 1.180 (in a solution of hyposulphite (thiosulphate) of soda) the figures 1.180 and 1.183 were obtained at 16° C. Water agitated with pure yeast does not reduce Fehling's solution. The residue obtained on boiling yeast with alcohol and evaporating the latter, has no reducing action, but gives a precipitate with barium hydrate. Yeast contains always traces of starch, amounting to from 0 to 2.04 per cent. On ignition moist yeast yields from 1.94 to 2.16 per cent. of ash. The percentage of nitrogen estimated by Kjeldahl's and the soda-lime processes was found to be 5.32 and 6.16 per cent. respectively, corresponding respectively to 33.25 and 38.50 per cent. of nitrogenous matter. This nitrogenous matter consists of nucleine. Petroleum ether extracts from yeast 1.4 per cent. of fatty matter. Starch added to yeast is estimated by boiling for one hour, with reflux condenser, 10 grms. of the yeast with 100 cc. of dilute (5 per cent.) nitric acid. The liquid after cooling is filtered, if necessary, decolorised by means of animal charcoal, and examined in the polarimeter. By multiplying the number of degrees observed by 8.52, the result gives the percentage of anhydrous starch in the yeast; by multiplying by 9.3 the percentage of air-dry starch is obtained. The quantities of added starch in commercial yeast range from 6.2 to 35 per cent. The results of the chemical analysis of yeast are not sufficient to test its quality; for this reason the fermenting capacity of a sample should also be examined. For the purpose the following new process is proposed. An apparatus is employed consisting of a small bulb with a lateral orifice. The bulb is further provided with a long vertical tube, over which is placed concentrically a wider tube divided into 100 cc. This tube is filled with water, which through a lateral tube, is displaced by the carbonic acid, subsequently

developed in the test. In the bulb is placed 0.5 grm. of the yeast to be examined, diluted with 0.5 grm. of water, 0.5 grm. of sugar dissolved in 5 cc. of water, and 1 grm. of washed sawdust. The lateral orifice of the bulb is then closed with an india-rubber stopper, and the apparatus placed in a water bath at 30° C. The quantity of carbonic acid is then observed every quarter of an hour for two hours and a half. The different readings may be marked on a system of co-ordinates, and a curve is thus obtained rising rapidly to a maximum and falling again with equal rapidity. The flatter the curve the poorer the fermentative quality of the yeast. Otherwise the quantity of carbonic acid evolved is taken as a measure of the quality of a yeast. The strongest yeast examined, evolved 86.2 cc. of carbonic acid in two and a half hours.—C. O. W.

*Gravimetric Methods for Estimating Sugars by their Cupric Reducing Power.* L. Grünhut. Chem. Zeit. **18**, 447—448.

For the gravimetric estimation of sugars Nihoul (Chem. Zeit. **17**, 500) recommends a modified form of the older paper-filtration method, in which the collected cuprous oxide is weighed as cupric oxide, in the place of the more recent asbestos-filtration method, in which the copper is weighed as metal. The experience of the present author is that the paper-filtration method not only gives non-concordant results, but also results which are too low. Experiments in support of this are described. The cause appears to be that cuprous oxide is not completely converted into cupric oxide by one treatment with nitric acid (compare Pavy, Zeits. Zuckerind., 1879, **29**, 291; Allihn, *ibid.* 1882, **32**, 878). When the ignition is performed by a gas-flame the reducing action of the latter appears to have considerable influence on the results. Soxhlet asserts (Zeits. Zuckerind., 1878, **28**, 373) that basic copper nitrate is volatile at 200°, and in view of the possibility of this being the cause of the low results the, the author re-investigated the matter, although Nihoul's experiments refute Soxhlet's observation. The author finds, however, that it is possible to obtain the theoretical quantity of cupric oxide by heating the nitrate for a long time at 200°. It is further shown that the cupric oxide finally obtained contained the same quantity of copper as was originally present in the nitrate. (Compare following abstract.)—A. R. L.

*Gravimetric Methods for the Estimation of Sugars by their Cupric Reducing Power.* E. Nihoul. Chem. Zeit. **18**, 881—882.

In reply to Grünhut (see last abstract) the author quotes experiments showing that the paper-filtration method is more accurate than the asbestos-filtration method. His contention is that the results obtained by the former method are accurate, whilst those obtained by the latter are too high, on account of the retention of organic matter by the asbestos-filter, which is not got rid of by ignition in a current of hydrogen. As to the reducing action of the flame, this is practically nil when not too large a flame is used together with a closed crucible.—A. R. L.

## ANALYTICAL AND SCIENTIFIC NOTES.

*Preparation of Phosphorus by means of the Reducing Action of Aluminium upon Phosphates and on the Oxidation and Inflammation of Aluminium by Sodium Peroxide.* A. Rossel. Bull. Soc. Chim. 1894, **11** 260.

On heating in a test-tube or porcelain crucible a quantity of sodium metaphosphate in contact with aluminium foil, the metal begins to fuse at the same temperature as the phosphate, the mixture inflames, and phosphoric anhydride is formed. If this reaction be carried out in an atmosphere of hydrogen, a distillate of phosphorus is obtained. This reaction may be shown as a lecture experiment. The analytical investigation of the reaction showed the latter to result in the formation of sodium aluminate, but at the same time a compound of aluminium and phosphorus is formed

which is not decomposed by heat but evolves phosphoretted hydrogen in the presence of water. This aluminium phosphide possesses the formula  $AlP_3$ , and it is obtained in the pure state by heating aluminium in a current of gaseous phosphorus. By the above reaction only about 3 per cent. of the phosphorus contained in the metaphosphate are obtained in the free state, but by adding silicic acid to the mass almost the theoretical yield of phosphorus is obtained according to the equation—



The same reaction takes place with metaphosphate of lime and more or less with all the other phosphates of lime excepting apatite. Superphosphates react so violently as to cause an explosion. Of a similarly violent character is the reaction between the sulphates of the alkaline earth metals and aluminium, which results in the formation of free sulphur.

Sodium peroxide is a most useful oxidising agent, which, however, should be used with some caution. Mixtures of aluminium and sodium peroxide ignite spontaneously with great violence and production of a high temperature.

—C. O. W.

*Formation of Sugar in the Liver.* G. Cavazzani. Centralbl. f. Physiol. **8**, 33.

THE quantitative estimation of glycogen and of glucose in the livers of living and dead animal shows (in the case of the latter the relation is exact) that the decrease in the amount of glycogen corresponds with the increase in that of sugar. The author is therefore of opinion, contrary to Seegen's view, that the formation of sugar in the liver takes place chiefly (probably exclusively) at the expense of the glycogen therein contained. The formation of sugar appears to be a true secretion process of the liver, influenced by special nerves.—A. R. L.

## New Books.

TECHNISCH-CHEMISCHES JAHRBUCH. 1892—1893. Ein Bericht über die Fortschritte auf dem Gebiete der Chemischen Technologie vom April 1892 bis April 1893. Herausgegeben von Dr. RUDOLF BIEDERMANN. Fünfzehnter Jahrgang. Mit 240 in den Text gedruckten Illustrationen. Berlin: Carl Heymann's Verlag. 1894. London: H. Grevel and Co., 33, King Street, Covent Garden. 12s.

For 15 years this Year-book of Chemical Technology has now appeared. In this, its last issue, it forms a substantial 8vo volume bound in cloth. An exceedingly useful feature in it is the "Bücherschau," or Report of New Books on various branches of science that have appeared during the year. This report is well classified, and deals with works on: 1. Physics. 2. Chemistry. 3. Chemical Technology. 4. Mineralogy, Geology, and Mining. 5. Miscellaneous. The work commences with an elaborate table of contents, followed by one of the journals of technology and science referred to in its pages. The subject-matter fills 550 pages, which are illustrated by 241 wood engravings. The subjects treated of are: I. Iron and Steel. II. The Alkali Metals. III. Gold and Silver. IV. Copper. V. Lead. VI. Zinc. VII. Tin. VIII. Nickel and Cobalt. IX. Tungsten, Chromium, Platinum Metals, Manganese, and Mercury. X. Chlorine, Bromine, and Hydrochloric Acid. XI. Sulphur, Sulphuric Acid. XII. Oxygen, Carbonic Acid, Nitric Acid. XIII. Soda. XIV. Potash Salts. XV. Ammonia and Cyanogen Compounds. XVI. Alkaline Earths, &c. XVII. Glass. XVIII. Earthenware. XIX. Cement. XX. Artificial Stone. XXI. Explosives. XXII. Illuminants. XXIII. Fuel. XXIV. Sugar. XXV. Starch, &c. XXVI. Wine. XXVII. Beer. XXVIII. Spirits. XXIX.



Fats, Soaps, Ethereal Oils, and Resins. XXX. Water. XXXI. Foodstuffs. XXXII. Manures. XXXIII. Organic Acids. XXXIV. Alkaloids. XXXV. Colouring Matters. XXXVI. Tanning. XXXVII. Glue and other Adhesives. XXXVIII. Textile Fibres. XXXIX. Paper. XL. Photography. XLI. Apparatus. The work concludes with an alphabetical index of authors and subject-matter, and a separate, well-classified index and list of patents, under headings corresponding with those specified above. This index thus serves also a separate and useful purpose, *viz.*, that of a classified record of patents published in the year.

**CHEMISCH-TECHNISCHES REPERTORIUM. UEBERSICHTLICH GEORDNETE MITTHEILUNGEN DER NEUESTEN ERFINDEUNGEN, FORTSCHRITTE UND VERBESSERUNGEN AUF DEM GEBIETE DER TECHNISCHEN UND INDUSTRIELLEN CHEMIE, MIT HINWEIS AUF MASCHINEN, APPARATE UND LITERATUR.** Herausgegeben von Dr. EMIL JACOBSEN. 1893. Zweites Halbjahr. Erste Hälfte. Mit in den Text gedruckten Illustrationen. 1894. Berlin: R. Gaertner's Verlagsbuchhandlung, Hermann Heyfelder, Schönebergerstr. 26, S.W. London: H. Grevel and Co., 33, King Street, Covent Garden.

This is the first issue for the second half-year of 1894, of Dr. Jacobsen's Repertory of Chemical Technology. It contains reports of the progress of the following branches of Chemical Industry:—Building Materials. Dyestuffs, Dyeing, and Calico-printing. Fats, Oils, &c. Fermented Liquors, Tanning, Leather, Glue, &c. Textiles. Glass and Pottery. Wood, &c. India-rubber. Cements, Adhesives, &c. Lakes, Varnishes, and Paints. Metals. The subject-matter covers altogether 200 pages. The issues of this work are quarterly.

**SELECT METHODS IN CHEMICAL ANALYSIS (CHIEFLY INORGANIC).** By WILLIAM CROOKES, F.R.S., Editor of the Chemical News. Third Edition, Rewritten and Enlarged. Illustrated with 67 wood-cuts. London: Longmans, Green and Co. New York: 15, East 16th Street. 1894. 21s.

In this, the third edition of the above-named well-known work, it is stated that such methods as have become thoroughly established since the issue of the second edition, are now removed, and replaced by others which have been proposed and found to be successful during the last eight years. It is also stated, that it must not be assumed, then, that processes thus discarded have ceased to possess value; they are in some cases thoroughly established, but the author, desiring this work to be mainly a collection of novel or little-known processes—"Select Methods,"—has consequently removed them. Other methods have of course disappeared, because, being tried, they have been found less effective than the substitutes now appearing. The book is one of large 8vo. size, and is strongly bound in cloth. It contains a well-classified table of contents, 697 pages of subject-matter, and an alphabetical Index. The last 10 pages of the work are devoted to "Useful Tables": 1. Conversion of Centigrade and Fahrenheit Degrees. 2. Mutual Conversion of French into English Weights and Measures. 3. Relative Values of French and English Weights and Measures. Baume's Hydrometer, Twaddell's Hydrometer, &c., &c.

The methods specified are comprised under the following headings and chapters:—I. Potassium, Sodium, Lithium, Cesium, and Rubidium (Ammonium). II. Barium, Calcium, Strontium, Magnesium. III. The Cerium Group. IV. Chromium, Uranium, &c. V. Zinc, Aluminium, Gallium, and Iron. VI. Manganese, Nickel, Cobalt. VII. Silver, Mercury, Copper. VIII. Cadmium, Gallium, Lead, Thallium, Indium, Bismuth. IX. Antimony, Tin, Arsenic, Tellurium, Selenium. X. Gold, Platinum, Palladium, Iridium, Osmium, &c. XI. Sulphur, Phosphorus, Nitrogen. XII. Iodine, Bromine, Chlorine, Fluorine (Cyanogen). XIII. Carbon, Boron, Silicium. XIV. Electrolytic Analysis, Gas Analysis. XV. Miscellaneous Processes and General Methods of Manipulation.

**JAHRBUCH DER CHEMIE.** Herausgegeben von RICHARD MEYER. Bericht über die Wichtigsten Fortschritte der Reinen und Angewandten Chemie, unter Mitwirkung von H. Beckurts, R. Benedikt, C. A. Bischoff, E. F. Dürre, J. M. Eder, C. Häussermann, G. Krüss, M. Märcker, W. Nernst, and F. Röhmman. III. Jahrgang. 1893. Braunschweig: Friedrich Vieweg und Sohn. 1894. 15s. Extra finely bound, 16s. 6d. London: H. Grevel and Co., 33, King Street, Covent Garden.

This is a handsomely and strongly bound volume of 8vo size, containing Table of Contents, 548 pages of Subject-Matter, and an Alphabetical Index of Subjects and Names of Authors. The subjects treated of are:—I. Physical Chemistry (By W. Nernst). II. Inorganic Chemistry (G. Krüss); Organic Chemistry (C. A. Bischoff). IV. Physiological Chemistry (F. Röhmman). V. Pharmacoeutical Chemistry (H. Beckurts). VI. Chemistry of Foods, &c. (H. Beckurts). VII. Agricultural Chemistry (W. Märcker and W. Schneidewind). VIII. Metallurgy (E. F. Dürre). IX. Fuels and Inorganic Chemical Technology (C. Häussermann). X. Explosives (C. Häussermann). XI. Technology of the Carbohydrates and Fermentation Industries (Märcker, Bühring, and Schneidewind). XII. Coal-tar and Colour-Chemistry (Richard Meyer). XIII. Chemical Technology of Textile Fibres (Richard Meyer). XIV. Photography (J. M. Eder and E. Valenta).

**MINERAL RESOURCES OF THE UNITED STATES, CALENDAR YEAR 1893.** DAVID T. DAY, Chief of Division of Mining Statistics and Technology. Washington: Government Printing Office. 1894. 50 cents.

LARGE 8vo volume, strongly bound in black cloth. It contains a Table of Contents, Letter of Transmittal, Introduction, and 794 pages of subject-matter, concluded by an alphabetical index.

This well-classified report of the Mineral Resources of the United States is itself a mine of valuable information, and deals with the following minerals, metals, &c., found and exploited in the United States of America:—

Iron, Gold and Silver, Copper, Lead, Zinc, Quicksilver, Manganese, Aluminium, Bauxite, Nickel and Cobalt, Tin, Antimony, Coal, Pennsylvania Anthracite (Coke), Petroleum, Natural Gas, Stone, Clays, Cements, Soapstone, Asphaltum, Abrasive Materials, Precious Stones, Fertilisers, Gypsum, Salt, Natural Sodium Salts, Sulphur and Pyrites, Fluorspar, Mica, Asbestos, Mineral Paints, Graphite, Barytes, and Mineral Waters.

## Trade Report.

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

#### CANADIAN TARIFF CHANGES—concluded.

##### Free List.

Boric acid and borax in bulk of not less than 25 lb., bromine, celluloid, chloride of lime, cinnabar, cobalt ore, cochineal, colours (metallie), copper precipitate, cyanide of potassium, drugs (crude), dyeing or tanning articles (crude), fertilisers (unmanufactured), gums, gutta percha (crude), iodine (crude), iron liquor, lead nitrate and acetate (unground), lime juice (crude), litharge, oxide of manganese, nickel, oils, oxalic acid, phosphorus, platinum, plumbago crucibles, potash, chlorate, bichromate, prussiate, and caustic in bulk; potassium chloride, quicksilver, quinine, red liquor (aluminium acetate), rosin and rosin oil, rubber (unmanufactured), salt, soda and its salts, sulphates of iron and copper, tartar emetic, turpentine (crude), ultramarine blue, varnish for ships' use.—*Board of Trade Journal.*

## THE NETHERLANDS.

*Exemption of Naphthalene from Import Duty.*

Mr. C. L. des Graz, Her Majesty's Chargé d'Affaires at The Hague, in a despatch to the Foreign Office, dated the 27th July, transmits copy and translation of a decision (*resolatie*), dated July 21st, emanating from the Minister of Finance, and published in the *Staatscourant*, to the effect that naphthalene, purified or unpurified, in powder, crystals, or irregular pieces, is free of import duty.—*Ibid.*

## THE UNITED STATES TARIFF.

We give below as nearly as can be at present ascertained the duties on drugs, chemicals, and other goods dealt in by druggists as fixed by the tariff bill which has just been adopted by the Conference between the Senate and the Congress in Washington, and will become law on August 25, or earlier if the bill is signed by the President. We quote these from the bill as it left the Senate. There may have been some modifications made since, but substantially we believe the table as we give it to be correct.

	Present Duty.	Future Duty.
<i>Drugs and Chemicals.</i>		
Acetic or pyroligneous acid .....	1½ c. to 4 c. per lb.	20 per cent.
Boracic acid .....	5 c. per lb.	2 c. per lb.
Chromic acid .....	6 c. "	4 c. "
Citric acid .....	10 c. "	25 per cent.
Tannic acid or tannin .....	75 c. "	60 c. per lb.
Tartaric acid .....	10 c. "	20 per cent.
Alcoholic perfumery, including Cologne water and other toilet waters, and alcoholic compounds not specially provided for .....	\$2 per gall. and 50 per cent. to \$2 per gall. and 25 per cent.	\$2 per gall. and 50 per cent.
Alumina, alum, alum cake, patent alum, sulphate of alumina and aluminous cake, and alum in crystals or ground .....	½ c. per lb.	½ c. per lb.
Ammonia, carbonate of, marlate of, or sal ammoniac, sulphate of .....	½ c. to 1½ c. per lb.	20 and 10 and 20 per cent.
Blackening of all kinds .....	25 per cent.	20 per cent.
Borax, crude, or borate of soda, or borate of lime, refined borax .....	3 c. to 5 c. per lb.	1 c. to 2 c. per lb.
Camphor, refined .....	4 c. per lb.	10 per cent.
Chalk, prepared, precipitated, French, red, and all other chalk preparations not specially provided for .....	20 per cent.	20 "
Chloral hydrate .....	50 c. per lb.	25 "
Chloroform .....	25 c. "	25 c. per lb.
All coal tar colours or dyes, by whatever name known, and not specially provided for .....	30 per cent.	25 per cent.
Cobalt, oxide of .....	30 c. per lb.	25 c. per lb.
Collodion and all compounds of pyroxylin .....	60 c. per lb. to 25 per cent.	40 c. and 50 c. per lb. to 45 per cent.
Quicksilver .....	10 c. per lb.	7 c. per lb.
Colouring for liquors .....	50 per cent.	50 per cent.
Drugs, such as barks, beans, berries, balsams, bulbs, bulbous roots, exscrecences, fruits, flowers, dried fibres, dried insects, grains, gums and gum resin, herbs, leaves, lichens, mosses, nuts, roots and stems, spices, vegetables, seeds aromatic (not garden seeds), seeds of morbid growth, weeds, and woods used expressly for dyeing; any of the foregoing drugs which are not edible, advanced in value or condition by re-refining or grinding .....	10 "	10 "
Extracts and decoctions of log-wood and other dyewoods, extract of sumac .....	½ c. per lb.	10 "
Extract of hemlock-bark .....	½ c. "	10 "

## THE UNITED STATES TARIFF—continued.

	Present Duty.	Future Duty.
Sulphuric ether .....	40 c. per lb.	40 c. per lb.
Spirit of nitrous ether .....	25 c. "	25 c. "
Fruit ethers or essences .....	\$2.50 "	\$2.50 "
Ethers not provided for .....	\$1.00 "	\$1.00 "
Gelatin, glue, isoglass, or fish-gline, and fish-sandals .....	1 c. per lb. and 20 to 30 per cent.	25 c. per lb.
Glycerine, crude and refined .....	1½ c. to 4 c. per lb.	1 c. to 1½ c. per lb.
Ink, ink-powders, and printers' inks .....	30 per cent.	25 per cent.
Iodoform .....	\$1.50 per lb.	\$1.50 per lb.
Lignin, extracts of, in paste, rolls, or other forms .....	50 c. "	50 c. "
Morphine and all salts thereof .....	50 c. per lb.	50 c. per lb.
Magnesia, carbonate of .....	1 c. per lb.	3 c. per lb.
Magnesia, calcined .....	8 c. "	7 c. "
Magnesia, sulphate of .....	3 c. "	3 c. "
Opium, aqueous extract of, for medicinal uses, and tincture of, as Laudanum .....	40 per cent.	20 per cent.
Opium, containing less than 9 per cent. of morphine, and opium prepared for smoking .....	\$12 per lb.	\$6 per lb.
<i>Medicinal Preparations.</i>		
All medicinal preparations of which alcohol is a component part, or in the preparation of which alcohol is used, not provided for in this Act .....	50 c. "	50 c. "
All medicinal preparations, not specially provided for in this Act .....	25 per cent.	25 per cent.
Alkalis, alkaloids, distilled oils, essential oils, expressed oils, rendered oils, and all combinations not specially provided for .....	25 "	25 "
Cosmetics, dentifrices, pastes, pomades, powders, and all toilet preparations and articles of perfumery not specially provided for .....	50 "	40 "
Santonine and all salts thereof containing 80 per cent. or over of santonine .....	\$2.50 per lb.	\$1.00 per lb.
Phosphorus .....	20 c. "	25 c. "
<i>P. Fish.</i>		
Bichromate and chromate of .....	3 c. "	25 per cent.
Hydriodate, iodide, and iodate of .....	50 c. "	25 c. per lb.
Nitrate of, or saltpetre, refined .....	1 c. "	1 c. "
Prussiate of, red or yellow .....	5 c. to 10 c. per lb.	25 per cent.
<i>Oils.</i>		
Alizarin assistant, or Turkey oil .....	40 c. to 80 c. per gall. to 30 per cent.	30 "
Castor oil .....	80 c. per gall.	35 c. per gall.
Cod-liver oil .....	50 c. "	20 per cent.
Linseed and poppy-seed oil, raw, boiled, or oxidised .....	32 c. "	20 c. per gall.
Fusel oil, or amyl alcohol .....	10 per cent.	10 per cent.
Hemp-seed oil and rape-seed oil .....	10 c. per gall.	10 c. per gall.
Olive oil, fit for salad purposes .....	35 c. "	35 c. "
Peppermint oil .....	80 c. "	25 per cent.
Seal, herring, whale, and other fish oil .....	8 c. "	25 "
<i>Paints, Colours, and Varnishes.</i>		
Baryta, sulphate of, or barytes, manufactured .....	\$1.12 to \$6.72 per ton.	\$3.00 per ton.
Blues, such as Berlin, Prussian, Chinese .....	6 c. per lb.	6 c. per lb.

## THE UNITED STATES TARIFF—continued.

	Present Duty.	Future Duty.
Blue fixe, or artificial sulphate of barytes, and satin white, or artificial sulphate of lime.	1 c. per lb.	25 per cent.
Black, made from bone, ivory, or vegetable.	25 per cent.	20 "
Paris green and London purple.	"	12½ "
Chrome yellow, chrome green, and other chromium colours.	4½ c. per lb.	3 c. per lb.
Olive and ochre earths, sienna, and sienna earths, amber and amber earths, ground in oil.	1 c. to 1½ c. per lb.	1½ c. "
Ultramarine blue, whether dry, in pulp, or mixed with water, and wash-blue containing ultramarine.	4½ c. per lb.	3 c. "
Varnishes, including so-called gold-stone or japan, and on spirit varnishes for the alcohol contained therein.	35 per cent. to 35 per cent., and \$1.32 per gall.	25 per cent. to 25 per cent., and \$1.32 per gall.
Vermilion red, and other colours containing quicksilver, dry or ground, in oil or water, and vermilion red not containing quicksilver, but made of lead or containing lead.	12 c. per lb.	20 per cent. to 6 c. per lb.
Milting and Paris white.	3 c. "	1 c. to 1½ c. per lb.
Zinc, oxide of, and white zinc, gold-stone, dry or ground, in oil.	1½ c. to 1½ c. per lb.	1 c. per lb.
All other paints, colours, and pigments, whether dry or mixed, or ground in water or oil, or other solutions, including tubes, lakes, crayons, smalts, and frostings, and not specially provided for.	25 per cent. to 30 per cent.	25 per cent.
<i>Liquid Products.</i>		
Acetate of lead.	2½ c. to 5½ c. per lb.	1½ c. to 2½ c. per lb.
Litharge.	3 c. per lb.	1½ c. per lb.
Nitrate of lead.	3 c. "	1½ c. "
Orange mineral.	3½ c. "	1½ c. "
Red lead.	3 c. "	1½ c. "
White lead, and white paint and pigment containing lead, dry or in pulp, or ground or mixed with oil.	3 c. "	1½ c. "
<i>Salts, &amp;c.</i>		
Bicarbonate of soda.	1 c. "	1 c. "
Hydiate of, or caustic soda.	1 c. "	1 c. "
Bichromate and chromate of.	3 c. "	25 per cent.
Sulphate or soda ash.	1 c. "	1 c. to 1½ c. per lb.
Silicate of soda.	1 c. "	1 c. per lb.
Sponges.	20 per cent.	10 per cent.
Straw, and all sorts of reed.	40 c. per oz.	30 c. per oz.
Sulphur, sublimed, refined, or flowers of.	\$8 to \$10 per ton.	20 per cent.
Sesame, ground.	15 c. per lb.	10 "
Tartar, cream of, and patent tartar.	6 c. "	20 "
Tartar and lees crystals, partly refined.	4 c. "	20 "
Reichell salts.	3 c. "	2 c. per lb.
<i>Soap.</i>		
Castile.	1½ c. "	20 per cent.
Fancy perfumed.	15 c. "	35 "
Medicinal.	15 c. "	35 "
Not provided for.	20 per cent.	10 "
<i>Sugar.</i>		
Raw.	Free	40 per cent.
Refined.	1½ c. per lb., above No. 16	10 "
Sugar candy and all confectionery, made wholly or in part of sugar.	5 c. per lb.	35 per cent.

## THE UNITED STATES TARIFF—continued.

	Present Duty.	Future Duty.
Saccharine.	"	25 per cent.
Glucose, or grape sugar.	1 c. per lb.	15 "
<i>Seeds.</i>		
Castor beans or seeds.	50 c. per bush.	25 c. per bush.
Flaxseed or linseed, poppy-seed, and other oil seeds, not specially provided for.	30 c. "	20 c. "
Garden seeds, agricultural seeds, and other seeds, not specially provided for.	20 per cent.	10 per cent.
<i>Miscellaneous.</i>		
Chicory-root, burnt or roasted, ground or granulated.	2 c. per lb.	2 c. per lb.
Cocoa, prepared or manufactured: chocolate, sweetened.	2 c. "	2 c. "
Cocoa butter or cocoa butterine.	3½ c. "	3½ c. per lb.
Dandelion-root and acorns, prepared, and other articles used as coffee, or as substitutes.	1½ c. "	1½ c. "
Starch, including all preparations, from whatever substance produced.	2 c. "	1½ c. "
Dextrine, burnt starch, gum substitute, or British gum.	1½ c. "	1½ c. "
Mustard, ground or preserved, in bottles or otherwise.	10 c. "	25 per cent.
Spices, ground or powdered, not specially provided for: capsaicum or red pepper, unground, and sage.	4 c., 2½ c., to 3 c. per lb.	3 c., 2½ c., to 1 c. per lb.
Vinegar.	1½ c. per gall.	7½ c. per gall.
Malt extract, fluid, in casks, in bottles, or jars, solid or condensed.	20 c. to 40 c. per gall. and 10 per cent.	15 c. to 30 c. per gall. and 30 per cent.
Corks, wholly or partially manufactured.	10 c. to 15 c. per lb.	10 c. per lb.
Matches of all descriptions.	10 c. per gross to 1 c. per 1,000	20 per cent.

The free list is almost the same as heretofore as far as this class of goods is concerned. It comprises nearly all crude drugs not especially named in the list quoted above. Epsom salts and copperas (both hitherto charged with a duty of  $\frac{25}{100}$  c. per lb.) are to be free, and medicinal coal-tar preparations and products of coal-tar not colours or dyes and not specially provided, are also now admitted to the free list. Ginger ale, ginger beer, and all artificial mineral waters are now to be charged at 20 per cent. *ad val.* Hitherto 13 cents per dozen bottles (if not exceeding  $\frac{1}{4}$  pint) was charged on ginger ale, ginger beer, lemonade, soda-water, and similar waters. No additional duty is to be levied on bottles.—*Chemist and Druggist.*

## UNITED STATES.

## Customs Decision.

Binoxide of barium, otherwise known as peroxide of barium, is dutiable at 25 per cent. *ad val.* under the provisions of paragraph 76 N.T.

## GREAT BRITAIN.

## New Customs and Excise Regulations.

An official publication of great interest to the trading public has lately been issued by the Revenue Departments. This is the revised code of regulations governing the deposit of goods liable to duty in Customs or Excise warehouses. In order to prevent the large increase of price to the consumer which would ensue from the payment of a duty at once upon goods liable thereto, such goods, on importation or manufacture, can be deposited in officially approved warehouses, and can remain there until the dealer desires to clear them for home consumption upon payment of duty. The code governing both Revenue Departments, which was first issued in 1880, has just been revised, and all the important alterations which have since taken place are now gathered up in one publication.—*Chemist and Druggist.*

## GENERAL TRADE NOTES.

## SALT DISCOVERY IN TURKESTAN.

According to the *Gazette du Turkestan* a commencement has been made with the working of a rich deposit of salt which has been discovered at a distance of 15 verstes from the eastern side of Lake Kang-Koul, not far from the road going by the heights of Touzgoun and Kizyl-Djik to Mouz-Kol. At the present time the salt is extracted at two places, situated at a distance of from 20 to 25 sagues from each other. At these places the salt appears at the surface of the soil. Borings have shown that the places where the salt appears at the surface form the meeting place of two beds of salt following opposite directions. The bed of salt is two or three archines in thickness.

## BOTANICAL EXPERIMENTS IN INDIA.

The *Calcutta Englishman*, in its issue for the 26th June, has the following note:—

In the Royal Botanic Gardens further experiments were carried on last year in the cultivation of plants of economic value. Reviewing the work of the 12 months, Dr. King says that recha plants still continue to be in occasional demand, and supplies have been issued gratuitously to various applicants. Seeds of coca—the source of the anæsthetic known as cocaine—have also been freely given to various tea-planters, but Dr. King is apparently by no means hopeful regarding the cultivation of this plant. In his opinion, the alkaloid can, from its nature, never come into very extensive use, and the demand for it is at present greatly below the supply. Planters have, however, been tempted by the high prices obtained for batches of coca leaves during recent years, and, in Dr. King's words, they have been led into taking up the cultivation "with more enthusiasm than discretion." Among other economic products, *Cola acuminata*, which produces the kola-nut, was also in demand to some extent, and a few applicants outside of India were supplied with the seed of the bhabar or babni grass. The history of this plant, as one of the principal materials used in the Indian paper-making industry, is given in brief in Dr. King's report. It appears that it first attracted his attention as a possible raw material for paper 25 years ago in the North-West Provinces.

In 1873 he sent home samples to a paper-maker in Scotland, who reported very favourably upon them, and a few years later Mr. Routledge, then a leading authority on paper, to whom specimens were submitted through the India Office, found them to be little inferior to esparto as a raw material. Mr. J. S. Gamble, of the Forest Department, subsequently discovered that this grass is common in the forests of Chota Nagpur, and samples were sent to the Bally Mill, then the only paper-making factory near Calcutta.

The qualities of the new grass were found to be so excellent that it was immediately adopted, and it has since become one of the most important raw materials of the industry. The inquiries that have been received of late from other countries appear to show that India will soon lose her present monopoly in this product. Dr. King points out, however, that for paper-making purposes the Japanese paper mulberry tree yields a fibre far superior to bhabar. His efforts to extend the cultivation of this tree in Bengal, where it can be readily grown, have not hitherto been successful; but the qualities which Dr. King claims on its behalf should ensure its eventual adoption. For the information of those who care to make the experiment, it may be noted that the Botanic Garden has still a small stock ready for issue to any applicant who may be forthcoming. There is obviously much room for improvement in Indian paper manufacture, and the introduction of the Japan paper mulberry tree may possibly enable the mills in the country to approximate somewhat more closely to home manufactures in quality as well as in price.

## LIME-CULTIVATION IN THE WEST INDIES.

There is an interesting note on the West Indian lime in a recent number of the *Kew Bulletin*. The sour lime-tree

(*Citrus medica*, L., var. *acida*, Brandis), which yields the lime-juice and essential oil of lime familiar in the drug trade, was probably introduced into the West Indian islands from the East. Lime-juice is obtained by compressing the fresh ripe fruits between heavy rollers. It is exported in the raw state or concentrated. The concentrated variety is obtained by evaporating the raw juice in copper or enamelled-iron pans until it is reduced to about one-eighth or one-tenth of the original bulk. When exported it is a dark, viscid fluid of the consistence of treacle. From the rind of the fresh fruits there is obtained, by the candelling hand process, a fine essence of limes exported in copper vessels. The process, which is a slow one, is performed by women and girls. The task per day is measured in fluid ounces. By distilling the raw lime-juice a spirit is obtained known as oil of limes. The essential oil of limes extracted by hand is far more valuable than the oil of limes, as the perfume of the latter is injuriously affected by the heat necessary in distillation.

Lime-trees were abundant in Dominica as long ago as 1791, but at that period they appear to have been grown only for their fruit. The lime-juice industry appears to have been started in Dominica by the late Dr. Murray, but shortly after the commencement of that gentleman's venture the Messrs. Sturge, of Birmingham, quite independently of Dr. Murray, and without any knowledge of his experiments, set to work to establish similar plantations in the neighbouring island of Montserrat. The industry appears to hold on prospects of being a remunerative one, for, according to a Dominica planter, it takes about 1,000*l.* to purchase about 20 acres of land and plant them with lime-trees, to build a house for the manager, to erect a mill with copper boilers, to concentrate the juice, to pay for superintendence, and cover all expenses for seven years. At the end of this time the estate would yield 10 hogsheads of concentrated lime-juice a year, worth 480*l.* The yearly cost of cultivation and manufacture would amount to 240*l.*, the rest being gross profit.—*Chemist and Druggist*.

## THE MINERAL INDUSTRIES OF SOUTH RUSSIA.

The official *Messenger of Finances* gives the following account of the mining and metallurgical industries of the south of Russia during the year 1893. The region under consideration comprises the following private works and mines: Eighteen iron mines in the government of Volynia and in the neighbourhood of the villages of Kriviy Rog, one lignite mine in the government of Kieff; five blast-furnace companies, namely, the Denesheffsky, Vugodensky, and Emelchinsky in the government of Volynia, and the Glantselsky Company in the government of Cherson; the Alexandroffsky Iron and Steel Works in the town of Ekaterinoslav, the Briansk and Dnieper Iron and Steel Works in the village of Kamensk, the Iron Works of the South Russian Dnieper Metallurgical Company, also near Ekaterinoslav, and also the Novorossisk Iron and Steel Works at Hughesoffa. The district also includes five manganese ore mines in the government of Ekaterinoslav, 27 coal mines in the neighbourhood of Bachmout, and Karakonsky and Stillsky iron mines in the region of Mariopol, the Auerbach mercury mines and works at Nikitoffka, and 60 coal mines in the neighbourhood of Lugansk. The output of these works and mines during 1893 and 1892 was as follows, in metric tons:—

	1892.	1893.	Increase.
Coal.....	2,025,200	2,159,350	134,150
Lignite.....	2,000	7,618	5,618
Iron ore.....	518,204	625,615	107,411
Manganese ore.....	28,945	70,450	41,505

The quick-silver mines owned by Messrs. Auerbach produced 61,200 tons of ore, from which 443,169 lb. of

mercury were obtained. The iron and steel works report the following output, in metric tons:—

	1892.	1893.	Changes.
Pig-iron .....	267,582	319,176	Inc. 51,644
Manufacture Iron .....	60,349	82,540	Dec. 27,800
Steel rails .....	110,900	133,774	Inc. 22,874
Other steel .....	29,350	27,360	Dec. 1,990

In the Donetz basin the coal industry continues to develop, but in 1893 the local demand for coal considerably exceeded the supply; this is partially explained by the abundant harvest of that year having claimed the coal miners, who are really peasant proprietors, for a longer period than usual, and also by the ravages caused by the cholera. The dearth of coal somewhat impeded production at the metallurgical works of the district.

The rapid development of the iron and steel industries of the south has evoked a corresponding growth in the production of iron ores. The exploitation of the manganese mines also shows remarkable progress. Notwithstanding the temporary check caused by the dearth of coal, the total production of pig-iron in the south of Russia shows an increase of 18 per cent., and of rails 20 per cent. over that of the preceding year.

In April 1893 the Donetz Iron and Steel Company started building new works, and at the close of the year had erected one blast-furnace with a production of 10,000 pounds (163 tons) of pig per day. The furnace is 65½ ft. high, and is furnished with four Cowper hot-blast stores. Attached to these works is a Schuterman and Cramer coal-washing plant capable of dealing with 325 tons of coal per day, and a set of 12 Bernard coke-ovens with a capacity of 81 tons of coke per day. The Alexandroffsky works have also erected a new blast-furnace, No. 4, with three Whitwell stoves, and a set of 62 Coppee coking-ovens, besides which they have supplemented their Bessemer and rolling plant with hydraulic machinery, and lastly, both the Dnieper and Novorossisk have added a fourth blast-furnace to their plant.

In 1893, 36,872 men were employed in the mining and metallurgical industries of the south of Russia. This total was distributed as follows:—13,322 in coal mines, 1,718 in iron mines, 895 in manganese mines, 298 in mercury mines, 12,618 at iron and steel works, 236 at mercury works, and 7,785 in quarries. There were 206 accidents, 80 of which were fatal.—*Engineering and Mining Journal*.

#### NATURAL MINERAL WATERS.

The Academy of Medicine has unanimously adopted the report of the Committee on Mineral Waters, presented by M. Albert Robin. There were only two members present. The recommendations are:—1. The sale of mineral waters which have been decanted or artificially rendered gaseous should not be allowed by the authorities. 2. Every application to the Ministry for authorisation should be accompanied by the certificate of a competent official that the water has not thus been already manipulated, and should embody the promise of the proprietor not to resort to these processes. 3. Reservoirs should be hermetically closed, and should be emptied at least every twenty-four hours. They should be so placed that the spring flows direct into the reservoir. Bottles should be thoroughly sterilised, and all causes of pollution during bottling carefully avoided. 4. Notice of these new arrangements should be sent to the proprietors of all springs already authorised, and three months should be given them to conform therewith.—*Chemist and Druggist*.

#### THE EXTRACTION OF NICOTINE FROM TOBACCO-LEAF.

The Government Tobacco Monopoly are giving this subject attention, and they claim to have succeeded in obtaining a product of high concentration and uniform quality. They think there will be a large demand for it for the destruction of insects in agriculture and horticulture, and also from South America and other sheep-raising countries as a

remedy for scab. Several millions of pounds of nicotine are annually sold in Uruguay and the Argentine Republic, but the quality is very variable. M. Frayssé, a Buenos Ayres merchant, after making two journeys in the countries in question, with a view to thoroughly studying the matter, has just been appointed special agent of the French Government for the sale of the product at Rio de la Plata. The Government process has been devised by M. Schlossing.—*Ibid*.

#### CHEMICAL CONGRESS.

It is announced that the International Congress of Applied Chemistry, which has just terminated its session for 1894 at Brussels and Antwerp, will hold its next meeting at Paris in two years time. The Association of French Chemists of Sugar Refineries and Distilleries is to be charged with the organisation of the congress in 1896. At the one just terminated M. F. Dupont, a French delegate, brought forward a proposition for the adoption of the French metric litre, to the exclusion of the Mohr or German litre, as a base for gauging and for the graduation of chemical instruments, which was unanimously carried. The French Centigrade thermometer was also adopted by the delegates in preference to the Réaumur or Fahrenheit thermometers.—*Ibid*.

#### THE ANNUAL REPORT (EIGHTEENTH) OF H.M. INSPECTORS OF EXPLOSIVES FOR 1893.

See under XXII., page 837.

NOTE.—With reference to the letter from the Home Office, published in the June No. of this Journal, p. 674, it will be seen from the above report that the authorities have reconsidered their determination to treat collodion cotton as an explosive.

#### THE COKE PROBLEM IN ENGLAND AND AMERICA.

Mr. Jos. D. Weeks, of Pittsburgh, one of the leading authorities in the United States on natural gas, petroleum, and coke, was recently commissioned by the American Geological Survey to make a report in his official capacity as the expert on coke of the United States Census. He was accompanied by Dr. F. L. Slocum, Pittsburgh, a well-known chemist and gas-train expert; Mr. J. L. Gardener, gas-expert; and Mr. Wm. Stanton, firebrick manufacturer. The by-product coke-works in this country, which have been visited, have included, among others, the following:—The West Crook works of Pease and Partners; the works and furnaces of Sir B. Samuelson, M.P.; the works of the Durham Coke and By-products Company, Lanchester; the works of the Brymbo Steel Company; and the works of Brunner, Mond, and Co. (Limited), at Northwich, near Liverpool, where the Smet-Solvay ovens are erected. The Belgian by-product works have included the Havrèville and Bois du Lac collieries, belonging to the leading coal company of Belgium, and possessing the deepest pits in the world (over 1,000 yards); and the Seraing Collieries of the celebrated John Cockerill Company. In Westphalia the concerns were the great Krupp works at Essen; the Constantin collieries, No. 11; the Germania collieries; the Julia collieries; and the Recklinhausen, No. 11, collieries. At all these the Otto-Hoffmann ovens are in use. Admission was also obtained to the Phoenix Coke-works at Ruhrort, the Hussener works at Gelsenkirchen, the Shamroek works near Bochum, and the Osterman works at Bochum—at the two latter of which beehive ovens are still in existence. On the question of yield and of by-product recovery, Mr. Weeks will report that the yield of coke is much larger by the close than by the open method of manufacture, and that the proportion of by-products recovered is about as follows:—Sulphate of ammonia, rather over 1 per cent. in weight of the coal charged; tar, 2½ to 3½ per cent.; and benzole, from ½ to 1 per cent., or slightly more. These by-products yield, after paying the expenses of condensation plant, a profit of from 2s. to 3s. and on to 4s. per ton of coal consumed. Regarding the alleged deterioration in coke for blast-furnace purposes, which has been subjected to the recovery processes compared with that made upon the beehive-oven principle, Mr. Weeks' report is likely to be definite. Although at first, in both Belgium and Germany, a prejudice existed among certain of the pig-iron making firms in this direction, no such prejudice exists now. The excellence to which the

close processes have been brought seems to have completely settled this question. The ironmasters of both countries are now convinced that the weight-bearing and heat-giving qualities of the coke are not deteriorated by taking out the ammonia, tar, and other waste products, and the retort fuel is regarded as in all respects as good for ironworks' uses as the beehive product. The almost complete manner in which the retort has superseded the beehive oven in Belgium and Westphalia—two of the leading ironmaking countries of the world—is, indeed, in itself, evidence sufficient on this point. After a most painstaking enquiry, in the course of which they have accumulated a vast amount of information, the party have come to the conclusion that there is nothing to prevent, but everything to recommend, the substitution of the retort and by-product oven for the beehive oven now almost exclusively used in America. Care is, however,

needed in building the ovens to proportion, their design and parts to the exact character of the coal to be used. For example, most of the coking coals of the great Connellsville and some other of the coking districts of America are superior in volatile matter to the European fuels, and the same applies to the coking coals of this country. Other points of difference might also be mentioned, which will have to be taken into consideration by American builders of the new retorts. A necessity also exists at the by-product oven for a more careful and intelligent management than is required in the beehive. It is declared to be almost impossible with the beehive oven to make bad coke from good American coal, and little supervision in the actual carbonising of the coal is required. In the case of the retort or by-product ovens, however, increased supervision must be exercised by the responsible oven-manager.—*Ironmonger.*

ANALYSIS OF EXPORTS OF PRINCIPAL BRITISH CHEMICAL MANUFACTURES FOR THE YEAR 1893 TO BRITISH COLONIES.

Colonies.	Alkali.	Bleach.	Cement.	Chemicals.	Coal Products.	CuSO <sub>4</sub> .	Dye-stuffs.	Manure, (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> .
	£	£	£	£	£	£	£	£
Channel Islands .....	..	..	8,119	3,337	..	..	..	..
Malta .....	2,093	..	2,110	..	..	..	..	..
South Africa .....	10,786	..	32,284	10,117	11,311	..	..	..
India:								
Bombay .....	6,013	1,526	12,132	67,156	8,061	..	6,897	..
Bengal and Burmah .....	32,958	15,038	21,106	31,817	57,964	2,017	2,498	..
Other possessions .....	2,152	192	27,821	13,871	1,259	..	56	..
Australia:								
South .....	3,911	2,716	1,106	13,586	15,595	..	1,301	..
Victoria .....	17,330		5,610	42,290		1,281		..
New South Wales .....	18,165		28,745	59,554		..		..
Queensland .....	4,127		2,872	13,937		..		..
New Zealand .....	6,291		13,120	52,160		1,215		..
Other colonies .....	698		3,661	4,618		1,168		..
British North America .....	63,705	12,924	53,250	38,218	5,575	..	2,302	..
West Indies .....	..	..	28,371	11,056	..	..	..	119,089
Other possessions .....	5,015	983	1,571	7,729	7,599	1,532	75	9,877
								Mauritius, 13,050

Colonies.	Manures, Other.	Medicines.	Paints.	Plated Ware.	Salt-petre.	Soap.	Sugar, Refined.	Tracle and Glucose.
	£	£	£	£	£	£	£	£
Channel Islands .....	40,409	7,444	10,076	1,004	..	8,288	1,576	..
Malta .....	..	5,639	4,102	..	..	3,286	29,711	..
South Africa .....	8,920	86,680	63,788	27,328	..	115,458	2,563	16,586
India:								
Bombay .....	..	69,660	70,970	11,300	..	53,298	1,585	..
Bengal and Burmah .....	Mauritius, 18,391	80,301	59,450	6,867	..	21,104	..	..
Other possessions .....	..	38,182	35,869	6,402	..	25,085	2,299	..
Australia:								
South .....	..	18,587	13,211	4,116	23,827	..	..	2,122
Victoria .....	..	47,181	40,832	16,762		12,727	..	
New South Wales .....	..	62,225	75,550	11,874		16,622	..	
Queensland .....	..	20,516	15,189	3,277		..	..	
New Zealand .....	7,033	45,092	51,592	13,111		8,578	..	
Other colonies .....	2,874	8,222	6,664	2,093	..	2,731	..	..
British North America .....	..	31,620	68,181	6,354	..	20,506	39,220	..
West Indies .....	94,137	49,189	23,908	3,187	..	85,665	1,274	..
Other possessions .....	2,975	30,502	12,090	3,122	3,801	51,059	21,275	1,373

## ANALYSIS OF EXPORTS OF PRINCIPAL BRITISH CHEMICAL MANUFACTURES FOR THE YEAR 1893 TO FOREIGN COUNTRIES.

Countries.	Alkali.	Bleach.	Cement.	Chemicals.	Coal Products.	CuSO <sub>4</sub> .	Dyestuffs.	Manure. (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> .
	£	£	£	£	£	£	£	£
Russia.....	122,062	55,230	9,697	126,688	48,996	10,346	22,548	12,797
Sweden and Norway.....	32,626	16,184	2,092	92,530	45,172	..	..	..
Denmark.....	15,569	5,447	..	17,087	8,990	..	..	..
Germany.....	49,133	30,396	2,365	236,258	141,472	3,737	22,897	401,038
Holland.....	21,976	13,379	21,024	138,078	233,640	1,348	17,967	112,321
Belgium.....	19,920	26,736	1,654	88,141	152,197	4,128	7,915	208,650
France.....	19,304	4,695	6,269	171,200	277,156	243,171	14,736	124,284
Portugal.....	14,844	4,273	5,119	15,522	..	17,908	1,567	..
Spain.....	103,092	6,796	6,771	43,465	49,106	4,030	5,109	184,799
Italy.....	74,128	15,583	..	48,591	79,458	104,926	7,681	15,245
Austria.....	9,887	..	..	7,295	23,410	6,300	..	{ Java } 88,617
Greece.....	8,021	..	..	2,669	..	..	..	..
Romania.....	8,344	..	4,791	4,211	..	..	..	..
Turkey.....	14,798	..	{ China } 3,485	12,880	..	..	{ China } 5,373	{ Foreign } West Indies 11,917
Japan.....	45,473	..	11,427	123,743	7,348	..	1,567	..
United States, E.....	977,224	328,221	178,160	349,894	58,218	..	80,390	58,957
United States, W.....	39,945	5,920	134,106	4,094	..	..	..	..
Chili.....	5,927	..	10,562	11,582	..	2,192	..	..
Brazil.....	30,062	..	29,783	11,021	5,719	..	..	..
Argentina.....	27,961	..	32,026	80,508	5,203	2,750	..	..

Countries.	Other Manures.	Medicines.	Prints and Colours.	Plated Wares.	Saltpetre.	Soap.	Sugar, Refined.	Treacle and Glucose.
	£	£	£	£	£	£	£	£
Russia.....	79,530	4,622	46,879	1,473	..	..	..	..
Sweden and Norway.....	61,954	..	21,395	4,233	..	3,558	80,782	87,921
Denmark.....	30,852	..	10,832	5,939	..	1,078	143,082	7,470
Germany.....	97,042	29,793	86,296	17,062	..	7,934	5,250	2,759
Holland.....	12,365	4,539	51,372	3,183	..	9,298	79,200	11,136
Belgium.....	6,966	13,217	62,324	4,607	..	14,295	17,491	16,912
France.....	187,525	20,794	93,624	17,614	..	14,798	9,404	19,909
Portugal.....	6,111	6,667	29,437	1,906	5,412	1,182	78,723	..
Spain.....	139,078	10,740	29,840	4,948	1,500	14,736	..	..
Italy.....	58,303	7,693	33,620	2,221	..	3,691	44,171	..
Austria.....	..	..	4,781	2,616	..	..	..	..
Greece.....	..	..	..	..	1,512	{ China } 65,437	..	..
Romania.....	..	{ Foreign } West Indies 61,292	17,863	6,076	..	..	..	..
Turkey.....	..	6,552	10,502	1,972	..	1,671	..	..
Japan.....	..	7,567	39,656	..	..	..	..	..
United States, E.....	21,929	37,726	126,177	10,376	1,516	51,375	219,827	..
United States, W.....	..	..	5,462	..	..	..	{ Uruguay } 110,074	..
Chili.....	..	13,202	25,329	4,079	..	5,968	..	..
Brazil.....	..	32,251	40,295	2,546	17,606	4,745	..	..
Argentina.....	..	18,544	52,790	4,907	..	3,194	8,938	..

—Ibid.

## ANALYSIS OF VALUE OF IMPORTS OF MANUFACTURES OF CHEMICAL INTEREST DURING 1893.

Article.	Germany.	Holland.	Belgium.	France.	United States of America.	Remarks.
	£	£	£	£	£	
Alkali .....	23,934	5,876	508	37,890	2,246	
Ammunition: Gunpowder .....	9,934	784	2,872	..	..	
„ Other explosives .....	33,650	20,124	6,727	21,732	7,902	
Brass and bronze manufactures .....	19,587	21,407	9,962	52,452	6,500	
Candles .....	7,141	23,843	25,474	..	..	
Caoutchouc manufactures .....	167,389	42,625	30,844	61,379	63,450	
Chemicals .....	332,966	260,100	56,327	296,664	59,695	Spain, 49,050 <i>l</i> ; Italy, 1,000 <i>l</i>
China .....	250,063	201,867	31,288	93,838	..	Japan, 37,498 <i>l</i>
Copper manufactures .....	8,620	37,557	9,462	14,013	2,141	
Drugs .....	95,974	52,118	21,753	54,957	144,745	Mauritius 23,141 <i>l</i> ; Ceylon, 25,715 <i>l</i> ; Austria, 24,646 <i>l</i> ; Japan, 51,467 <i>l</i> ; Peru, 42,695 <i>l</i> ; Bombay, 12,521 <i>l</i> ; Hong Kong, 55,166 <i>l</i>
Dyes: Aniline .....	4,980	165,384	4,558	12,082	..	
„ Alizarine .....	..	293,845	..	..	..	
„ Other .....	2,139	13,692	..	3,339	..	Spain, 2,716 <i>l</i>
Dye extracts .....	5,225	975	21,078	226,528	15,685	Austria, 16,957 <i>l</i> ; Canada, 9,530 <i>l</i>
Glass: Window .....	47,118	166,153	288,136	1,476	..	
„ Plate, rough .....	6,702	565	38,137	1,961	3	
„ „ other .....	11,749	3,407	228,483	2,029	..	Italy, 655 <i>l</i>
„ Manufactures .....	656,995	267,328	418,916	215,317	8,184	Sweden, 77,601 <i>l</i>
Iron and steel: Steel, unwrought .....	4,472	4,107	12,714	..	..	Sweden, 63,755 <i>l</i>
„ Iron girders .....	..	136,019	283,205	..	..	
„ Iron manufactures .....	353,369	931,043	423,119	120,915	349,442	Norway, 62,998 <i>l</i> ; Sweden, 268,259 <i>l</i> ; Canada, 6,880 <i>l</i>
Leather: Dressed .....	234,850	311,473	59,417	683,055	1,481,583	Russia, 24,963 <i>l</i> ; Canada, 28,574 <i>l</i>
„ Enamelled .....	..	47,274	..	8,393	39,445	
Matches .....	10,253	33,201	65,932	..	..	Sweden, 20,960 <i>l</i> ; Norway, 47,935 <i>l</i>
Methylic alcohol (not dutiable) .....	15,694	2,603	4,549	..	16,748	
Oil: Seed .....	185,767	59,495	167,083	172,750	89,850	
„ Chem., essential, perfumed .....	10,878	7,690	6,819	57,112	31,209	Ceylon, 22,788 <i>l</i> ; Italy, 101,951 <i>l</i> ; Hong Kong, 7,041 <i>l</i>
Painters' colours .....	216,948	436,230	210,416	61,281	43,891	
Paper: Printing and writing .....	77,060	73,044	87,748	19,408	9,111	Austria, 17,584 <i>l</i> ; Sweden, 32,239 <i>l</i> ; Norway, 41,192 <i>l</i>
„ Board .....	71,400	313,259	9,741	14,221	..	Sweden, 16,825 <i>l</i>
„ Wood pulp .....	199,235	63,526	..	..	15,956	Canada, 4,170 <i>l</i> ; Sweden, 329,199 <i>l</i> ; Norway, 538,260 <i>l</i>
Saltpetre .....	70,112	21,533	13,165	184	..	Benzal, 195,450 <i>l</i>
Sugar: Refined .....	5,993,904	1,671,195	474,833	2,331,774	27,965	Austria, 81,780 <i>l</i>
„ Unrefined beetroot .....	4,592,558	118,527	742,320	610,561	..	Austria, 145,889 <i>l</i>
„ Glucose .....	43,421	2,200	..	7,728	640,390	
Varnish (not containing alcohol) .....	3,670	..	2,062	1,783	21,651	
Vinegar .....	..	5,323	4,551	3,100	..	
Yeast .....	118,925	258,304	..	187,271	..	
Zinc manufactures .....	104,957	203,658	72,320	10,406	..	

--Ibid.





ABSTRACT OF IMPORTS OF FOREIGN AND COLONIAL MERCHANDISE FOR THE YEARS 1892 AND 1893—*cont.*

Articles.		Quantities.		Value.	
		1892.	1893.	1892.	1893.
<i>Manure—continued.</i>				£	£
Phosphate of lime and rock.....	Tons.	314,130	323,527	655,880	591,467
Unenumerated .....	"	69,332	9,692	108,581	129,719
Margarine.....	Cwts.	1,705,350	2,099,970	3,712,884	3,655,344
Matches.....	Value £	..	..	597,654	592,599
<i>Metals:—</i>					
Copper ore and regulus.....	Tons.	229,087	166,008	3,877,479	3,318,962
„ unwrought.....	"	36,834	48,945	1,714,698	1,975,895
„ manufactures.....	Value £	..	..	15,325	72,719
Iron ore.....	Tons.	3,780,503	1,965,864	2,716,829	2,702,928
„ bars.....	"	75,915	5,820	692,250	593,663
Steel, unwrought.....	"	6,483	8,934	62,456	80,506
Iron and steel manufactures.....	Cwts.	2,875,567	997,567	2,532,118	2,545,659
Lead, pig and sheet.....	Tons.	182,782	188,219	1,979,436	1,855,255
Silver ore.....	Value £	..	..	3,632,169	3,066,131
Tin.....	Cwts.	589,365	671,162	2,743,814	2,892,107
Zinc—crude.....	Tons.	52,793	56,926	1,162,591	1,067,397
„ manufactures.....	Cwts.	379,161	368,843	163,089	336,353
Not otherwise enumerated.....	Tons.	2,230	2,670	284,317	391,563
Milk, condensed or preserved.....	Cwts.	481,374	501,005	199,288	1,669,755
Oil nuts and kernels.....	Tons.	55,492	69,689	676,653	849,357
<i>Oil:—</i>					
Train, blubber, or sperm.....	Tons.	21,121	19,939	415,181	389,861
Animal.....	Cwts.	65,057	49,953	119,149	12,160
Cocoa-nut.....	"	164,569	155,347	191,389	202,885
Olive.....	Tons.	20,476	17,269	762,516	652,041
Pal'm.....	Cwts.	1,068,589	1,169,944	1,169,499	1,458,642
Seed.....	Tons.	22,033	39,164	555,832	718,867
Turpentine.....	Cwts.	516,597	453,235	599,333	492,220
Chemical, essential, and perfumed.....	Lbs.	1,127,574	1,392,989	244,572	274,388
Oil seed cake.....	Tons.	311,872	283,542	2,147,699	1,369,774
Painter's colours and pigments.....	Value £	..	..	1,043,189	160,329
Paper and pasteboard (except hangings).....	Cwts.	2,886,672	2,915,646	2,372,558	2,313,312
Paper hangings.....	"	7,734	7,236	39,443	37,768
<i>Paper making materials:—</i>					
Rags.....	Tons.	23,632	20,759	211,065	199,155
Esparto.....	"	212,967	185,459	1,029,961	879,431
Pulp of wood.....	"	190,946	215,584	981,925	1,189,310
Paraffin.....	Cwts.	552,572	768,695	744,794	813,637
Petroleum.....	Gallons.	130,186,085	155,125,967	2,446,996	2,546,769
<i>Pitch and tar:—</i>					
Pitch.....	Cwts.	96,254	95,226	26,984	27,744
Tar.....	Barrels.	131,886	107,216	86,412	69,921
Pyrites of iron and copper.....	Tons.	694,411	612,818	1,955,503	1,655,497
Quicksilver.....	Lbs.	4,274,274	3,929,558	396,184	341,186
Rosin.....	Cwts.	1,681,393	1,570,173	984,959	894,252
Saltpetre.....	"	398,355	242,568	267,931	213,873
<i>Spirits:—</i>					
Unenumerated, unsweetened.....	Proof. Galls.	1,594,123	1,727,786	87,926	94,897
Sweetened and perfumed of all kinds.....	"	133,431	139,375	225,333	231,751

ABSTRACT OF IMPORTS OF FOREIGN AND COLONIAL MERCHANDISE FOR THE YEARS 1892 AND 1893—*cont.*

Articles.		Quantities.		Value.	
		1892.	1893.	1892.	1893.
Sugar:—				£	£
Refined and candy .....	Cwts.	10,620,671	11,550,540	9,061,948	10,603,604
Unrefined .....	"	16,295,947	16,032,113	10,706,909	11,459,344
Molasses .....	"	616,012	584,747	180,610	176,332
Glucose .....	"	918,570	1,235,800	566,433	694,502
Tallow and stearine .....	Lbs.	239,445,067	249,546,451	1,747,968	2,161,407
Wax .....	Cwts.	28,197	34,458	123,505	123,330
Yeast .....	"	235,020	221,403	611,141	573,905

—*Ibid.*

## ABSTRACT OF THE EXPORTS OF PRODUCE AND MANUFACTURES OF THE UNITED KINGDOM FOR YEARS 1892 AND 1893.

Articles.		Quantities.		Value.	
		1892.	1893.	1892.	1893.
Alkali .....	Cwts.	5,885,600	5,832,700	2,119,440	1,857,928
Ammunition:—					
Gunpowder .....	Lbs.	7,935,800	7,065,300	203,327	194,277
All other kinds .....	Value £	..	..	1,092,553	1,014,614
Candles .....	Lbs.	18,375,500	19,011,300	344,208	352,070
Caoutchouc, manufactures of .....	Value £	..	..	1,215,307	1,185,597
Cement .....	Tons.	492,615	437,565	902,910	744,424
Chemical products:—					
Dyestuffs .....	Value £	..	..	443,437	451,766
Sulphate of copper .....	"	..	..	297,807	419,363
Unenumerated .....	"	..	..	1,974,518	2,145,652
Coal products: naphtha and paraffin .....	"	..	..	1,332,273	1,275,062
Earthenware: china and bricks .....	"	..	..	2,257,598	2,174,224
Glass:—					
Plate .....	Square ft.	2,158,076	1,684,500	116,467	86,051
Flint .....	Cwts.	93,739	83,530	225,528	215,982
Common bottles .....	"	753,742	639,202	360,343	305,781
Unenumerated .....	"	222,088	194,316	182,967	158,126
Leather: unwrought .....	"	131,452	115,115	1,245,210	1,372,681
Lucifers and matches .....	Value £	..	..	124,425	97,136
Manure (including sulphate of ammonia) .....	Tons.	539,134	352,588	2,137,811	2,300,421
Medicines and drugs .....	Value £	1,013,231	945,483	1,013,231	945,483
Metals:—					
Iron and steel .....	Tons.	2,739,279	2,856,574	21,765,768	20,592,507
Copper, unwrought .....	Cwts.	845,822	566,884	2,061,444	1,367,292
Copper, wrought, yellow metal .....	"	294,582	290,987	729,068	682,174
Copper, wrought, other sorts .....	"	324,332	339,096	972,907	972,301
Brass .....	"	108,215	115,047	434,537	458,233
Lead .....	Tons.	58,162	48,871	708,510	552,601
Tin .....	Cwts.	112,989	134,775	544,067	606,399
Zinc .....	"	196,211	194,513	178,276	158,631
Oil, seed .....	Tons.	67,232	66,193	1,322,718	1,438,070
Oil and floorcloth .....	Sq. yards.	16,442,200	17,536,000	712,837	706,677
Painters' colours and materials .....	Value £	..	..	1,525,152	1,433,780

ABSTRACT OF THE EXPORTS OF PRODUCTS AND MANUFACTURES OF THE UNITED KINGDOM FOR YEARS  
1892 AND 1893—*cont.*

Articles.		Quantities.		Values.	
		1892.	1893.	1892.	1893.
Paper:—				£	£
Hanging....	Cwts.	58,978	70,954	148,480	149,965
Other sorts .....	"	873,033	852,801	1,151,204	1,333,375
Plated and gilt wares.....	Value £	..	..	274,016	255,735
Rags and other materials for paper .....	Tons.	57,648	72,219	107,889	361,542
Salt .....	"	654,136	636,135	539,262	504,509
Soap .....	Cwts.	510,799	605,083	58,114	614,257
Sugar, refined .....	"	902,327	1,122,252	681,273	902,394

—*Ibid.*

## BOARD OF TRADE RETURNS.

## SUMMARY OF IMPORTS.

Articles.	Month ending 31st July.	
	1893.	1894.
	£	£
Metals.....	1,732,540	1,452,151
Chemicals and dyestuffs.....	421,558	451,820
Oils.....	620,563	747,101
Raw materials for non-textile industries.	4,282,287	4,063,668
Total value of all imports .....	33,203,191	31,845,553

## SUMMARY OF EXPORTS.

Articles.	Month ending 31st July.	
	1893.	1894.
	£	£
Metals (other than machinery) ....	2,789,176	2,429,516
Chemicals and medicines .....	642,667	657,024
Miscellaneous articles.....	2,457,727	2,141,421
Total value of all exports.....	19,651,374	18,398,536

## IMPORTS OF OILS FOR MONTH ENDING 31ST JULY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Cocoa-nut..... Cwt.	11,109	34,581	14,801	41,075
Olive .....	760	2,964	29,076	99,257
Palm .....	104,461	108,400	124,533	114,203
Petroleum .....	11,533,882	13,888,220	195,485	245,605
Seed .....	2,023	2,187	51,138	45,861
Train, &c.....	2,356	3,426	43,683	56,040
Turpentine .....	76,316	62,191	82,498	68,613
Other articles .. Value £	..	..	78,940	76,417
Total value of oils...	..	..	620,563	747,101

## IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDING 31ST JULY.

Articles.		Quantities.		Values.	
		1893.	1894.	1893.	1894.
				£	£
Bark, Peruvian ..	Cwt.	8,458	1,639	15,635	7,721
Bristles.....	Lb.	105,065	350,093	87,062	51,687
Caoutchouc.....	Cwt.	21,656	19,254	241,489	177,908
Gum:—					
Arabic.....	"	5,369	2,521	15,096	7,612
Lac, &c.....	"	6,878	6,720	32,885	33,905
Gutta-percha ....	"	4,496	4,105	20,911	34,698
Hides, raw:—					
Dry.....	"	39,818	38,143	102,729	87,693
Wet.....	"	53,010	43,208	116,231	88,684
Ivory.....	"	1,033	890	44,231	36,346
Manure:—					
Guano.....	Tons	1,700	451	7,332	1,917
Bones.....	"	2,332	2,142	9,464	11,298
Nitrate of soda...	"	3,814	6,506	33,822	57,705
Phosphate of lime ..	"	37,250	35,930	64,334	67,667
Paraffin.....	Cwt.	37,587	20,835	36,703	22,194
Linen rags.....	Tons	960	1,122	5,347	11,403
Esparto.....	"	15,054	11,626	71,639	51,813
Pulp of wood ....	"	18,106	23,806	90,690	119,454
Rosin.....	Cwt.	123,097	121,512	40,666	29,894
Tallow and stearin ..	"	141,097	157,240	189,796	191,787
Tar.....	Barrels	12,087	12,538	8,145	6,931
Wood:—					
Hewn.....	Loads	259,087	248,514	494,746	465,774
Sawn.....	"	809,514	787,225	1,032,930	1,089,489
Staves.....	"	10,384	8,207	52,546	26,378
Mahogany .....	Tons	2,628	3,334	19,020	26,662
Other articles.... Value £	..	..	..	816,800	743,928
Total value.....	..	..	..	4,282,287	4,063,668

Besides the above, drugs to the value of 48,579*l.* were imported as against 68,482*l.* in July 1893.

## IMPORTS OF METALS FOR MONTH ENDING 31ST JULY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Copper:—			£	£
Ore..... Tons	10,391	9,759	58,925	46,287
Regulus ..... "	11,283	5,041	251,773	100,298
Unwrought .... "	3,140	5,666	141,190	226,843
Iron:—				
Ore..... "	350,667	432,807	236,231	291,661
Bolt, bar, &c.... "	7,009	3,957	63,681	37,071
Steel, unwrought.. "	739	603	6,613	5,050
Lead, pig and sheet "	16,471	11,601	157,924	105,641
Pyrites ..... "	62,041	50,119	112,567	85,032
Quicksilver..... Lb.	45,000	37,925	3,950	3,153
Silver ore..... Value £	..	..	280,624	166,995
Tin..... Cwt.	44,687	48,786	185,913	169,715
Zinc ..... Tons	5,744	4,459	100,606	69,198
Other articles ...Value £	..	..	130,443	142,202
Total value of metals	..	..	1,732,540	1,452,151

## IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH ENDING 31ST JULY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Alkali..... Cwt.	8,061	12,221	6,840	10,582
Bark (tanners', &c.) „	45,513	34,069	20,298	14,671
Brimstone..... „	74,948	21,769	18,983	5,062
Chemicals..... Value £	..	..	167,524	104,411
Cochineal ..... Cwt.	59	241	380	1,451
Cutch and gambier Tons	1,725	2,550	36,692	54,690
Dyes:—				
Aniline..... Value £	..	..	15,626	36,615
Alizarin ..... „	..	..	26,923	18,528
Other ..... „	..	..	629	6,398
Indigo ..... Cwt.	376	1,634	6,396	23,587
Nitrate of potash . „	16,388	25,312	14,136	22,478
Valonia ..... Tons	3,169	2,812	41,671	35,178
Other articles... Value £	..	..	126,960	118,219
Total value of chemicals	..	..	421,558	451,820

## EXPORTS OF METALS (OTHER THAN MACHINERY) FOR MONTH ENDING 31ST JULY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Brass..... Cwt.	11,340	10,170	£ 42,533	£ 28,993
Copper:—				
Unwrought..... „	67,197	29,086	160,104	62,453
Wrought..... „	26,935	22,224	77,064	58,786
Mixed metal.... „	22,174	16,327	51,621	34,425
Hardware..... Value £	..	..	175,181	143,464
Implements..... „	..	..	108,869	93,494
Iron and steel.... Tons	276,209	228,478	1,872,005	1,526,448
Lead .....	3,632	5,061	38,720	50,318
Plated wares... Value £	..	..	24,264	21,833
Telegraph wires „	..	..	77,305	393,483
Tin..... Cwt.	14,651	9,680	65,517	35,110
Zinc .....	14,552	14,816	12,527	9,869
Other articles .. Value £	..	..	63,463	52,810
Total value .....	..	..	2,769,176	2,429,516

## EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDING 31ST JULY.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Gunpowder..... Lb.	662,100	733,400	£ 15,148	£ 14,971
Military stores.. Value £	..	..	119,611	74,126
Candles..... Lb.	1,508,300	1,546,200	27,558	26,630
Caoutchouc..... Value £	..	..	94,343	107,359
Cement..... Tons	42,574	35,964	71,954	61,128
Products of coal Value £	..	..	83,789	77,904
Earthenware ... "	..	..	164,661	131,668
Stoneware..... "	..	..	12,362	13,829
Glass:—				
Plate..... Sq. Ft.	171,830	85,548	9,642	5,140
Flint..... Cwt.	7,765	6,430	18,953	14,843
Bottles..... "	50,276	42,986	24,898	21,697
Other kinds.... "	19,768	13,237	14,024	10,505
Leather:—				
Unwrought .... "	10,574	10,627	104,047	98,435
Wrought ..... Value £	..	..	26,737	18,544
Seed oil..... Tons	5,513	3,338	120,111	71,208
Floorcloth ..... Sq. Yds.	1,527,900	1,569,600	64,570	57,089
Painters' materials Val. £	..	..	120,401	106,606
Paper ..... Cwt.	79,054	71,597	123,728	105,941
Bags..... Tons	3,533	3,937	21,969	21,963
Soap..... Cwt.	51,290	45,853	52,736	49,233
Total value .....	..	..	2,457,727	2,141,421

**EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING  
31ST JULY.**

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Alkali..... Cwt.	352,568	444,000	129,471	121,393
Bleaching materials ..	122,978	99,750	59,380	38,686
Chemical manures. Tons	32,824	41,329	173,235	211,769
Medicines..... Value £	..	..	73,509	69,922
Other articles ... ..	..	..	225,074	215,254
<b>Total value .....</b>	..	..	612,667	657,024

15,206. W. Hood. Improvements in evaporating and distilling apparatus. August 9.

15,249. C. J. Bagley, L. Roberts, and the Moor Steel and Iron Co., Ltd. Improved arrangements and apparatus for the utilisation of the waste heat of furnaces of the open-hearth type. August 10.

**COMPLETE SPECIFICATIONS ACCEPTED.\***

1893.

15,209. K. E. Libert and J. A. Wentzel. Improvements in machines for compressing gases and the like. August 15.

23,595. J. Y. Johnson.—From C. Gouelle. Improvements in and connected with oil and other like presses. July 25.

1894.

9958. T. Craney. Improvements in evaporating apparatus. August 8.

9959. T. Craney. Improvements in evaporating apparatus. August 8.

**Monthly Patent List.**

\* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

**I.—GENERAL PLANT, APPARATUS, AND  
MACHINERY.**

**APPLICATIONS.**

13,857. G. Fletcher. Improvements in multiple-effect and other vessels for evaporating liquids. July 18.

14,014. E. J. Barbier. Improvements in or relating to condensing towers. July 20.

14,184. F. Kleemann. Improvements in the process of and apparatus for heating and concentrating liquids. Complete Specification. July 24.

14,192. W. P. Thompson. Improvements in separating mixed gases or liquids of different specific gravities and in apparatus therefor. July 24.

14,431. W. W. Pilkington. Improvements in gas generators or producers and furnaces and appliances for smelting glass, for melting and heating metals, alkalis, and other substances, and for heating steam-generators and for like purposes. July 27.

14,553. N. Teissier and G. L. A. Negre. Improvements in the manufacture of retorts of refractory material. July 28.

14,554. A. Lawrence, and Bennett, Sons, and Shears, Limited. Improvements in apparatus for heating and cooling liquids. July 28.

14,766. H. L. Doulton and L. E. Sanceau. Improvements in condensers used for the condensation of acid fumes. August 1.

14,767. H. L. Doulton and L. E. Sanceau. Improvements in the filling of towers used in the purification of acid and for such like purposes. August 1.

15,150. B. Dawson. Improvements in gas producers. August 8.

15,200. G. Bell and G. W. Bell. Improvements in the utilisation of the waste products of gas and the like engines. August 9.

**II.—FUEL, GAS, AND LIGHT.**

**APPLICATIONS.**

13,758. F. Mayer. Improvements in apparatus for manufacturing gas. Complete Specification. July 17.

13,841. A. Scott and P. McLean. Improvements in and relating to fire-clay and metal retorts for the manufacture of gas from coal, oil, tar, or other material. July 18.

14,196. A. D. Shrewsbury. Improvements in coke ovens. July 24.

14,761. C. T. Cayley. An improved furnace for the manufacture of oxygen. August 1.

14,772. J. Lyle. Improvements in block agglomerated and briquette fuels. August 1.

14,885. W. A. Fourness. Improvements in apparatus for generating and carburetting water-gas. August 3.

14,995. P. Dvorkovitz. Improvements in apparatus for obtaining gas and by-products from liquid hydrocarbons. August 4.

15,066. J. David. Manufacture of variegated candles and apparatus therefor. August 7.

15,290. S. B. Boulton, T. B. Haywood, H. E. Boulton, and H. Fergusson. See Class III.

15,323. J. Schrader. An improved process for purifying petroleum. August 11.

**COMPLETE SPECIFICATIONS ACCEPTED.**

1893.

16,220. R. Nobel. Improvements in the method of burning and controlling the combustion of solid fuel, and in hearths or furnaces therefor. August 1.

17,811. E. Manfield. Improvements relating to the manufacture of gas. August 1.

1894.

12,761. W. P. Thompson.—From E. Scott. A new or improved combustible material and process of manufacturing the same. August 8.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

#### APPLICATIONS.

13,847. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis and A. Haller, France. Process for purifying benzene by removing the thiophene which it contains. July 18.

15,290. S. B. Boulton, T. B. Haywood, H. E. Boulton, and H. Fergusson. Process for the purification or washing of coal-tar, naphtha, naphthalene, mineral burning oils, and the like. August 10.

### IV.—COLOURING MATTERS AND DYES.

#### APPLICATIONS.

13,695. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of nitro-amidoanthraquinone. July 16.

14,056. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of azo-colouring matters, derivatives of the 1,4- $\alpha$ -naphtholsulphonic acid from corresponding azo-colouring matters of naphthionic acid. July 21.

14,134. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of the 1,4,6 and 1,4,7- $\alpha$ -naphtholdisulphonic acids. July 23.

14,135. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of  $\alpha$ -naphtholmonosulphonic acid 1,4 from  $\alpha$ -naphtholcarbonate. July 23.

14,137. J. C. L. Durand, D. E. Huguenin, and A. J. J. d'Andiran. Manufacture of blue galloeyamin colouring matters. July 23.

14,461. G. W. Johnson.—From Kalle and Co. Improvements in and connected with the manufacture and production of chemical products suitable for use as or for dyes. July 27.

14,600. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Production of methylglyoxalidine CHN. July 30.

14,601. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. A process for the production of fast azo-colours upon the fibre. July 30.

14,745. J. C. L. Durand, D. E. Huguenin, and A. J. J. d'Andiran. Manufacture of new bases and colouring matters derived therefrom. August 1.

14,983. J. C. L. Durand, D. E. Huguenin, and A. J. J. d'Andiran. Manufacture of new colouring matters derived from muscarine. Filed August 4. Date applied for, January 16, 1894, being date of application in France.

15,064. J. C. L. Durand, D. E. Huguenin, and A. J. J. d'Andiran. Manufacture of new condensation products and colouring matters for dyeing and printing. Complete Specification. August 7.

15,067. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of  $\alpha$ -naphthylamine sulphonic acid. August 7.

15,205. T. R. Shillito.—From J. R. Geigy and Co., Switzerland. Production of green and blue dyes or colouring matters. August 9.

15,305. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. The production of yellow colour in or upon silk by the aid of diazo-compounds or nitrosamines. August 10.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

14,089. H. Goebel. See Class XI.

17,542. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Improvements in the production of oxyanthraquinone. July 25.

17,543. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of a novel colouring matter. July 25.

17,544. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Improvements in dyeing wool with sulphonic acids of anthraquinone colouring matters. July 25.

18,317. B. Willecox.—From The Badische Anilin und Soda Fabrik. The manufacture and production of new disazo dyes. July 25.

18,374. B. Willecox.—From The Badische Anilin und Soda Fabrik. The manufacture and production of new dyes of the phenanthrene series. August 1.

18,925. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik. Improvements in the manufacture of alphabetadimethyleurhodines. August 8.

1894.

12,214. O. Imray.—From The Society of Chemical Industry, Basle. Manufacture of polyoxythionine colouring matters dyeing on mordants. August 1.

### V.—TEXTILES, COTTON, WOOL, SILK, Etc.

#### APPLICATIONS.

13,672. E. Richard-Lagerie. Improvements in apparatus for extracting suint from wool and other substances. Complete Specification. July 16.

13,677. E. Richard-Lagerie. See Class XII.

14,036. T. Seddon and C. Long. An improved preparation for use in the manufacture of wool, woollen textiles, and the like. July 21.

14,299. W. Pickstone and A. Greenhalgh. An improved thick, soft, absorbent cloth. Complete Specification. July 25.

14,363. H. S. Close and W. O. Roff. Improvements in or in connection with retting flax, jute, or other similar fibrous materials, and eliminating the gummy or other matter therefrom, and utilising the resultant residuals. July 26.

14,788. J. S. Lenton and R. B. Caldieott. Improvements in the manufacture of textile fabrics, and in apparatus therefor. August 2.

14,903. T. Burrows. Improved means or apparatus for de-gumming or otherwise treating the fibres extracted from the plants, stems, or straws of flax, hemp, ramie, or the like. August 3.

15,140. A. Gruschwitz. An improved process for retting, cleansing, and deoxidising textile fibres. Complete Specification. August 8.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

19,095. H. Jacob. New or improved process and machinery for imparting a silky appearance to cotton and other yarns. August 15.

19,177. T. Pickles. Improvement in the treatment of cotton or linen warps or warp yarns for weaving purposes. August 15.

1894.

11,754. W. E. Heys.—From G. Mahard. Improvements in the process of and apparatus for scouring and de-greasing wool. July 25.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

### APPLICATIONS.

14,092. F. Barraclough. Improvements in apparatus for dyeing and otherwise treating yarns. July 23.

14,136. W. Mather. Improvements in apparatus for printing on paper or fabrics. July 23.

14,193. E. B. Manby. An improved thickening and fixing medium for colours in calico printing and dyeing and padding, also applicable to finishing purposes. July 24.

14,223. H. H. Lake.—From M. E. Waldstein, A. H. Peter, and E. Spott. United States. Improvements relating to the dyeing of fibre. Complete Specification. July 24.

14,275. J. Rhodes, jun. Improvements in apparatus for scouring and dyeing slubbing or slivers of fibrous material. July 25.

14,334. M. Cockroft and S. J. Pegg. Improvements in or appertaining to dyeing machines. July 26.

14,374. B. Walsh. Improvements in kiers or vessels for bleaching textile fabrics. July 26.

14,565. J. Booth and L. Thornley. Improvements in apparatus for dyeing, veneering, and washing hat bodies. July 30.

15,017. T. Illingworth. Improvements in apparatus for steaming textile fabrics. August 7.

15,305. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. See Class IV.

### COMPLETE SPECIFICATIONS ACCEPTED.

1894.

13,210. H. W. Sanderson. Improvements in and relating to the printing of textile fabrics. August 15.

## VII.—ACIDS, ALKALIS, AND SALTS.

### APPLICATIONS.

13,697. L. Lederer. A new process for manufacturing sodium persulphate, and for disinfection and preservation by means or persulphates. July 16.

14,054. W. Schulte and F. A. Sapp. Improvements in the manufacture of cyanide of potassium and in furnaces for producing cyanide of ammonia. July 21.

14,154. G. S. Albright and J. J. Hood. Improvements in the manufacture of sulphocyanides. July 23.

14,215. J. W. Scott. Improved process of rectifying sulphuric acid and means or apparatus therefor. July 24.

14,228. E. W. Enequist. Improvements in the process of manufacturing carbonic acid and in the production of by-products, such as sodium sulphate and magnesium sulphate. Complete Specification. July 24.

14,503. G. T. Beilby. A process for the recovery of hydro-cyanic acid from mixtures. July 28.

14,520. W. B. Maxfield.—From W. Dieterle and L. Rohmann, Germany. Producing nitric acid free of halogen and nitroso by blowing air into the mixture of nitric and sulphuric acid. Complete Specification. July 28.

14,856. A. Buisine. Improvements in extracting sulphur from raw pyrites or from its residuum, and recovering the by-products. August 2.

14,894. C. P. Peak, J. Peak, H. Peak, and A. G. Haddock. Improvements in the manufacture of soda crystals or washing soda. August 3.

14,905. E. Dresel and J. Lenhoff. Process for the manufacture of neutral sodium carbonate, neutral sodium sulphite, and sodium borates from sodium chloride. Complete Specification. August 3.

14,954. J. S. MacArthur. Improvements in apparatus for obtaining cyanides. August 4.

14,985. W. L. Wise.—From Solvay and Co., Belgium. Improvements in or relating to the manufacture or treatment of bicarbonate of soda. August 4.

14,986. W. L. Wise.—From E. Solvay, Belgium. Improvements in or relating to the manufacture of bleaching powder and other hypochlorites. August 4.

14,987. W. L. Wise. From Solvay and Co., Belgium. Improvements in or relating to the separation of caustic alkalis from the liquors obtained by the electrolysis of alkaline salts. August 4.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

16,760. A. Gordon and J. Gordon. Improved process and apparatus for the manufacture of cyanides. August 8.

18,831. W. Wolters. Improvements in the process of concentrating sulphuric acid in lead vessels. July 25.

1894.

12,881. H. H. Lake.—From L. Sternberg. Improvements in apparatus for obtaining anamonia and ammonia-salts from nitrogenous organic matters. August 8.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

### APPLICATIONS.

13,797. J. Slaytor. Improvements in or relating to moulds for casting balls of glass or the like material. July 17.

14,006. G. Bamberg. Improvements in apparatus particularly adapted for the direct manufacture of alkaline silicates and glass, but also applicable to other purposes. July 20.

14,018. J. F. Russell. An improved imitation stained glass. July 21.

14,875. J. C. Bailey. Improved methods of preparing "transfers" for use in decorating pottery ware. August 3.

14,938. W. Grüne, sen. Improvements relating to the ornamentation of porcelain, glass, and the like. Complete Specification. August 3.



## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

13,888. C. Copeland. Improved seggars for use in enamelling, hardening on lustre, and majolica kilns. August 8.

1894.

13,017. P. Shelly. Process of decorating pottery, glass, or similar goods. August 15.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

## APPLICATIONS.

13,926. J. B. Filloh, E. Hebrard, and L. Loustalot. Improvements relating to the treatment of wood for preserving and other purposes. July 19.

14,081. F. A. P. Stolte. Improvements in fireproof ceilings and floors. July 21.

14,145. J. E. Golding. Improvements in the construction of floors and ceilings. July 23.

14,210. W. W. Hughes. Improvements in or relating to drying cement and other materials. July 24.

14,308. A. E. Carey. Construction in combinations of plastic building materials and metal. July 25.

14,342. F. H. Brooke. Improvements in bricks for paving and the like. July 26.

14,494. W. Thomlinson. Improvements in the treatment of slag for the manufacture of cement. July 28.

14,599. E. Groebe. Improvements in the preservation of wood. Complete Specification. July 30.

14,699. W. Whittaker. Improvements in fireproof and waterproof ceilings and flues. July 31.

14,846. L. Mack. Improvements relating to the manufacture of cement from schist and bituminous marl. Complete Specification. August 2.

14,934. A. Muirhead and G. Hackford. Improvements relating to the manufacture of artificial stone and the like. August 3.

15,156. J. Roy. Improvements in or relating to the construction of fireproof floors or the like. August 8.

15,231. C. Howard. Improved method of vulcanising and drying wood. Complete Specification. August 9.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

16,470. T. C. Kitto. The asphalt and lead cement paving. August 15.

1894.

8,475. J. L. Kerr. Artificial plaster of Paris, made from the lime residue resulting from the reduction of sulphate of ammonia. July 25.

## X.—METALLURGY, MINING, Etc.

## APPLICATIONS.

13,683. J. P. V. Ploeg. Improvements in or relating to the chemical extraction of copper, silver, gold, and other metals accompanying them, from ores and materials containing them, with or without recovery of non-metals combined with them, and recovery of the chemicals used in the process. July 16.

13,851. W. T. Gibbs. Process for the treatment of nickeliferous iron ores. July 18.

13,868. H. C. Barker and R. Pearson. Improvements in the method of extracting gold from gold ores, quartz, and auriferous alluvials, and collecting and retaining float gold. July 18.

13,920. J. A. Mays. Process of and apparatus for comminuting molten substances. July 19.

14,155. J. J. Hood. Improvements in extracting metals. July 23.

14,306. Davies Bros. and Co., Lim., E. A. Davies, and S. T. Thomas. A novel or improved method of preparing metal sheets for the purpose of galvanising them or otherwise coating them with metal or metallic alloy, and apparatus for use therein. July 25.

14,327. A. F. B. Gomess. Improvements in the manufacture of aluminium and aluminium alloys, and the electro-deposition of the same. July 25.

14,481. A. Mieg. Improvements relating to the manufacture of tungsten articles and to apparatus therefor. July 27.

14,496. C. J. Sandahl. Improvements in the manufacture of iron. July 28.

14,502. J. W. Dixon and W. Skinner. An improved metallic alloy. Complete Specification. July 28.

14,547. E. L. Mayer. Improvement in the treatment of gold and silver ores, and of auriferous and argentiferous products derived from such ores. July 28.

14,568. J. H. Roberts and J. Cooper, jun. Improvements in the method of and apparatus for hardening and tempering steel wire. July 30.

14,649. E. Tarin. Improved process for the manufacture of aluminium. July 31.

14,706. W. W. Fyfe. Improvements in means for treating crushed complex or refractory ores in process of amalgamation. July 31.

15,225. S. Hufty and J. K. Caldwell. Certain improvements in processes of converting cast iron into malleable iron or steel. August 9.

15,339. J. Maetear. Improvements in the obtainment of precious metals. August 11.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

9045. H. W. G. Fairfax. Improved method of and apparatus for hardening and moulding or shaping steel plates and other articles. August 8.

15,507. S. O. Cowper-Coles. Improved method of and apparatus for pickling metal articles. August 1.

19,235. R. Brown. Improvements in apparatus for treating ores of precious metals with chemical agents. August 15.

19,375. W. F. Berner. Improvements in the manufacture or production of metals from their ores, and in plant or apparatus therefor. August 8.

1894.

9536. A. G. Fell. Improvements in methods of treating lead ores. July 25.

10,439. A. H. Moore and G. Whitlock. Improvements in and relating to the manufacture of combined iron and steel ingots. July 25.

11,797. E. D. Kendall. Improvement in the method or process of treating gold and silver ores, and a composition of matter for the same purpose. July 25.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### APPLICATIONS.

13,756. C. N. Waite. Improvements in or connected with electrolytic cells, and diaphragms therefor. Complete Specification. July 17.

13,795. W. P. Thompson.—From C. M. Barber, United States. Improvements in the process of electro-plating, and apparatus therefor. July 17.

13,836. C. J. Yarnold. Improvements in apparatus employed in the evolution and collection of hydrogen and oxygen gases by electrolysis. July 18.

14,327. A. F. B. Gomess. See Class X.

14,536. C. F. Parkinson and W. Gatwood. Improvements in apparatus for heating and welding by electricity. July 28.

14,634. C. E. Buell. Galvanic battery. Complete Specification. July 31.

14,642. V. I. Feeny.—From F. Duhero and P. Mohrdieck, United States. Electrical battery. Complete Specification. July 31.

14,689. F. Tallack, F. W. Tallack, and L. Hendle. Improvements in secondary batteries. July 31.

14,769. R. W. Hill. Improvements in or relating to apparatus for the production of sulphuric acid by the aid of an electric current. August 1.

14,770. R. W. Hill. New or improved automatic apparatus for use in and in connection with the production of sulphuric and other acids. August 1.

14,858. J. Y. Johnson.—From E. Javaux, C. F. Gallois, and F. Dupont, France. Improvements in the purification of saccharine juices by electrolysis. August 2.

15,276. M. Kiliani, W. Rathenau, C. Suter, and the Elektrochemische Werke Gesellschaft mit Beschränkter Haftung. Improvements in or connected with diaphragms for electrolytic purposes. Complete Specification. August 10.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

14,089. H. Goebel. Improvements relating to the manufacture of Paris blue or Berlin blue, and of Berlin green, by electrolysis. July 25.

14,130. J. Hargreaves and T. Bird. Improvements in the electrolysis of iodides, bromides, nitrates, and other salts. July 25.

14,131. J. Hargreaves and T. Bird. Improvements in the electrolysis of chlorides, iodides, bromides, nitrates, and other salts, and in apparatus therefor. July 25.

15,264. P. Marino. Improvements in plating metallic surfaces and also prepared surfaces of non-conducting substances by electro-chemical deposition. August 15.

17,866. L. W. Schöffer and G. E. W. Schöffer. An improved manufacture of electric accumulators. July 25.

1894.

11,664. T. Drake. An improved method, means, or process of producing caustic soda and chlorine gas, or caustic potash and chlorine gas, by the electrolysis of common salt in solution, or chloride of potassium in solution. August 1.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

### APPLICATIONS.

13,672. F. Richard-Lagerie. Improvements in apparatus for extracting suet from wool and other substances. Complete Specification. July 16.

13,677. F. Richard-Lagerie. Improvements in machines for extracting potash from the grease contained in sheep's wool. Complete Specification. July 16.

13,853. A. Gross, S. Seidenberger, and S. Leitersdorfer. Improvements in or relating to cleansing or washing agents. Complete Specification. July 18.

14,821. W. O. Robbins. A new or improved apparatus for treating substances containing fats and oils. Complete Specification. August 2.

14,946. S. Rosenblum and F. L. Battet. Improvements in soap, oils, greases, petroleum, mineral, vegetable, fish, and animal fats and oils. August 4.

15,086. B. J. B. Mills.—From J. H. Campbell and C. H. Campbell, United States. Improvements in the process of refining and improving butter, lard, oils, and other fatty matters. August 7.

15,228. J. O. Klimsch. Improvements relating to the manufacture of soaps. Complete Specification. August 9.

### COMPLETE SPECIFICATIONS ACCEPTED.

1894.

11,885. H. Perl. Improvements in and connected with soap pieces. July 25.

12,367. J. van Ruymbeke and W. F. Jobbins. Process of and apparatus for obtaining glycerine and other products from soapmakers' spent lyes. August 1.

13,104. F. Heilbron. Improvements in or applicable to soap in tablets, cakes, or bars. August 15.

## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, ETC

### APPLICATIONS.

13,873. W. M. Williams. Improvements in copying ink. July 19.

14,375. I. Frankenburg. Improvements in stoves for vulcanising and deodorising india-rubber waterproof garments. July 26.

14,407. D. Marcus. Improvements in varnishes. July 27.

14,598. R. J. Mostyn and F. Strickland. Improvements relating to chalk for manual use. July 30.

14,758. G. C. Dymond. From L. Auerbach and Co., Germany. Improvements in the manufacture of water-colour paints. August 1.

15,251. W. B. Priest. Improvements in the manufacture of white lead. August 10.

15,266. E. van den Hoff. An improved process for the manufacture of white lead. August 10.

15,317. W. B. Priest. Improvements in the manufacture of lead pigments. August 10.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

11,972. A. Macdonald. An improvement in the manufacture of sulphate of lead pigment. July 25.

17,477. J. Anderson, jun. Improvements in treating rubber waste for the separation of foreign substances therefrom: also applicable to other purposes. August 8.

17,581. J. E. Bedford, and C. S. Bedford. Improvements in the manufacture of a drying medium for paints and of oil varnish. July 25.

1894.

7465. A. Angell. Improvements in the manufacture of oil paints and compositions from carbonised or calcined ironstone or iron ore. July 25.

13,004. J. Clasen. A new or improved binding medium for size colours and method of manufacturing the same. August 8.

#### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

##### APPLICATIONS.

14,012. J. Y. Johnson.—From F. Kornacher and Diesel, Weise, and Co., Germany. Improvements in tanning leather. July .

14,162. J. Hibbert and E. Roberts. Improvements in apparatus for boiling starch and size. July 25.

14,165. J. Hibbert and E. Roberts. Improvements in apparatus for boiling starch and size. July 23.

14,697. F. Hartmann. A new or improved process for the production from skins or hides of a substitute for whalebone, horn, or the like. Complete Specification. July 31.

14,820. Brooke, Simpson, and Spiller, Limited, and R. J. Friswell. A new substance suitable for tanning, dyeing, and other purposes, and the processes for its production. August 2.

18,949. J. T. Hardy. An improved method or process of fastening the colours upon and within coloured leathers. August 8.

##### COMPLETE SPECIFICATION ACCEPTED.

1894.

13,414. A. Hermann. An improved process for colouring tanned hides by means of smoke. August 15.

#### XV.—AGRICULTURE AND MANURES, ETC.

##### APPLICATION.

14,195. W. Davidson. Improvements in apparatus for sowing artificial manure. July 24.

#### XVI.—SUGARS, STARCHES, GUMS, ETC.

##### APPLICATIONS.

14,606. R. H. Hunstock. An improved method of and apparatus for making syrups. July 30.

14,842. H. H. Leigh.—From G. Cambray, France. Improved process for the treatment of the juice of sugar-canes. Complete Specification. August 2.

14,858. J. Y. Johnson.—From E. Javaux, C. F. Gallois, and F. Dupont, France. See Class XI.

15,031. H. Walker. See Class XXI.

17,632. C. Steffen. Process for purifying and decolourising saccharine juices and sugar solutions generally. August 15.

##### COMPLETE SPECIFICATION ACCEPTED.

1894.

9054. H. H. Lake.—From A. L. Tédesco. Improvements relating to the manufacture of sugar, syrups, alcohol, and beer. August 8.

#### XVII.—BREWING, WINES, SPIRITS, ETC.

##### APPLICATIONS.

14,011. E. Weymar. Improvements in the method of and means for obtaining infusions of hops. July 20.

14,741. H. A. Leverett and A. Martin. A new method for maturing wines, spirits, oils, tobacco, or any other vegetable production. August 1.

14,918. L. Prochazka. An improved process and apparatus for rendering soluble the proteine in mash and wort. August 3.

14,919. L. Prochazka. An improved process and appliances for mashing and brewing in breweries. August 3.

17,912. A. W. Billings. Improvements in apparatus for the manufacture of beer. August 1.

##### COMPLETE SPECIFICATION ACCEPTED.

1893.

9054. H. H. Lake.—From A. L. Tédesco. See Class XVI.

#### XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

##### APPLICATIONS.

##### A.—Chemistry of Foods.

13,667. C. Greenhalgh. Improvements in the manufacture of candied lemon, orange, or citron peel. July 16.

13,712. R. G. Nash. Improvements in or relating to the sterilisation and preservation of milk. July 16.

14,015. H. J. N. Berge. Treatment of potable waters by alkalisation and subsequent neutralisation with a view to their sterilisation and purification. July 20.

14,179. G. Weddell. A new and improved baking powder. July 24.

14,332. E. Howarth. A new and improved process or means of preserving vegetables, cereals, and farinaceous foods in a cooked or partially cooked state or condition. July 26.

14,433. P. A. Noll. Process for manufacturing a compote from jelly. July 27.

14,675. B. F. McIntyre. Improvements in process of and apparatus for condensing and preserving milk. Complete Specification. July 31.

15,166. J. Swan. An improved method of treating eggs. August 8.

15,168. B. Biggs. Improvements in preserving extracts from lemons and the like. August 8.

*B.—Sanitary Chemistry.*

13,777. W. J. Pughley. Improved means of purifying the effluent waste waters of tin-plate works and the like. Complete Specification. July 17.

14,760. H. L. Sulman. Improvements in the treatment of sewage. August 1.

14,927. C. Jones. Improvements in the method or process of treating and utilising town refuse. August 3.

15,265. S. Brown. The purification of sewage or sewage matter, or the effluent from sewage or sewage matter. August 10.

*C.—Disinfectants.*

14,067. E. J. B. P. Jodelay and M. J. E. Laurans. Improvements in apparatus for mixing liquids designed for disinfecting and other purposes. July 21.

15,361. J. Y. Johnson.—From F. von Heyden, Germany. Improvements in the manufacture of antiseptic agents. August 11.

COMPLETE SPECIFICATIONS ACCEPTED.

*A.—Chemistry of Foods.*

1893.

15,762. J. Fordred. Improvements in the treatment of eggs. August 1.

17,732. E. Maris. Improvements in and in apparatus for the treatment of alimentary substances. July 25.

1894.

13,031. H. L. C. Paulsen. Improvements in and relating to the preservation of food. August 15.

*B.—Sanitary Chemistry.*

1893.

18,416. L. Archbutt and R. M. Decey. Improvements in the purification of sewage. August 8.

19,069. H. Lockwood. Improvements in the construction of tanks for the subsidence of the precipitate or sludge resulting from the chemical treatment of sewage or other foul waters. August 15.

1894.

12,903. Count Heinrich von Puckler. Improved means for purifying and disinfecting air, and for consuming deleterious gases and vapours. August 8.

*C.—Disinfectants.*

1893.

10,465. P. Hakansson. Improved antiseptic and preserving agents. August 1.

XIX.—PAPER, PASTEBOARD, Etc.

APPLICATIONS.

14,384. H. O. Band. Improvements in the manufacture of parchment. Complete Specification. July 26.

14,923. A. Gray. Improvements in the manufacture of articles impregnated with celluloid and the like, applicable also in the recovery of spirit and other solvents from celluloid and like solutions, and from varnishes. August 3.

COMPLETE SPECIFICATIONS ACCEPTED.

1893.

15,332. W. H. Coldwell. Improvements in apparatus used in the manufacture of paper-pulp. August 15.

1894.

14,440. E. Partington. Improvements in the treatment of "sulphite pulp" used in the manufacture of paper and the like from wood. August 15.

13,139. S. Hahn. A process of manufacture of films imitating mother-of-pearl. August 15.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

APPLICATIONS.

14,138. L. R. Seammell. An improved process for the extraction and estimation of eucalyptol. Complete Specification. July 23.

14,249. A. M. A. F. E. van Dyck. Improvements in or relating to camphor. July 24.

15,171. P. P. Monnet. A process for the manufacture of a scent essence. Complete Specification. August 8.

15,348. H. E. Newton.—From The Farnefabriken vormals F. Bayer & Co., Germany. The manufacture or production of chemical compounds. August 11.

COMPLETE SPECIFICATIONS ACCEPTED.

1893.

17,539. J. C. W. F. Tiemann. Production of substances suitable for manufacture of perfumery and aromatics. August 8.

17,703. A. Baur. Production of artificial musk from hydrogenated aromatic hydrocarbons. July 25.

17,781. A. Baur. A process for the production of artificial musk. July 25.

19,628. A. Muller and H. Dubois. An improved method of producing carbon tetrachloride. August 15.

20,245. L. O. Helmers. Improvements in the process of obtaining aqueous solutions of phenols and other substances. August 8.

1894.

12,796. F. W. Fletcher. Improvements in cinchona preparations. August 8.

XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.

APPLICATIONS.

14,161. J. Joly. Improvements in or relating to photographing in colours. Complete Specification. July 23.

15,031. H. Walker. A solution for use in mounting photographic prints. August 7.

## XIII—EXPLOSIVES, MATCHES, ETC.

## APPLICATIONS.

No. 1. J. J. J. J. J. Improvements in or relating to  
the use of blast charges in mines. Complete Specification.  
July 16.

14,064. T. Malson, W. A. Malson, S. R. Malson, and F. Smith. Improved fuse head for use in blasting. July 21.

11,665. W. A. Malson, T. Malson, S. R. Malson, and E. Smith. Improved electric fuse head for use in blasting. July 21.

14,068. R. Odeyn. A new or improved combustible composition. Complete Specification. July 21.

15,307. L. G. Searson. Improvements in the manufacture of gunpowder. August 11.

PATENT UNCLASSIFIABLE.

### APPLICATION.

13,838. G. Hagemann. Improvements in the manufacture of artificial cork. Complete Specification. July 18.

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## Journal and Patent\* Literature.

Class.	Page
I.—General Plant, Apparatus, and Machinery .....	869
II.—Fuel, Gas, and Light .....	872
III.—Destructive Distillation, Tar Products, &c. ....	874
IV.—Colouring Matters and Dyes .....	875
V.—Textiles: Cotton, Wool, Silk, &c. ....	880
VI.—Dyeing, Calico Printing, Paper Staining, and Bleaching .....	880
VII.—Acids, Alkalis, and Salts .....	884
VIII.—Glass, Pottery, and Enamels .....	888
IX.—Building Materials, Chays, Mortars and Cements..	888
X.—Metallurgy .....	889
XI.—Electro-Chemistry and Electro-Metallurgy .....	890
XII.—Fats, Oils, and Soap Manufacture .....	891
XIII.—Pigments and Paints; Resins, Varnishes, &c.; India-Rubber, &c. ....	893
XIV.—Tanning, Leather, Glue, and Size .....	894
XV.—Mannres, &c. ....	894
XVI.—Sugar, Starch, Gum, &c. ....	—
XVII.—Brewing, Wines, Spirits, &c. ....	895
XVIII.—Chemistry of Foods; Sanitary Chemistry and Water Purification; Disinfectants .....	897
XIX.—Paper, Pasteboard, &c. ....	900
XX.—Fine Chemicals, Alkaloids, Essences, and Extracts .....	902
XXI.—Photographic Materials and Processes .....	—
XXII.—Explosives, Matches, &c. ....	—
XXIII.—Analytical Chemistry .....	904

## I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

*The Pneumatic Pyrometer.* H. Morton. Engineering, 1894, 251—252.

A PERFECT pyrometer should possess the following characteristics:—

1. Applicable to all temperatures between the softening point of platinum and the freezing point of liquid air.
2. Giving constant indications by mere inspection, without any manipulation or calculation.
3. Allowing its indications to be observed at once in a number of locations at a considerable distance.
4. Operating automatically in every respect, and requiring no skill or special intelligence in its use.

The author having direct knowledge of an instrument proved to possess this remarkable combination of good qualities, draws the attention of others to the subject. An instrument which is said to fulfil the above conditions is the pneumatic pyrometer invented in the first instance by Mr. E. A. Uehling, M.E., and finally developed by him in combination with Dr. Alfred Steinbart. Its operation depends upon the action described as "effusion" by Graham in 1846 (*Phil. Trans.* 1846, 274), namely, the passing of a gas through a minute aperture in a thin plate. Suppose that we have a closed space or vessel through which a current of gas is forced or drawn by a constant pressure, and that the entrance and exit openings are minute apertures of the same size. In this case the differences of pressure causing the flow through the two apertures will be, roughly speaking, equal; or, in other words, the available force will divide itself between the two. Thus, suppose a certain pressure is available to force the gas through the apparatus, this will at first be all available to drive the air into the vessel, but the pressure within will at once begin to rise, because the air will come

in faster than it goes out, until the difference of pressure between the air in the interior of the vessel and the space into which it is delivered is equal to the difference between the pressure inside and outside the vessel. When this condition is established, the gas will pass out of the vessel as fast as it enters, and thus the pressure within the vessel will be constant. If arrangements are made by which the air forced in is heated at the instant of entrance, but cooled within to its original temperature at exit, experience shows that the density of the heated air and the mass introduced in a given time will be less, and (the outflow temperature being kept constant) more air will go out than comes in, until the interior pressure is reduced sufficiently to compensate for the diminished supply. We thus see that a rise in temperature of the entering air will cause a fall of pressure or tension in the air within the vessel, and this change in tension can be directly observed by a manometer connected with the vessel. Though calculation, as well as experiment, shows that the variations in density grow less in proportion with the rise in temperature, yet even at 1,200° F. to 1,500° F., with an available air pressure equal to 28 in. of water column, there will be a change of tension equal to 1 in. of water for each 100° F.

The first practical application of a pyrometer embodying the above principle has been to the hot blast of an iron-smelting furnace.

The pneumatic pyrometer, as illustrated, is shown as attached to the hot blast main of a blast furnace (Fig. 2), and is especially designed for that purpose. It consists of a platinum tube  $B^3$   $\frac{1}{2}$  in. in diameter and of sufficient length to extend very nearly through the lining of the hot blast main, to the shell of which it is secured by the coupling  $a$ , and the gland  $b$ , which is secured to the flange  $c$  bolted to the shell. Concentric within the tube  $B^3$ , is placed a smaller platinum tube  $c^1$   $\frac{3}{8}$  in. in diameter (Fig. 3), which extends very close to the end of the tube  $B^3$ , and is held in position by the coupling  $f$ . The connection between  $a$  and  $f$  is provided with a branch  $h$ , to which is connected a filter  $B^1$ . The latter consists of a chamber about 2 in. in diameter and 10 in. long, loosely filled with cotton-wool. The inner pipe  $c^1$  continues by the pipe  $i$  to the vessel  $C$  (Fig. 1), and within this by the pipe  $j$  and worm  $k$  to the coupling  $C^1$ , which is shown in detail (Figs. 4 and 5). The first small aperture is located at the end  $d^1$  of the inner tube  $c^1$ , and the second aperture is located in the coupling  $C^1$ , as shown in Fig. 4 at  $l$ . From  $C^1$  the tube continues by  $m$  to the top of the chamber  $A$ , which is connected with an aspirator (not shown) by the pipe  $n$ , and with the manometer  $q$  by the bent tube  $o$ ; it is further provided with a stopcock  $p$  and with a tube  $r$ , which is open to the atmosphere at the upper end, and reaches to within 3 in. of the bottom of the chamber  $A$ . The upper end of  $r$  passes through a stuffing-box, and is provided with a rack and pinion and wheel  $s$ , by which it may be raised or lowered. The pipe  $i$  communicates through the capillary tube  $t$  with the manometer  $u$ , and may be prolonged by means of a T-piece  $v$  and tube  $w$  to the office or any other place or places where it may be necessary or desirable to indicate the temperatures to be measured. The manometer tubes  $q$  and  $u$  have their lower ends immersed in coloured water contained in the reservoir  $R$ . The vessels  $A$  and  $C$ , which are made of brass tubes 7 in. in diameter, are respectively 4 ft. 8 in. and 1 ft. 6 in. long, and are connected with the water-supply pipe by the pipe connection  $E$   $F$   $T$  and  $E$   $S$   $N$ . A temperature regulator is shown at  $G$ , and with the valve  $K$  closed, and  $L$  and  $I$  open, is kept filled with water to the level of the overflow  $H$ . The pipe and worm  $x$   $y$   $z$ , which is connected with the steam or hot-air main, continually keeps the water boiling, and because  $C$  is open to the atmosphere through  $D$ , a constant temperature of 212° F. is maintained.  $A$  is the suction regulator. By opening the stopcock  $p$  and the valve  $M$  it is filled with water to the proper level. The tube  $r$  must be immersed into the water in  $A$  exactly 4 ft., which can be readily done by the wheel  $s$ . By these means the essentials for exact determination of temperatures are supplied. The aspirator, with a somewhat greater capacity than is required by the apertures, is turned on, the excess of exhaustion being

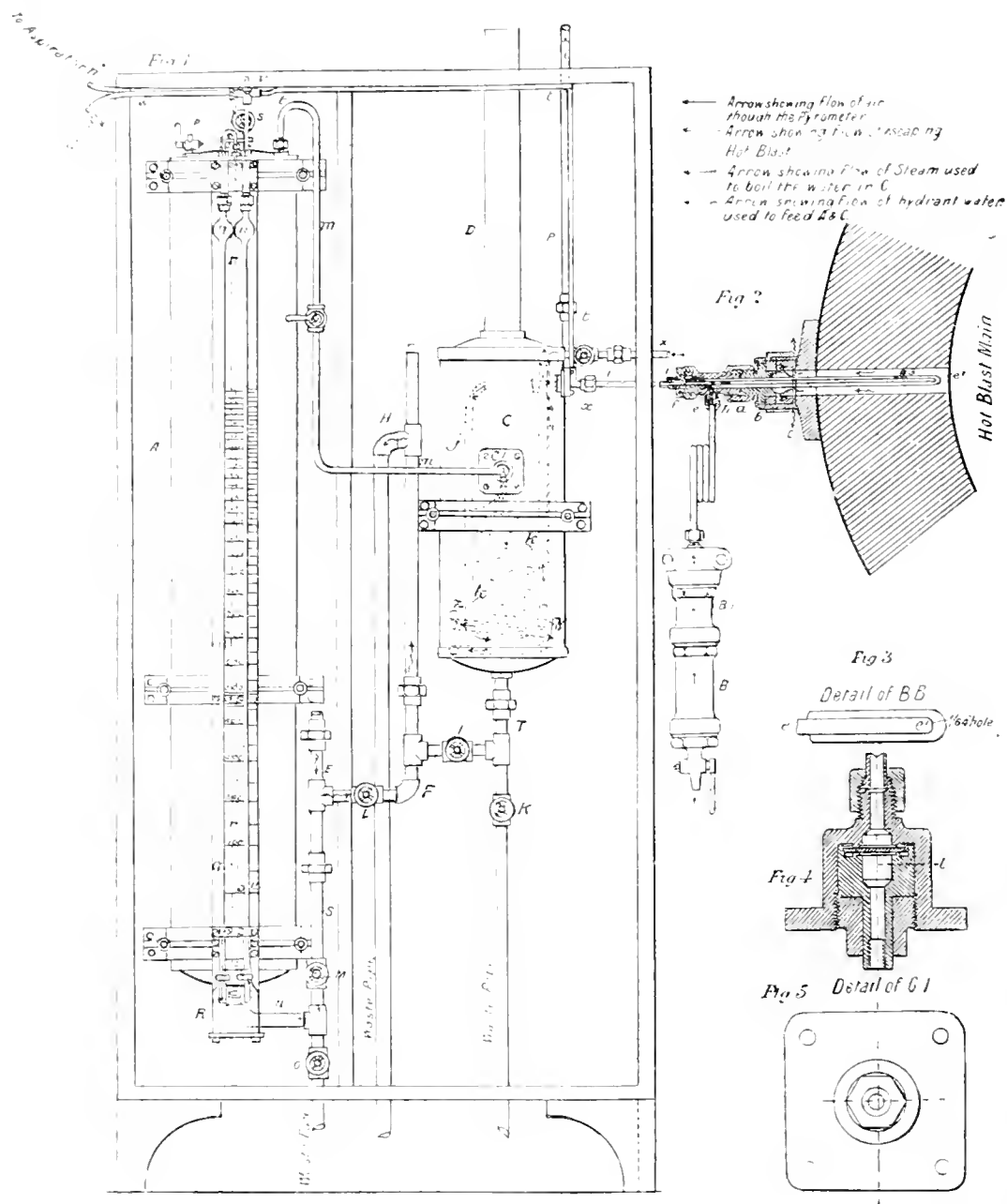
\* Any of these specifications may be obtained by post by remitting 8d.—the price now fixed for all specifications, postage included—to Sir Henry Reader Lusk, Comptroller of the Patent Office, Southampton Buildings Chancery Lane, London, W.C.



corrected through the tube *rr* against a constant head of 4 ft. of water, the suction at *C*<sup>1</sup>, the second aperture, cannot vary appreciably above or below that point. By the manometer *qq* the suction may be at any moment observed, and will be found standing steadily at the point 4 so long as the tube *rr* dips into the water to the proper depth.

The water in *A* very gradually evaporates, and as this occurs to an appreciable amount, *rr* is lowered; when it has reached the bottom it is again raised, and the waste is supplied through the valve *M*. This, in continual use, is found necessary about once a month.

The suction being applied, the air from the atmosphere enters through the filter *B*<sup>1</sup>, where it is freed from dust,



flows through *h* into the platinum tube *B*<sup>3</sup>, where it assumes the temperature of the hot blast in the main *B*, at which temperature it enters the first aperture at the end of *e e*<sup>1</sup>, by which it returns and passes through *i i* into *j*, and the worm *k k* located in the boiling water maintained in *C*. (Melting ice or any other constant temperature would answer as well.) In its passage through the worm it assumes the constant normal temperature at which it flows through the

second aperture located in *C*<sup>1</sup>, as shown in the detailed section (Fig. 4), thence through *m m* into the regulator *A*, and from it by the pipe *n n* to the aspirator.

As already explained, the tension in the chamber or tube between the two apertures is a correct indication of the difference of temperature at which the air flows through each of them respectively, therefore the manometer *u u* supplied with a proper index and connected to the tube *i i*

by the capillary tube *t t t*, will continuously and accurately indicate the temperature in B. The temperature manifesting itself as tension of a continuous current of air through *i i*, *j j*, *k k*, the length of the capillary tube *t t t* does not affect the accuracy of the indications, neither does the number of manometers connected, so long as the connections are perfectly air-tight. It is, therefore, quite feasible to indicate the temperature in as many places as may be desirable. It is also immaterial, so far as the accuracy of the indications is concerned, at what distance the tension and temperature regulators A and C are located from the place at which the temperature exists which is to be measured, so long as the area of the tube between the two apertures is sufficiently in excess of the area of the apertures. The latter being only  $\frac{1}{8}$  in. (and it can be made even less),  $\frac{3}{8}$ -in. pipe is ample for distances below 500 ft.

The pneumatic pyrometer can, therefore, readily be made semi-portable. By making the tube *i i* of sufficient length and flexibility, it would be quite feasible to change it from the smoke-box to the furnace, or into any portion of a flue of a boiler which may be undergoing a test.

Changes of temperature of the air in the conduit between the apertures affect the accuracy of the indications only momentarily. By virtue of the continuous flow of the air through the tube equilibrium is very soon established, and then the difference of temperature at which the air flows through the respective apertures determines the tension irrespective of its temperature while flowing from one aperture to the other. This property renders the pneumatic pyrometer available for measuring the temperature of very small areas as well as large ones. For instance, it is just as feasible to measure with it the temperature of the contents of a 10-grm. crucible as of 40 tons of molten steel contained in a huge ladle; and it can be made to indicate the temperature of the flame of a Bansen burner as accurately as heat of the flame issuing from a converter.

To call the above-described instrument a pyrometer is really a misnomer, for while it is capable of measuring temperatures up to where platinum ceases to be a solid, its range for low temperatures terminates only where air ceases to be a perfect gas. Its telemetric properties would seem to render it particularly applicable in cold-storage plants and all other industries where it is important to know the temperature of a place without going into it. The most important effect of the telemetric feature of this instrument will manifest itself in the moral force which it exerts on those who have to be relied upon to maintain the necessary or desirable temperatures. It is one thing to give instructions that certain temperatures shall be maintained, and another to know that the orders are carried out. If you have the means of knowing whether your orders have been faithfully carried out, they generally will be carried out, and *vice versa*.

*Notes on the Manufacture of Sulphuric Acid in America.* (Siphon.) G. Lunge. Zeits. angew. Chem. 1894, 133—137.

See under VII., page 884.

## PATENTS.

*Improvements in Centrifugal Separators.* O. Linden, Stockholm, Sweden. Eng. Pat. 9533, May 15, 1893.

WITHIN a rotating cylindrical drum is placed an annulus containing a number of radial channels, connecting the central space with a series of zig-zag vertical channels, running from the enclosed space at the bottom of the drum to the discharge outlet at the top. The liquid to be treated is led into the lower part of the drum by a central pipe, from whence it flows into the zig-zag channels, the heavier portion being retained there, but the lighter is caused to flow to the central annular space through the radial channels by the centrifugal action, and is there discharged at the top by suitable orifices into a collecting cistern. The heavy liquid also mounts up until it reaches the top of the apparatus and is discharged through a similar

set of orifices. To prevent the channels from being filled up with dirt or slime, a number of pockets are arranged outside the channels, communicating with these latter by orifices situated at the points of the zig-zag channels farthest from the centre.—E. G. C.

*An Improved Furnace.* R. Dempster, Des Moines, Iowa, U.S.A. Eng. Pat. 11,412, June 10, 1893.

THE improvements are means for supplying heated air to the products of combustion in furnaces, more especially in boiler furnaces, consisting of hollow fire-bridges, a series of flues in the side walls, and a heating coil.—R. B. P.

*Improvements in Hot Air Evaporating and Drying Machine.* O. T. Newton, Egremont, Cheshire, Eng. Pat. 14,924, August 1, 1893.

THE machine consists of a closed casing, divided into chambers by horizontal division plates, for evaporating, drying, and cooling respectively. The material to be treated is fed from a reservoir into a pivoted tray, the outer end of which overhangs and rests upon one of the buckets of an endless chain stretched over polygonal drums at each end. As each bucket passes under the tray it receives a charge of liquid material, and the hot air forced into the chamber through perforated pipes causes this liquid to rapidly evaporate, leaving a pasty mass, which is delivered at the other end of the chamber, and scraped out of the buckets by fixed knives. The pasty mass is then passed through a pair of rollers and delivered on to an endless band, where it is further dried by hot air and delivered to another travelling band to be cooled by currents of cold air, until it finally attains the temperature of the atmosphere, when it is delivered into receivers. The supply of material is regulated by a governor, actuating a weighted quadrant, and arranged to cut off the supply when the machine is not in motion.—E. G. C.

*Improvements relating to Rotary Drying, Pulverising, and Evaporating Machinery.* A. von Podewils, Munich, Germany. Eng. Pat. 15,772, August 19, 1893.

THE apparatus is intended to treat faecal matters, meat, bone, slaughter-house, fish, and vegetable refuse, and other matters requiring high temperatures for being dried and pulverised. It consists of a rotating drum, with end covers adapted for being heated, and formed of two conical or spherical discs arranged base to base; the axle of the drum is riveted to one of the two inner discs and connected by a stuffing box with the other; a pipe is led through one of the hollow trunnions (forming the axle) to serve as an outlet for either vapour or liquid. To pulverise the material in the drum, prism-shaped scrubbers are provided, with edges so arranged as to be kept sharpened by their rotating motion within the cylinder. The drum is heated externally by the hot gases from a furnace, and to prevent overheating when the drum stops, a governor rotated from the trunnions by a train of wheels controls the plate-valve of the flue, which conveys the hot air and gases to the casing round the drum. Modified constructions are described and illustrated.—E. G. C.

*Improvements in Apparatus for Separating Foreign Substances from Waste Rubber, Bark, Guai, and other Materials.* J. Anderson, junr., Manchester. Eng. Pat. 17,477, September 16, 1893.

AN oblong sheet of wire or gauze is surrounded by a wood framing to form a shallow tray, which latter is supported by rollers upon inclined guides, so that it is capable of rapidly oscillating to and fro under the influence of a crank and connecting-rod arrangement: a number of transverse undercut strips of wood are also arranged upon the upper surface of the gauze. The rubber or other substance to be treated is fed on to the upper end of the tray in a pulverised condition, and the heavier particles, such as nails, eyelets, and

the like, by the jiggling action set up, are arrested by the under cut wood stops, and pass out by means of inclined channels, to bags placed at the side, while the lighter and purer material passes over the strips, and is finally discharged at the lower end of the tray into a suitable box or receptacle.—E. G. C.

*Improvements relating to the Manufacture of Filters.* H. H. Lake, London. From Bittel and Co., Worms-on-the-Rhine. Eng. Pat. 19,873, October 21, 1893.

THE filters hitherto made by cementing together pulverised materials have been unsuitable for use with active solvents, such as acids or alkalis. The material prepared according to this process resists all the ordinary chemical solvents, except hydrofluoric acid.

Quartz, preferably as quartzose sand, and powdered glass are mixed together, and the mixture having been placed in boxes or moulds, is heated above the melting point of the glass, and is maintained at this high temperature for a considerable time. A chemical action takes place under these conditions, an acid silicate, or hard glass, being formed by the combination of the glass and quartz. The burning and annealing is carried out in horizontal moulds for the purpose of obtaining plates of uniform density. The structure into which the plates of the filtering material are formed consists of two plates connected by bolts without the employment of external frames, such plates being kept at the required distance apart by strips of packing material inserted between them at their peripheries.—V. C.

*Improvements in Evaporating Apparatus.* Y. Craney, Bay City, Michigan, U.S.A. Eng. Pat. 9958, May 22, 1894.

THE invention described is the combination of one or more furnace evaporating pans with two or more steam evaporating pans, whereby the steam generated in the furnace pans may be used for double or triple expansion in the steam pans. The furnace pan is a cylindrical casing, having an internal furnace provided with feed doors, and the combustion products therefrom circulate through a number of horizontal tubes and an external casing, thereby heating and evaporating the liquid, the vapour of which is led out by a goose neck to a condenser, or, if it is to be used for double or triple expansion, into the steam pans. The steam evaporating pans are formed of a sheet-metal casing having within a steam chamber connected with an outer steam ring and evaporating chamber. From the top of the evaporating chamber extends a goose neck having a condenser in the discharge leg. The steam evaporating pans are placed in a line, with a furnace pan at each end, and are connected by piping provided with valves, so that any combination of pans can be at work at one time. The settling chambers are below the pans, to which they are connected by a discharge leg connected to a trunk provided with a suitable elevator for carrying off the salt crystals as they form. With this arrangement any pan may be repaired while the others are in full work.—E. G. C.

*Improvements in Evaporating Apparatus.* T. Craney, Bay City, Michigan, U.S.A. Eng. Pat. 9959, May 22, 1894.

THE invention described is the combination of a series of steam evaporating chambers, of the type described in the Eng. Pat. 9958, May 22, 1894 (see preceding abstract), each provided with a discharge pipe for vapour and a settling leg or chamber, whereby each evaporator may be operated independently or in a series. To effect this the discharge pipes are connected to a common trunk having feed pipes to the various pans controlled by valves, whereby connection can be made with any of the pans.—E. G. C.

## II.—FUEL, GAS, AND LIGHT.

*American and Russian Petroleum.* Becker. Zeits. angew. Chem. 1894, 216—221.

AFTER dealing at some length with the characteristics of American and Russian mineral oils, and the influence of competition, &c. on the quality of the commercial article, the author gives in detail the results of a series of comparative tests applied to two petroleum, the one being American the other Russian (Nobel). The points to be elucidated were:—1. Physical properties. 2. Illuminating power (during a nine hours' continuous trial). 3. Consumption of oil. 4. Odour of the products of combustion. 5. Consumption of wick. The results are given in considerable detail. The palm was awarded to the Russian oil on account of:—(1.) Its distinctly higher flashing point, greater purity, and comparative freedom from products of low boiling point. (2.) Its constant illuminating power as compared with the American oil, which fluctuated between wide limits. (3.) Its distinctly higher efficiency as expressed in grms. of oil consumed per normal candle-hour.—H. T. P.

*The Manufacture of Water-gas in New York.* G. Lunge. Zeits. angew. Chem. 1894, 137—142.

ILLUMINATION by means of water-gas is rapidly gaining favour in America. In the first instance this is due to the fact that anthracite is plentiful in the States and gas coals scarce. The illuminating power of carburetted water-gas is greatly in excess of that of the best coal-gas; and whilst it is true that carburetted water-gas contains three times the quantity of carbon monoxide present in coal-gas, yet the danger arising from the prevalence of this poisonous gas is greatly minimised by the penetrating odour which it acquires in the process of carburetting. As regards costs of labour and superintendence, the comparison is said to be likewise in favour of water-gas.

At the Municipal Gas Works Tessié du Motay's process is in operation. The plant consists of five sets of twin generators, 2.5 × 2.5 metres in section, 3.5 metres high, and charged with anthracite and coke. Air is introduced below the bars for 10 minutes, and the gases so produced are allowed to escape. Steam of 200 lb. pressure is then passed in, and the resulting water-gas is led into a chamber, where it impinges against heated plates down which a current of naphtha is flowing. In the gases thus saturated with naphtha vapours, the latter are fixed by passing through a retort raised to white heat by means of liquid fuel. The product is then cooled, scrubbed, and finally purified with lime.

Ten generators and 18 sets of carburetting retorts produce in the winter season seven million cb. ft. of gas, containing 70 per cent. of water-gas proper and 30 per cent. of illuminating gases derived from naphtha. 440 lb. of anthracite and coke, and 260 lb. naphtha of 0.706 sp. gr., yield 10,000 cb. ft. of gas of 28 candle-power. Deposition of naphthalene in the pipes may be prevented by passing kerosene of 0.810 sp. gr. into the white-hot retorts.

Lowe's process is much superior, in which air and steam are passed alternately for a period of 10 minutes through the generator charge. When blowing with air the resulting gases are used for heating the carburetors, of which there are two in series; these are fed with a cheap crude mineral oil from Ohio. One of Lowe's cylinders measures 4 metres in diameter and 6 m. high, and supplies 1½ million cb. ft. of gas in 24 hours. The gas leaves the generator at 15 candle-power and the superheater at 28—30 candle-power.

Humphreys' apparatus is equally praised. It consists of two generators, two carburetors, and two superheaters. The generators are connected by means of a flue, and each of them is also in connection with a carburettor and a generator. When starting, air is blown into both generators; steam is introduced into the superheater to the right, and after passing downward in this and upward in the carburettor, it enters the generator to the right in a superheated condition; it then passes down this and upward in the left generator, and the gas so produced entering the left

carburettor, meets here four streams of oil. The gas mixture is "fixed" in the left superheater, and the operation is subsequently reversed.

Oxide of iron does not seem to find favour for purifying gas, and is almost entirely replaced with lime prepared from oyster shells. Attempts are now being made to revivify the spent lime.—H. A.

*The Action of Heat upon Ethylene.* V. B. Lewes.  
Proc. Royal Soc. 55, 90—107.

AFTER reviewing the work which has been carried out on the effect of heat on ethylene, and has led to the text-book equations, i.  $C_2H_4 = C_2 + 2H_2$  (as representing the reaction at very high temperatures), and ii.  $C_2H_4 = C + CH_4$  at lower temperatures, the author describes a long series of experiments of his own on this subject. In these he has passed ethylene, both alone and with hydrogen, and at various rates through 140 mm. of platinum tube 2 mm. in diameter, heated to various temperatures, the temperatures being determined by a Le Chatelier platinum-rhodium pyrometer inserted in the tube. Experiments made to test the point showed that platinum had no especial action on the hot gases, and could therefore be safely used. Experiments were also made on the effect of similar heating, on methane, ethane, and benzene, probable products of the reactions.

Below  $800^\circ C$ . practically no action takes place; at that temperature, traces of acetylene are formed. Between  $800^\circ$ — $900^\circ C$ . the acetylene increases in quantity, and large quantities of methane are generated, accompanied by liquid products. This action increases until just below  $1,200^\circ C$ . when hydrogen begins to appear amongst the products of decomposition, whilst at the same moment deposition of carbon commences, and the formation of oil begins to decrease, until at  $1,500^\circ$  the decomposition of the ethylene is practically complete and the products are mainly hydrogen and carbon, with a little undecomposed methane. The quantity of acetylene found is never large, but this is probably due to the nascent acetylene polymerising so readily between  $900^\circ$  and  $1,300^\circ C$ . and thus producing the oils found, and to the decomposition of the acetylene at high temperatures. Methane was found to be very stable at  $1,000^\circ C$ . slowly decomposed between that and  $1,300^\circ C$ . and rapidly at  $1,500^\circ$ . Benzene began to decompose rapidly above  $1,000^\circ$ . Ethane decomposes even below  $900^\circ C$ . with liberation of hydrogen and formation of ethylene and acetylene. This latter fact shows that ethane cannot be a primary decomposition product of ethylene, or hydrogen would have been formed below  $1,200^\circ C$ . when that gas was heated.

As the result of his experiments the author concludes that the primary action of heat on ethylene is expressed by the equation  $3C_2H_4 = 2C_2H_2 + 2CH_4$ , whilst the final decomposition is  $C_2H_4 = C_2 + 2H_2$ , and that between these two extremes there occur a large number of reactions due to the polymerisation of the acetylene formed from the ethylene, and also, at higher temperatures, from the methane, according to the equation  $2CH_4 = C_2H_2 + 3H_2$ .

—L. T. T.

*The Carburetting of Coal-Gas with Benzene.* H. Ries.  
J. Gasbeleucht. 37, 1894, 1.

ABOUT one-third of gas from Bohemian coal was carburetted with benzene and mixed in a gasometer of 1,800 cub. m. capacity, with uncarburetted gas. In order to fully increase the lighting power of 1 cb. m. of gas 4 grms. of benzene are necessary, which result agrees with the former researches of Bunte on the small scale. Benzene is so readily taken up by coal-gas that the most primitive apparatus suffices for the treatment, which is not difficult.

—T. A. G.

*Improvements in the Paraffin and Mineral Oil Industries.*  
F. W. Mann. Chem. Zeit. 18, 1894, 863.

See under III., page 875.

PATENTS.

*Improvements in and relating to the Manufacture of Fuel.*  
W. B. Hartridge, London. Eng. Pat. 12,389, June 23, 1893.

CARBONACEOUS material, such as granulated coal or coke, or vegetable waste, is incorporated with an agglutinating material, such as crude dextrin. The pasty mixture is moulded into the form of a casing or shell, with a loose cover. The shell is then filled with granulated coal, &c., the cover fixed on, and the whole dried. The shell, being porous, acts as a retort; the gas generated within it escaping through the pores and burning freely over its surface.—R. R. P.

*Improvements in Coking Processes and Apparatus, and in the Recovery of the Products of Distillation.*  
J. Bowing, Tilbury, Essex. Eng. Pat. 12,903, July 1, 1893.

THIS invention relates to improvements in the retort apparatus described in a former specification (Eng. Pat. 16,622, 1892; this Journal, 1893, 255). The improved retort consists of a vertical cylinder with an upper and a lower cover plate, through each of which there is a central hole. The top plate has, in addition, an outlet for the products of distillation, and a charging hole. When charging the retort a mandril is passed through the central holes in the cover plates to form a core. The material to be coked is then charged in, being packed closely round the mandril, and if necessary moistened with water, or tar, to make it cohere. The mandril is then carefully withdrawn, leaving a central due cored out through the mass to be coked. Heat is then applied by burning a suitable fuel, such as gas, in this central due, only just sufficient air being admitted for the combustion of the fuel.—R. B. P.

*Improvements in the Production of Illuminating Gas.*  
J. Smith, Sunbury-on-Thames. Eng. Pat. 14,545, July 28, 1893.

THE object of this invention is the production of oil-gas for enriching coal gas, especially that obtained from inferior coal. The inventor claims a cast-iron retort somewhat similar to the usual coal-gas type, but provided with one or more longitudinal partitions extending from the front end to within a short distance of the back end, where the chambers thus formed communicate with each other. The retort is set either alone or in the stack over the coal retorts, and so heated by the same furnace. The oil is injected as a spray by superheated steam at the front end of the retort, and the vapour formed passes backwards and forwards through the several chambers, and finally leaves through the front end of the retort by the ascension pipe, and enters the hydraulic main, which is constructed somewhat larger than usual. The gas carries with it a quantity of tarry matter, the condensation of which is facilitated by a stream of cold water entering the dip-pipe, and passing with the gas into the hydraulic main. The gas, water, and tar leave the hydraulic main in the usual way. The oil is supplied from a suitable reservoir through a graduated cock to a funnel, where air may also be admitted, the quantity being regulated by the second cock on the pipe leading from the funnel to the injector. Cocks are fitted to the front end of the retort and upon the outlet from the hydraulic main, to ascertain the condition of the gas, according to which the supply of oil is regulated.

Detailed drawings of this retort and a modification of the same accompany the specification.—S. P. E.

*Improved Apparatus for Vaporising Oil.* R. H. Laird and W. H. Laird, New Jersey, U.S.A. Eng. Pat. 15,432, August 14, 1893.

IN vaporising "crude oils," the best results are said to be obtained when the oil is distributed in thin sheets over large generating surfaces or trays, and the heat applied by a coil

of steam-pipes placed between the trays. At 212° F. the so-called "alcoholic series of vapours" is driven off. The heat is gradually increased to 300° F., when the lightest of the "oleic vapours" are given off; and so on at 450° and 650° F. respectively, when still heavier oils are expelled. The trays are arranged in a closed receptacle, made of boiler-plates. One or more standards or pillars are erected inside. Suitable perforated metal trays, sloping from the centre to a point near their peripheries, are passed over each standard, and rest upon radial arms affixed to the standard. The trays have a depending flange upon their edges, which rest upon the radial arms and enclose a space between the arms and trays, which are arranged in tiers to the top of each standard. A perforated coil of pipe is placed between the trays, and emits steam or hot water into the space between the trays. The oil runs from a feed-pipe on to the highest tray, falling over its edge on the next one, and so on till it reaches the bottom.—J. J. K.

*Improvements in Furnaces for Burning Refuse Material, such as Town's Refuse.* J. B. Allott and J. McC. C. Paton, Nottingham. Eng. Pat. 15,717, August 18, 1893.

See under XVIII. B., page 898.

*Improvements in the Method of Burning and Controlling the Combustion of Solid Fuel, and in Hearths or Furnaces therefor.* R. Nobel, Geta Aby, Sweden. Eng. Pat. 16,220, August 28, 1893.

This invention relates to specially constructed furnaces by means of which the author claims to be able to regulate the consumption of solid fuel without preventing the complete combustion of the same. Each of the several types of furnaces illustrated and referred to in the specification consists essentially of a closed fuel-chamber provided with one or more openings, through which a regulated supply of air enters. The bottom of this chamber is constructed of brickwork, through which one or more openings are left for the escape of the gases generated, or fire-bars may be employed. As the gases leave the fuel-chamber, where incomplete combustion only takes place, they meet with a further supply of air to complete the combustion, and then pass on to the flue surrounding the boiler or apparatus to be heated. The air supply to the gases is preferably heated by passing through tunnels in the brickwork or through hollow fire-bars. The ash-pit, like the fuel-chamber, is closed, and provided with a suitable door for removing the ashes. Accompanying the specification are a number of sectional drawings, illustrating several modifications of this type of furnace for special purposes.—S. P. E.

*Improvements relating to the Manufacture of Gas.* E. Mansfield, Manchester. Eng. Pat. 17,811, September 22, 1893.

This invention relates to plant for the manufacture of gas for heating and lighting purposes from sawdust, shavings, straw, leaves, cones, and similar vegetable products. The retorts are set in suitable brickwork, either vertically, horizontally, or the upper portion vertically and the lower portion inclined. Upon the upper or mouth end of the retort, outside the brickwork, and occupying the usual position of the hydraulic main in coal-gas works, is fixed a hopper, at the bottom of which a slide door allows the woody material to drop into the retort. The gas produced by the dry distillation of the vegetable matter is conveyed away in the ordinary manner to the hydraulic main, and purified as desired on its way to the gas-holder. The retorts are also provided with doors at the lower end, through which the ashes and carbon may be removed. Under the retorts there is a suitable furnace for burning coal or coke, the gases from which meet a second supply of air in the combustion-chamber, and thence pass to flues surrounding the retorts. Accompanying the specification are a series of drawings showing the setting of the retorts and furnaces, and giving other details.—S. P. E.

*Improvements in Saturators and Jets for producing Oxy-hydrogen or Lime Light.* A. Suiter, London, Eng. Pat. 18,224, September 28, 1893.

According to the inventor, when saturators are employed for producing hydrogen for oxy-hydrogen or lime light, the flame is apt to pass back and fire in the mixing chamber of the jet—an objection which this invention claims to remedy. The apparatus is said to be less in weight and bulk than the ordinary form, and requires less time to get into working order. The mixing chamber or burner of the jet is used as a saturator or vaporiser by stuffing it with a porous material that dips into a cistern also stuffed with a porous material containing beezoline or a similar volatile compound. By this means the saturator is rapidly heated, being in actual contact with the flame. The saturator is fitted with an adjustable sheath with projecting arms to conduct more or less heat, as the volatile nature of the beezoline or similar compound may require, and thus enable an excess of vapour to be produced and prevent the flame from passing back.—S. P. E.

*A New or Improved Combustible Material and process of manufacturing the same.* E. Scott, Chattanooga, U.S.A. Eng. Pat. 12,761, July 2, 1894.

This is a cheap fuel which is said to readily ignite, burn vigorously with perfect combustion, and to be adapted for kindling less combustible materials, also serving to diffuse around aromatic and other compounds. Logs of wood are generally employed, preferably of a resinous character; they are cut into lengths of about one foot, which are split down into slabs of one inch thick. These slabs are carried by an endless conveyor through a closed vessel, in which they are subjected to steam or hot water until they are sufficiently soft to yield to the pressure of the rolling-machine. This latter apparatus, consisting of two ridged or corrugated rollers, mounted in a strong frame and gear-driven, somewhat opens or separates the fibres without actually disintegrating the slabs, and produces a flexible fibrous woody material, which, being very porous, allows the air to penetrate. This mat-like material may be saturated with any aromatic compounds, and is easily rolled into compact bundles for transport.—S. P. E.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Spontaneous Ignition of Dried Petroleum Sludge.* R. Kissling. Zeits. angew. Chem. 1894, 197—199.

The occurrence in question was observed in a mineral-oil refinery. On opening the man-hole of a retort at the finish of a distillation, showers of sparks were seen to fall from the upper narrowed portion of the dome; and on removal of the latter, the phenomenon was found to be due to the spontaneous ignition of the sludge which had collected in a thick layer on a projecting portion of the dome. When freed from petroleum-oil, the sludge, formed a fine, black, flocculent powder having the following percentage composition:—Carbon, 67; iron, 24; sulphur, 9; obviously a mixture very liable to spontaneous ignition. A rough experiment showed that the dry powder when heated in the open air ignited at about 250° C. When saturated with paraffin oil, however, it did not fire even at 400° C.

It is probable, therefore, that towards the end of the distillation, the temperature in the retort must have been sufficiently high to expel the bulk of the oil from the sludge, the latter being subsequently ignited by the influx of air when the retort was drawn.—H. P. P.

*The Petroleum Industry in Baku in 1893.* Chem. Zeit. 18, 1894, 1130.

THE production of petroleum is becoming concentrated in the three districts of Balachani-Sabuntsehi, Bibi-Eibat, and the Romany Plain. The output of oil in 1893 was 337,051,834 poods, of which 324,763,197 poods (about 5,235,000 tons), or an increase of 13.6 per cent. over the previous year, was further worked in the refineries, the remainder being employed as fuel, or wasted. The increase is mainly due to the greater yield from fountains, where no pumping is needed, about one third of the total production being from this source. These fountains have lowered the price of crude petroleum, which varied between 2.75 kopeks (about  $\frac{1}{4}$ d.) per pood (36 lb.) in winter and 0.75 kopek ( $\frac{1}{4}$ d.) in summer. In 1893 there were about 38 fountains, and 458 wells where pumping was necessary, the average yield per pumping well being about 470,000 poods (about 7,577 tons) per annum. In 1893 there were 249.5 versts (about 165 miles) of pipe-lines, varying from 3 to 6 ins. in diameter, connecting the wells and factories, &c., and estimated to be capable of manufacturing about 1,440,000 poods (about 23,000 tons) of oil per day. In 1893 there were only 85 refineries, of which many were not working to their full capacity. This change is due to the growth of the large establishments at the cost of the small ones. The products of these refineries are about as follows:—Refined petroleum, 85,633,971 poods (about 1,380,000 tons); oils, 6,232,740 poods (109,000 tons); benzine, 252,193 poods (4,000 tons); goudron, asphalt, gasolin, &c., 437,620 poods (7,000 tons); and petroleum residuals, 143,538,890 poods (about 2,310,000 tons). These numbers are the exported amounts, and therefore exclusive of those used on the spot. The best works distil exclusively, or at least mainly, by driving superheated steam into the oil, and employ dephlegmators, though some works employ only fire heating. The vessels for acid and alkali treatment are usually fitted with air-agitators, but settling tanks are very commonly wanting, and in consequence the agitators or collecting tanks are used for this purpose with unsatisfactory results. From scarcity of fresh water, washing is often neglected or inefficiently carried out. Similarly, on economical grounds, insufficient quantities of acid and alkali are often used in purification, and in some cases a part of the soda has been replaced by the cheaper quicklime with doubtful results. Formerly the distillation was carried as far as possible, an average of 36.5 per cent. of the crude petroleum having been obtained as distillate. Now that the value of the residuals has risen and is generally higher than that of the oil, the distillation is usually not carried so far, only about 29.5 per cent. of the crude oil being distilled over. In addition to this crude oil, even asphalt, gasolin, &c., are sometimes added to the residuals.

The sulphuric acid and caustic soda used in the purification are now all made in the district, the first sulphuric acid chamber having been erected in 1880. In 1893 there were four factories making together about 9,000 tons of acid of 65°–66° Baumé, whilst in three regenerative factories about 2,700 tons of black acid were recovered. In 1893 there was one factory in work making soda from Glauber salt, and one Leblanc works in course of construction.—L. T. T.

*Improvements in the Paraffin and Mineral-Oil Industries.* F. W. Maun. Chem. Zeit. 18, 1894, 863–864.

A PAPER by Kern on this subject sets forth an account of some of the author's experiences during 12 years in America.

*Oil-gas.*—The economy of the so-called water-gas manufacture has caused water-gas almost completely to supplant retort coal-gas, since the manufacturer can produce a gas of 20–22 c.p. at a cost of 15 instead of 60 cents per 1,000 cub. ft., whilst the consumer obtains a gas of twice the luminosity at half the cost. The materials used embrace crude oils and by-products and residues from them, in

addition to coke or anthracite, the greater yield of gas from oils of low specific gravity being balanced by the higher luminosity of that from heavy oils, which permits the use of a larger proportion of water-gas. Any sulphur is removed by passing the gas through moistened sawdust, and then through slaked lime, sometimes mixed with ferrous sulphate. The author has retorted heavy mineral oils without steam, in order to obtain aromatic hydrocarbons, but the utility of the latter for the manufacture of dyestuffs was diminished through contamination by a small proportion of paraffins; a gas of great luminosity was obtained as a by-product. Some of the heavy mineral oils may be made to yield an excellent material for printers' ink.

The remainder of the paper contains brief reports on the subjects of the "*Influence of Temperature on the Gasification of Oils*," "*The Estimation of Paraffin*," "*On refining*," and "*On the use of petroleum as a boiler anti-incrustation medium*." On this latter subject it is stated that the action in the boilers is purely mechanical, saturating the deposit and preventing adhesion, but is perfect and characteristic of mineral oil. Light oils would be useless by reason of their volatility. (This Journal, 1893, 739; and 1894, 135.)—W. G. M.

*American and Russian Petroleum.* Becker. Zeits. angew. Chem. 1894, 246.

See under II., page 872.

#### PATENT.

*Improvements in the Process of Obtaining Aqueous Solutions of Phenols and other Substances.* L. O. Helmers. Hamburg, Germany. Eng. Pat. 20,245, October 26, 1893.

See under XVIII. C., page 900.

#### IV.—COLOURING MATTERS AND DYES.

*Some Sulphonations of Naphthalene Derivatives.* O. Dressel and R. Kothe. Ber. 27, 1193–1210.

THE naphthol and naphthylamine sulphonic acids, the importance of which in the manufacture of dyes has long been recognised, have only in recent years been submitted to scientific investigation. The establishment of the laws governing the substitutions in the naphthalene nucleus is due chiefly to the researches of Clève, and of Armstrong and Wynne.

The authors have studied the sulphonic acids derived from  $\beta$ -naphthylamine-7-sulphonic acid,  $\beta$ -naphthylamine-3, 7-disulphonic acid, and  $\beta$ -naphthol-7-sulphonic acid, and have come to the conclusion that Armstrong and Wynne's theory does not entirely hold good. They find that at high temperatures migrations of the sulphonic groups are possible in the naphthalene nucleus, even in the presence of sulphuric anhydride. Further, that by the influence of the 2-amido group sulphonation may under certain conditions take place in the 4-position, and that all  $\beta$ -naphthol and  $\beta$ -naphthylamine sulphonic acids containing a sulphonic group in the  $\alpha$ -ortho-position to the hydroxyl- or amido-group are distinguished by the splitting off of this sulphonic group on heating with dilute mineral acids, this reaction occurring in the main more easily in the case of the naphthol- than of the naphthylamine-sulphonic acids. Interesting and important details are given leading to these conclusions.—F. M.

*8-Anthraquinone.* K. Lagodzinski. Ber. 27, 1438—39.

By heating 8-anthrol with the calculated quantity of sodium nitrite and zinc chloride in alcoholic solution, and digesting the zinc compound formed with caustic soda, the sodium compound of nitroso-anthrol is obtained. The free nitroso-anthrol, separated from this by the addition of an acid, is by reduction with stannous chloride converted into amido-anthrol, which, by oxidation in a sulphuric acid solution with chromic acid, yields a new anthraquinone. The process of formation is analogous to that of 1,2-naphthoquinone from 8-naphthol. The new anthraquinone produces with ease the azine reaction with phenylene diamine, and is therefore an ortho-derivative, containing the two oxygen atoms probably in the position 1,2.—F. M.

*The Reduction of Nitro Compounds.* A. Wohl. Ber. 27, 1432—1437.

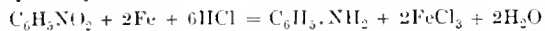
By the action of zinc-dust and water upon nitrobenzene *β*-phenylhydroxylamine is produced, according to the equation—

$$\text{C}_6\text{H}_5\text{NO}_2 + 2\text{Zn} + 3\text{H}_2\text{O} = \text{C}_6\text{H}_5\text{NH(OH)} + 2\text{Zn(OH)}_2$$

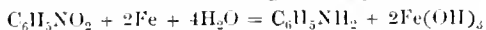
The instability of this compound in aqueous solution at elevated temperatures leads to the formation of azoxybenzene, which, by the reducing action of the zinc-dust, is converted into azobenzene. Aniline is formed at the same time by the direct reduction of the hydroxylamine. The consequent difficulties in the way of obtaining a satisfactory yield of phenylhydroxylamine were found to be obviated by the employment, in the place of pure water, of solutions of salts capable of forming insoluble basic double salts with zinc hydroxide, such as calcium, magnesium, and zinc chlorides. The addition of alcohol to the mixture is advantageous. By gradually adding 75 grms. of zinc-dust to a hot solution of 60 grms. of nitrobenzene and 6 grms. of anhydrous calcium chloride in 250 cc. of 60 per cent. alcohol, from 70 to 75 per cent. of the theoretical yield of hydroxylamine is obtained. Its separation from the mixture is effected by filtration and quick distilling off of the alcohol from the filtrate. The oily liquid which settles out solidifies on cooling to a cake, which after washing with water and drying is recrystallised from benzene.

Phenylhydroxylamine melts at 81—82°. It is readily soluble in alcohol, ether, carbon bisulphide, chloroform, and hot benzene; very slightly in petroleum spirit. Hot water dissolves 10 per cent., cold water 2 per cent. Phenylhydroxylamine is by the action of hot dilute mineral acids converted into amidophenol, and possesses the property of combining readily with aldehydes and phenyl-mustard oil, and of forming a benzoyl compound, ethyl derivatives, &c. In concentrated sulphuric acid it dissolves with a deep blue colour.

By substituting finely pulverised iron for the zinc-dust employed in the above reduction process, aniline alone is obtained. This observation furnishes the explanation of the fact that in the manufacture of aniline on the large scale only a very small portion ( $\frac{1}{30}$ th) of the acid required by the equation—



is actually used. The mechanism of the reaction is in reality the following:—First of all the iron combines with the hydrochloric acid, forming ferrous chloride, the nascent hydrogen reducing a corresponding quantity of nitrobenzene. The acid being used up, the iron now acts upon the nitrobenzene direct, according to the equation—



The ferrous chloride formed in the first stage of the process combines with the ferric hydroxide, forming a basic salt, thus playing the part of an acid and increasing the energy of the reaction. The correctness of this explanation of its action is proved by the fact of its being replaceable by calcium chloride and other salts which cannot act otherwise.

The presence of free acid or alkali and of nascent hydrogen have hitherto been considered essentials in the reduction processes practised in the laboratory, but the

preparation of phenylhydroxylamine and the analogous production of aniline on a manufacturing scale are examples of the possibility of effecting reductions in neutral solution by the direct conversion of finely-divided metal into hydroxide or highly basic salt.—F. M.

*The Behaviour of Triphenylmethane Colouring Matters to Nascent Bromine.* W. Vaubel. Chem. Zeit. 18, 1894, 1093.

The colouring matters of the triphenylmethane series, which contain amido and hydroxyl groups, possess the property of taking up bromine, a reaction which is more or less sharply defined. The test is carried out in the manner already described (Chem. Zeit. 1893, 1263, 1897; this Journal, 1894, 518), and acetic acid is used as a solvent for those colouring matters insoluble or sparingly soluble in water. *Tetramethyl-diamido-triphenylmethane* takes up 2 atoms of bromine, a further quantity acting as an oxidising agent. The sulphate of *Malachite Green S* takes up 2 atoms, the end reaction being well defined. *Paraleucaniline* takes up 6, whereas *Leucaniline* only takes up 5, and *Pararosaniline* and *Rosaniline* take up 5 and 4 atoms of bromine respectively, the end point in all these cases being well marked. *Hexamethyl-p-leucaniline* takes up 3 atoms, whilst *Crystal Violet* takes up 1 atom quickly and two very slowly. *Methyl Green* takes up 2 atoms with a sharp end reaction, whilst *Iodine Green* does not react readily with nascent bromine. *Aurin*, *Phenolphthalein*, and *Auramine* all take up 4 atoms of bromine. The phenylated rosanilines react similarly, and certain of the sulphonic acids are being investigated as to their behaviour towards nascent bromine. It is not proposed that the method should replace dyeing tests, but it is suggested that there are cases in which it might be employed technically.

—T. A. L.

## PATENTS.

*Improvements relating to the Manufacture of Paris Blue or Berlin Blue, and of Berlin Green, by Electrolysis.* H. Goebel, Garzweiler, Germany. Eng. Pat. 14,089, July 20, 1893.

THESE improvements relate to a process of manufacturing Paris blue or Berlin blue (Prussian blue), and Berlin green, by ozone produced by electrolysis; the process consisting in the precipitating of a solution of yellow prussiate of potash by means of a ferrous salt, such as green vitriol, suspending this precipitate in water, and subjecting it to electrolysis.

It is stated that a Paris blue or Prussian blue of an extremely vivid violet reflex and coppery lustre is obtained. Sulphuric, hydrochloric, nitric, or phosphoric acids may be added to the electrolyte so that it may contain from 1—20 per cent. of acid. The precipitate is only introduced next the anode, the contents of which compartment are kept stirred, as well as those of the cathode, if a precipitate is produced there. The hydrogen may be taken up by manganese peroxide or organic nitro derivatives. When the electrolytic action is continued too long the blue becomes more and more faint, and ultimately changes to dark Berlin green, which, when moist or in moist air, reverts to blue. A sample of the anode liquor is filtered and the operation stopped as soon as the filtered sample shows a reddish-violet tint.—J. C. R.

*Manufacture of 2,7-Amidonaphtholdisulpho Acid and of Colouring Matters derived therefrom.* C. D. Abel, London. From "The Actien Gesellschaft für Anilin Fabrikation," Berlin, Germany. Eng. Pat. 16,199, August 28, 1893.

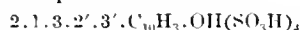
By sulphonating 2,2'-dihydroxynaphthalene under suitable conditions it is converted into a disulphonic acid which is easily soluble in water and is not precipitated by hydrochloric acid. The acid sodium salt is less soluble in water than the free acid, and is nearly insoluble in acid brine and insoluble in alcohol. When heated with ammonia under



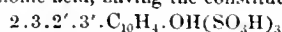
pressure it yields an amido-naphthol disulphonic acid:—25 kilos. of dihydroxynaphthalene disulphonic acid are heated with 75 kilos. of liquor ammonia containing 23 per cent. of ammonia for 14 hours to 180°–220° C. The melt is dissolved in water and the new acid is precipitated with hydrochloric acid. It is sparingly soluble in water, whilst the sodium salt is easily soluble, and the solution shows a bluish-green fluorescence. Mineral acids precipitate the acid from a solution of its sodium salt, but acetic acid does not. The 2,2'-amido-naphthol disulphonic acid is to be employed for the production of colouring matters, and for this purpose may itself be diazotised and combined with amines and phenols and their derivatives; or the acid itself will combine with diazo- or tetrazo-compounds, the combination taking place in an alkaline or a slightly acid solution. The colouring matters thus obtained contain a diazotisable amido group, which may be diazotised and combined on the fibre, the colours produced varying from brown to greyish-black.—T. A. L.

*The Manufacture or Production of Sulpho Acids derived from β-Naphthol-β-Monosulpho Acid.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 17,141, September 12, 1893.

It has hitherto been supposed that the only acid produced by sulphonating β-naphthol sulphonic acid F (2,2'-naphthol sulphonic acid) is 2,3,2'-naphthol disulphonic acid, but the present specification describes processes for the production of a tri- and a tetra-sulphonic acid from the F acid. These two acids on hydrolysis lose one sulphonic acid group, and are converted into new naphthol di- and tri-sulphonic acids. All these naphthol sulphonic acids when heated with ammonia, produce the corresponding naphthylamine sulphonic acids, and one of the β-naphthylamine tri-sulphonic acids so formed when fused with caustic soda gives rise to a new amido-naphthol disulphonic acid. The following new acids are described:—β-naphthol trisulphonic acid, 2,1,3,2'.C<sub>10</sub>H<sub>4</sub>.OH(SO<sub>3</sub>H)<sub>3</sub>. One kilo. of sodium 2,2'-naphthol sulphonate is mixed with 3 kilos. of fuming sulphuric acid containing 25 per cent. of anhydride, and allowed to cool. The melt is then heated to 80°–90° C. until a sample no longer combines with diazo compounds, when the whole is poured on the ice, neutralised at a low temperature with slaked lime, the calcium salt converted into the sodium salt, and the solution of this evaporated down. On hydrolysing this acid by boiling it with 10 times its weight of 10 per cent. hydrochloric acid it is converted into so-called β-naphthol-β-disulphonic acid of Ger. Pat. 44,079, the sulphonic acid group of the trisulphonic acid ortho to the hydroxyl having been removed on hydrolysis. β-naphthol tetrasulphonic acid—



20 kilos. of sodium 2,2'-naphthol sulphonate are gradually added to 80 kilos. of fuming sulphuric acid containing 40 per cent. anhydride and heated to 120°–130° C. for 8–10 hours. The melt generally goes solid, and sulphonation is complete when a test no longer gives a colour with diazo-compounds, and gives a white sandy barium salt sparingly soluble even in boiling water. The melt is then added to ice, neutralised with lime, precipitated with sodium carbonate, and the solution of the β-naphthol sodium tetrasulphonate is evaporated to dryness. The constitution of the acid was determined by converting it first into a trisulphonic acid by heating with dilute acid, and then into the corresponding naphthylamine trisulphonic acid from which the amido group was removed. The naphthalene trisulphonic acid so obtained was identical with the 2,3,3'-naphthalene trisulphonic acid of Eng. Pat. 11,865 of 1892 (this Journal, 1893, 595). As already mentioned, when boiled with dilute acids the tetrasulphonic acid is converted into a trisulphonic acid, having the constitution—



the sulphonic acid group ortho to the hydroxyl having been split off as in the case of the trisulphonic acid mentioned above. This new trisulphonic acid differs from the former one in combining with diazo-compounds, and it is to

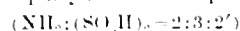
be used for the preparation of azo colouring matters. All the naphthol sulphonic acids described can, on heating with ammonia, be converted into the corresponding naphthylamine sulphonic acids; thus 2,1,3,2'-naphthol trisulphonic acid gives 2,1,3,2'-naphthylamine trisulphonic acid, which, on heating with dilute mineral acids, yields 2,3,2'-naphthylamine disulphonic acid. In a similar manner—



gives 2,1,3,2'.C<sub>10</sub>H<sub>4</sub>.NH<sub>2</sub>(SO<sub>3</sub>H)<sub>4</sub>, which on hydrolysis yields 2,3,2'.C<sub>10</sub>H<sub>4</sub>.NH<sub>2</sub>(SO<sub>3</sub>H)<sub>3</sub>, or this last acid may be obtained directly by the action of ammonia on 2,3,2'.C<sub>10</sub>H<sub>4</sub>.OH(SO<sub>3</sub>H)<sub>3</sub>. This last-named acid, when fused with caustic soda at 180°–240° C., gives a new amido-naphthol disulphonic acid, having probably the following constitution, 2,2',3,3'.C<sub>10</sub>H<sub>4</sub>.NH<sub>2</sub>.OH(SO<sub>3</sub>H)<sub>2</sub>. This acid yields a diazo compound which can be salted out in yellow flakes. It also combines with diazo and tetrazo compounds, yielding valuable dyestuffs.—F. A. L.

*The Manufacture or Production of Sulpho Acids derived from β-Naphthylamine-β,β'-Disulpho Acid.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 17,141A, September 12, 1893.

By sulphonating 2,3,2'-naphthylaminedisulphonic acid with fuming sulphuric acid the first product of the reaction is 2,1,3,2'-naphthylaminetrisulphonic acid, which, on further heating with fuming sulphuric acid, is converted into a mixture of two isomers, the sulphonic acid group in the position 1 "wandering" into the 3' and 4'-position forming the (2,3,2',1') and (2,3,2',3') naphthylaminetrisulphonic acids. When the latter of these two acids is further acted on by fuming sulphuric acid it is converted into 2,1,3,2',3'-naphthylaminetetrasulphonic acid. For the preparation of the 2,1,3,2'-naphthylamine trisulphonic acid, 1 kilo. of β-naphthylamine-β-disulphonic acid—



is mixed with 3–4 kilos. of fuming sulphuric acid containing 40 per cent. of anhydride and heated for several hours to 80°–90° C., until a sample, when diazotised and combined with R-salt, gives an easily soluble brownish orange-red colouring matter. The melt is then poured on to ice, and the acid converted at a low temperature into its calcium salt, from which the sodium salt is obtained. By continuing the action of the sulphuric acid for about 20–30 hours, and raising the temperature of the melt to 130° C., the acid first produced is converted into two isomeric trisulphonic acids. The melt also contains a β-naphthylamine tetrasulphonic acid, which, on hydrolysis, is converted into one of the isomeric trisulphonic acids simultaneously produced. The melt therefore as obtained above is poured on to ice, diluted with water, and boiled. The solution then contains two trisulphonic acids, which can be separated by the difference in solubility of their sodium salts. Taking the quantities given above, the solution of the sodium salt is evaporated to 5 litres and then acidulated with hydrochloric acid, when the acid sodium salt of 2,3,2',3'-naphthylamine-trisulphonic acid separates in long needles and can be filtered off. The solution is saturated with salt, when the acid sodium salt of 2,3,2',4'-naphthylaminetrisulphonic acid crystallises out in nodules. In order to prepare the tetrasulphonic acid, 1 kilo. of 2,3,2',3'-naphthylaminetrisulphonic acid, or of its acid sodium salt, is mixed with 3–4 kilos. of fuming sulphuric acid containing 40 per cent. of anhydride and heated at 120°–140° C. until a sample carefully poured on to ice, diazotised and combined with R-salt, yields an easily-soluble brown colouring matter. The colouring matter from diazotised 2,3,2',3'-naphthylamine trisulphonic acid and R-salt is only partially soluble. The melt is then poured on to ice and neutralised at a low temperature with slaked lime. The sodium salt is prepared from the solution of the calcium salt, and after evaporation the tetrasulphonic acid is precipitated by adding hydrochloric acid to the cold solution. All the β-naphthylamine sulphonic acids herein described are to be employed for the production of dyestuffs.—T. A. L.

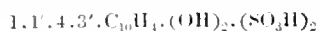


*The Manufacture or Production of  $\alpha$ -Naphthylamine  $\beta$ - $\alpha$ - $\beta$ -Trisulpho Acid and of other Sulpho Acids derived therefrom.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 17,141n, September 12, 1893.

THE new  $\alpha$ -naphthylamine trisulphonic acid referred to has the constitution  $1.2.2'.1'.4'.11_1.NH_2.(SO_3H)_3$ , and can be obtained by the action of sulphonating agents on  $1.4'.2$ -naphthylamine disulphonic acid of Ger. Pat. 69,555,  $1.2.4'$ -naphthylamine disulphonic acid of Ger. Pat. 56,563,  $1.2$ -naphthylamine sulphonic acid, or  $1.4'$ -acetamidonaphthalene sulphonic acid. In order to obtain it from one of the disulphonic acids the acid sodium salt is mixed with three times its weight of fuming sulphuric acid, containing 40 per cent. of anhydride, and heated for about 10 hours to  $120-130^\circ C$ . The melt usually becomes solid, and after stirring into ice-water the liquor is saturated with lime, converted into the sodium salt, and after evaporation acidulated, when the acid sodium salt crystallises out on cooling. The salt so obtained forms nodular aggregates of fine white needles readily soluble in water, the solution showing a green fluorescence. The diazo compound crystallises in yellow needles readily soluble in water. On heating this acid under pressure with an equal weight of caustic potash and of water to  $180-200^\circ C$ , a new amido-naphthol disulphonic acid is obtained, which precipitates when the melt is acidulated. The new acid, which probably has the constitution  $1.4'.2.2'.C_{10}H_4.NH_2.OH.(SO_3H)_2$ , gives a sparingly soluble acid, but an easily soluble neutral sodium salt, the solutions showing a blue fluorescence. The diazo-compound can be salted out as a mass of orange-red crystals, and when added to alkalis produces an unstable blue colour which rapidly changes to orange. Ferric chloride added to a solution of the acid sodium salt produces a claret-red, whilst chloride of lime gives a yellow colour. The amido-naphthol disulphonic acid and the naphthylamine trisulphonic acid are to be used for the production of colouring matters, and the latter is also to be employed for obtaining other new sulphonic acids of the naphthalene series.—T. A. L.

*The Manufacture or Production of Sulpho Acids derived from Naphthalene  $\alpha$ - $\beta$ - $\alpha$ - (or  $\alpha$ - $\beta$ - $\alpha$ -) Trisulpho Acid, or analogous Sulpho Acids.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 17,141c, September 12, 1893.

By the action of nitric acid on  $1.3.4'$ -naphthalene trisulphonic acid, the nitro-group enters the molecule in the  $1'$ -position yielding  $1.4.1'.3'.C_{10}H_4.NO_2.(SO_3H)_3$ . This constitution was proved as follows:—The nitro-acid, on reduction, yields  $\alpha$ -naphthylamine trisulphonic acid, which, on diazotisation and boiling with dilute sulphuric acid, gives a naphthosultone disulphonic acid. This, on fusing with alkalis, gives an  $\alpha$ -naphthol trisulphonic acid different from the  $\alpha$ -naphthol trisulphonic acid S of Eng. Pat. 3397 of 1890 (this Journal, 1892, 999), which has the constitution  $1.2.4.1'.C_{10}H_4.OH.(SO_3H)_3$ . Starting from  $1.4'$ -naphthalene sodium disulphonate, which on sulphonation at  $130^\circ C$  is converted into the  $1.3.4'$ -naphthalene trisulphonic acid, the specification describes the preparation of the various derivatives mentioned above. If, instead of diazotising the  $1.4.1'.3'$ -naphthylamine trisulphonic acid, it is fused with alkalis, a new amido naphthol disulphonic acid, of the formula  $1.1'.4.3'.C_{10}H_4.NH_2.OH.(SO_3H)_2$ , is produced. The  $\alpha$ -naphthol trisulphonic acid obtained from the  $1.4.1'.3'$ -naphthylamine trisulphonic acid, when fused with alkalis, gives a new dihydroxy-naphthalene disulphonic acid having the constitution—



By carrying the fusion with alkalis to a higher temperature, another sulphonic acid group is replaced, and there results  $1.1'.3.3'$ -tri-hydroxy-naphthalene sulphonic acid, which, like the other derivatives mentioned, can also be used for the production of colouring matters.—T. A. L.

*Improvements in the Production of Oxy-Anthraquinone.* O. Imray, London. From "The Farbwerke vormals Meister, Lucius, and Brüning," Hoechst-on-the-Maine, Germany. Eng. Pat. 17,542, September 18, 1893.

CONTRARY to what has been previously stated, it is found that nitro-anthraquinone, when boiled with caustic alkalis, is converted into *o*-hydroxy-anthraquinone. Nitro-anthraquinone, obtained by nitrating anthraquinone in sulphuric acid, is heated in a vessel provided with a reflux condenser with an equal weight of caustic soda and 10–20 times its weight of ethyl or methyl alcohol. The boiling is continued for about two days, when the liquor is poured into cold water, and the sparingly-soluble sodium salt separates out. The *o*-hydroxy-anthraquinone is purified by precipitation from its dilute alkaline solution.—T. A. L.

*Manufacture of a Novel Colouring Matter.* O. Imray, London. From "The Farbenfabriken vormals Meister, Lucius, and Brüning," Hoechst-on-the-Maine, Germany. Eng. Pat. 17,543, September 18, 1893.

THE colouring matter described is obtained by the action of alkalis on the product formed by the acid reduction of dinitro-anthrachryson disulphonic acid. By reducing dinitro-anthrachryson disulphonic acid in an acid solution a colouring matter is obtained which dyes wool violet from an acid bath and chromed wool blue. By boiling the product obtained with a somewhat dilute sodium carbonate solution for about two hours, a red crystalline compound gradually separates. The solution is made slightly acid and the colouring matter is precipitated with salt from the hot solution. It forms a red crystalline powder dissolving in water with a red and in dilute alkalis with a blue colour, whilst an excess of alkali precipitates the alkali salt of the sulphonic acid, which is sparingly soluble. In concentrated sulphuric acid the dyestuff dissolves with a fluorescent bluish-red coloration. The free sulphonic acid is only slightly soluble in hot alcohol and acetone. The colouring matter dyes wool red from an acid bath and blue on a chrome mordant. The shades produced are said to be fast to light and fulling.—T. A. L.

*Improvements in the Manufacture of Beta-methyl-eurhodine.* B. Willeox, London. From "The Badische Anilin and Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 17,572, September 18, 1893.

THIS is an extension of Eng. Pats. 10,138 and 10,138A of 1892 (this Journal, 1893, 593 and 594), giving an alternative method for the production of  $\beta$ -methyl-eurhodine, which originally was prepared by heating the azo dyestuff from diazo sulphanilic acid and methyl-*p*-toluidine, together with  $\alpha$ -naphthylamine hydrochloride in presence of a suitable diluent. According to the present specification the same product is obtained by the interaction of  $\beta$ -hydroxy-naphthoquinone imide and methyl-*o*-tolylene diamine. The following is the method employed—About 173 kilos. of  $\beta$ -hydroxy-naphthoquinone imide, 136 kilos. of methyl-*o*-tolylene diamine, and 1,000 kilos. of glacial acetic acid, are heated on the water-bath. When the reaction is complete, which is ascertained by working up a test from time to time until no increase in the amount of colouring matter formed takes place, the melt is repeatedly extracted by boiling with very dilute hydrochloric acid. The extracts are filtered, and when cold are precipitated with caustic soda. The brown precipitate obtained is filtered, washed with water, and dissolved in dilute hydrochloric acid, from which, after filtering, the  $\beta$ -methyl-eurhodine is precipitated by adding salt. It may be further purified by dissolving in a slight excess of ammonia, and, after filtering, reprecipitating with salt. The  $\beta$ -methyl-eurhodine hydrochloride so obtained is a red crystalline powder, which dissolves easily in water to a red solution.—T. A. L.

*The Manufacture and Production of New Diazo Dyes.* B. Willcox, London. From "The Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 18,317, September 29, 1893.

THE colouring matters obtainable according to this invention are produced by the action of diazo compounds on 1,4-naphthylene diamine, the combination taking place first in an acid and afterwards in an alkaline solution. Dyestuffs from diazo compounds and naphthylene diamine appear to have been obtained according to Eng. Pat. 17,466 of 1887 (this Journal, 1888, 839), but these compounds on treatment with nitrous acid are diazotised, whereas the products obtained according to the present invention are destroyed by nitrous acid. About 23 kilos. of 1,4'-naphthylene diamine are dissolved in 600 litres of water, cooled, and the diazo-sulphanilic acid from 19.5 kilos. of sodium sulphanilate, 50 kilos. of 27 per cent. hydrochloric acid, 400 litres of water, and 7 kilos. of sodium nitrate, is run in. The formation of the colouring matter is not complete for about 12 to 24 hours, during which the mixture is stirred. When a sample on boiling no longer evolves nitrogen, a solution of 120 kilos. of caustic soda in 1,000 litres of water is added. A second quantity of the diazo compound from 21.5 kilos. of sodium naphthionate, 50 kilos. of hydrochloric acid, 1,000 litres of water, and 7 kilos. of sodium nitrite in 10 per cent. solution, is then run in and the mixture is stirred for 24 hours. As the diazo dyestuff forms, the red solution of the azo dyestuff turns blue; and when a sample, after acidifying, on boiling no longer gives off nitrogen, the colouring matter is precipitated with salt, filter-pressed, and dried. The dyestuff thus obtained forms a bluish-black powder, which dissolves to a blue solution in water.—T. A. L.

*Improvements in the Manufacture of Alpha-beta-dimethyl-eurhodines.* J. Y. Johnson, London. From "The Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 18,925, October 9, 1893.

ACCORDING to Eng. Pats 10,138 and 10,138A of 1892 (this Journal, 1893, 593 and 594), of which this an extension,  $\alpha$ -methyl-homo-eurhodine is prepared by the interaction of an amido-azo compound of *p*-toluidine and methyl- $\alpha$ -naphthylamine, and is subsequently converted into the dimethyl-eurhodine by alkylation. It has now been discovered that both alkyl groups may be directly introduced into the non-alkylated eurhodines by the prolonged action of an alkyl iodide. Thus homo-eurhodine,  $C_{17}H_{15}N_3$ , when treated with methyl iodide, yields a mixture of the hydriodides of  $\alpha$ -methyl-homo-eurhodine,  $\alpha$ - $\beta$ -dimethyl-homo-eurhodine, and unaltered homo-eurhodine. After basifying, the mixture is again treated with methyl iodide, and this process is repeated until the product consists only of dimethyl-homo-eurhodine. This is practically carried out as follows:—About 26 kilos. of homo-eurhodine, 30 kilos. of methyl iodide, and 150 kilos. of methyl alcohol, are heated for four hours in an enamelled autoclave to 120° C. The melt is then diluted with water, the precipitate filtered off, and the product is treated in a fine state of division with 200 kilos. of alcohol and 42 kilos. of caustic soda-lye containing 28 per cent. NaOH. Then precipitate with water, wash, press, and dry, and again treat the dry residue with 30 kilos. of methyl iodide and 150 kilos. of methyl alcohol for four hours at 120° C., and work up as before. If the product still contains appreciable quantities of eurhodine and  $\alpha$ -methyl-eurhodine the treatment must be repeated. Their presence may be detected as follows:—The product is dissolved in just sufficient hydrochloric acid and carefully neutralised with ammonia, when any eurhodine, or methyl eurhodine separates as a yellow precipitate, whereas the dimethyl derivative remains in solution. The crude product obtained is boiled with 1,000 litres of water containing 0.25 per cent. of hydrochloric acid, and the filtered extract is precipitated with salt. In order to further purify it, it is dissolved in water, ammonia is added in slight excess, and after stirring half an hour the precipitate is filtered off and salt is added to the filtrate to throw down

the  $\alpha$ - $\beta$ -dimethyl eurhodine hydrochloride, which is filter-pressed and dried. The lower homologue from the eurhodine,  $C_{16}H_{13}N_3$ , can be obtained in a similar manner.

—T. A. L.

*Processes for the Production of Dyestuffs.* A. Bang, Leeds. From G. A. Dahl, Barmen, Prussia. Eng. Pat. 18,958, October 10, 1893.

BLUE to violet dyestuffs are obtained by the action of di- $\beta$ -naphthyl-*m*-phenylene diamine sulphonic acids on the nitroso derivatives of alkylated aniline or toluidine, or on certain amido-azo compounds and their sulphonic acids. A solution of 56.1 kilos. of sodium di- $\beta$ -naphthyl-*m*-phenylene diamine disulphonic acid in 500 litres of water, cooled to 5°–10° C. and acidified with 20 kilos. of 30 per cent. acetic acid, is slowly mixed with 28 kilos. of nitroso-dimethyl-aniline hydrochloride. When the nitroso compound has disappeared, the solution is neutralised with soda and the dyestuff salted out. It dyes wool deep blue from an acid bath. A similar dyestuff is obtained by heating together to about 90–95° C. a mixture of 49 kilos. of sodium dimethyl-amido-azo-benzene sulphonate, 58 kilos. of sodium di- $\beta$ -naphthyl-*m*-phenylene diamine sulphonate, 600 litres of water, and 20 kilos. of 30 per cent. acetic acid for about four hours. The solution is neutralised with soda, and the dyestuff separated by adding salt.—T. A. L.

*Improvements relating to the Production of Pyrocatechine.* J. H. Baum, Frankfort-on-the-Maine, Germany. Eng. Pat. 21,853, November 15, 1893.

PYROCATECHOL is produced from  $\alpha$ -phenol disulphonic acid by fusing it with about an equal weight of caustic soda and hydrolysing the pyrocatechol sulphonic acid formed by heating to a high temperature with 25–30 per cent. sulphuric acid. About 10 kilos. of sodium  $\alpha$ -phenol disulphonate are fused with 10–15 kilos. of caustic soda in an autoclave for 8–10 hours at a temperature of 280–300° C. and a pressure of 2–3 atmospheres. The melt is dissolved in dilute sulphuric acid, neutralised with chalk, evaporated, cooled, and filtered, and the mother-liquor is heated in an autoclave with 10 kilos. of 50 per cent. sulphuric acid to 180–220° C. Steam is then blown through the solution obtained until all the phenol is removed, and after having been decolorised with animal charcoal the pyrocatechol is extracted with ether. The yield is said to be 50 per cent. of the theoretical.—T. A. L.

*Manufacture of Polyoxanthionine Colouring Matters Dyeing on Mordants.* O. Imray, London. From "The Society of Chemical Industry in Basle," Basle, Switzerland. Eng. Pat. 12,214, June 23, 1894.

THE nitroso derivatives of 2,3'- and 2,2'-dihydroxy-naphthalene on treating with sodium bisulphite and hydrochloric acid, are converted into amido-dihydroxy-naphthalene sulphonic acids, which on oxidation with nitric acid yield hydroxy-naphthoquinone sulphonic acids. Colouring matters are obtained from these compounds by heating them together with the thiosulphonic acids of aromatic *p*-diamines in acetic acid, or the nitroso derivatives of the secondary or tertiary aromatic amines may be condensed with the hydroxy-naphthoquinone sulphonic acids in presence of thiosulphates. Nitroso-dihydroxy-naphthalene obtained from 400 kilos. of 2,2'-dihydroxy-naphthalene is stirred into 2,000 kilos. of a solution of sodium bisulphite or 36° B. at 20–30° C. The solution is then filtered, made acid with hydrochloric acid, heated to 50° C., and left to stand for 10 hours. The 1,2,2,4'-amido-dihydroxy-naphthalene sulphonic acid separates out and crystallises from water in needles. By oxidation with 30 to 60 per cent. nitric acid, it yields hydroxy-naphthoquinone sulphonic acid, of which the potassium salt can be precipitated by adding potassium chloride to the nitric acid solution. The formation of a dye-stuff therefrom is illustrated as follows:—A concentrated solution of 7 kilos. of sodium nitrite is

mixed with 107 kilos. of a 28 per cent. solution of methylbenzyl-aniline sodium sulphate acidulated with 21 kilos. of hydrochloric acid of 21 B. After standing 12 hours, 35 kilos. of 40 per cent. acetic acid and a solution of 50 kilos. of sodium thiosulphate are added, the whole being then warmed to 80—100 °C. until the nitroso compound has disappeared. A concentrated solution containing 30 kilos. of the potassium salt of 2'.1.2.4-hydroxy-naphthoquinone sulphonic acid is then added, and the whole is heated for 2—3 hours. The new dye-stuff separates from the dark-coloured liquid as a green crystalline powder. It dyes wool mordanted with chromium salts from an acid bath a pure bright blue, fast to light, washing, and fulling.—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Treatment of Tussah Silk before, during, and after Dyeing.* H. Silbermann. *Färber Zeit.* 5, 1894, 198—201, 216—217, and 234—236.

See under VI., page 880.

*The Influence of Weighting on the Specific Gravity of Silk.* Chem. Zeit. 18, 1894, 744.

See under XXIII., page 907.

*Commercial Cellulose Derivatives.* C. Beadle. Chem. Trade J. 1894, 128.

See under XIX., page 900.

## PATENTS.

*Improvements in Apparatus for Gassing Yarn and other Threads.* S. Clough, Steeton, Yorks. Eng. Pat. 10,736, June 2, 1894.

The invention consists in an improved burner with small apertures in the side, so placed that the flame is emitted horizontally. The thread thus passes through the whole length of the flame, the singeing dust is blown away, and can neither fall into the burner nor adhere to the yarn.

—R. B. B.

*Improvements in or appertaining to Apparatus for Sizing Yarn.* J. Maag, Winterthur, Switzerland. Eng. Pat. 11,013, June 6, 1894.

A saving in cost is effected by applying the size direct in the cold condition, instead of boiling it before use. To prepare the yarn for sizing, the threads from the warp rollers pass directly to a receptacle, in which they are subjected, by means of an injector or otherwise, to a current of moist air. Thence they are led into the size-box, excess of size is removed by squeezing-rollers, and the threads pass into the upper part of a drying chamber, where they meet with air of moderate dryness and warmth; by means of rollers they take a circuitous course, passing gradually downwards, encountering hotter and drier air as they descend, and leaving the chamber close to the source of heat. To separate threads which are stuck together, they are led to a set of polished rollers arranged like a furnace-grating; the threads pass alternately above and below these rollers, and are finally wound on a common warp-beam.

—R. B. B.

*Improvements in the Process of and Apparatus for Scouring and Degreasing Wool.* W. E. Heys, Manchester. From G. Malard, Paris, France. Eng. Pat. 11,754, June 18, 1894.

The principal object of this patent is to obtain the solution resulting from wool-scouring in a state of higher concentration than is usual at present. The machine consists of a long travelling apron, underneath which are a number of tanks containing the scouring liquid. The liquid is pumped from these tanks on to perforated distributors, which shower it down upon the wool. It falls into a trough or shoot, so pivoted between two compartments that the liquid may be allowed to fall either into the compartment from which it was pumped or into the next. The fresh scouring liquor is led into the tank nearest to the delivery end of the apron, and the liquor increases in density passing towards the feed end. A portion of the liquid pumped from the tank at the feed end is diverted from the distributor into a small box in which floats a hydrometer. When the liquid reaches a certain density the hydrometer in rising closes the contact of an electric circuit, and rings an electric bell. The attendant then operates a rod, which turns the shoot above the last tank, so that the liquid passes into a special tank for receiving concentrated liquor; and at the same time, by means of a float in the last tank, the next shoot diverts liquid from the last but one into the last, and so on; fresh liquid from a supply vessel filling up the emptied tank at the delivery end. When the electric bell ceases, the attendant reverses the rod, and the liquid again flows through the wool into the vessel whence it was pumped, until the required density is once more attained.—R. B. B.

## VI.—DYEING, CALICO PRINTING, PAPER-STAINING, AND BLEACHING.

*Treatment of Tussah Silk before, during, and after Dyeing.* H. Silbermann. *Färber Zeit.* 5, 1894, 198—201, 216—217, and 234—236.

The tussah silk does not contain silk-glue encircling the individual fibres and binding them together, but the whole fibre is impregnated with the bast; the same applies to the colouring matter, which is not separate as in real silk, but penetrates through the whole fibre. The raw tussah fibre contains 5 to 6 per cent. of mineral compounds, which are reduced to 0.25 per cent. by removing the bast. These salts consist of potash, soda, silicates, carbonates, and sulphates of lime, magnesia, and alumina. The organic constituents of the bast consist largely of oxidised tannin compounds in combination with iron, which are of a brownish colour; a lime salt, which is precipitated by a tannin solution in the presence of acetate of soda; and a wax-like fat of an acid character (about 1 per cent.), which can be easily extracted with alcohol. The acid character of the bast renders it necessary to employ alkaline chemicals for its removal, while a neutral soap answers the purpose with genuine silk. The alkaline treatment with potash, soda, and ammonia was early adopted in Europe (Riot, Eng. Pat. 1609, 1855). The following successful method of removing the bast is now adopted:—

*Removal of Fatty Substance or Stripping.*—This treatment consists in removing from the fibre the fatty and gummy matters and also a certain quantity of the natural colouring matter. The cocoons are softened in boiling water before unreeling, and the cleansing bath contains crystal soda in the proportion of 10 per cent. of the weight of the silk with 20 to 30 times as much water. Commencing with tepid water, it is gradually raised during a quarter of an hour to 40° C., the tussah being agitated, first quickly and then slowly, until the bath assumes a dirty-brown colour, when it is run off. After half an hour's agitation the silk is removed and freed from water by

wringing or by centrifugal machines, losing by the treatment about 8 to 10 per cent. of its raw weight. For certain purposes, as insulating telephone wires, &c., it is unnecessary to remove the bast entirely from the tussah, simply washing in three baths of tepid water to free the fibre from fat and dirt, drying and stretching being sufficient.

**The Boiling off.**—A special method for a thick thread tussah is as follows:—After removing the fat, the silk is put in a fresh bath of 30 per cent. of crystal soda at 10° to 15° C., and raised during half an hour to 85° to 90° C., at which temperature it is boiled for 1 to 1½ hours. After running off and thoroughly washing, it is, with the usual precautions, treated in a boiling bath of 10 per cent. fuming hydrochloric acid for about 10 minutes. It is a fact that part of the brown colouring matter, which at 60° to 65° C. is removed from the bast, fixes itself upon the fibre when the temperature reaches 80° to 90° C., and the boiled tussah becomes a deeper shade of brownish-yellow by the action of the acid.

By this treatment (a better one for fine silks, though more costly) the fat is removed in a soda bath (10 to 15 per cent. crystal soda), heated to from 50 to 60° C. The silk is then put into a boiling soap bath and well agitated without further heating. After three-quarters of an hour the bath is again raised to 95° C. For silk of fine fibres less soap is required; generally about 30 per cent. of good olein soap suffices. The removal of the bast is performed in about 1 to 1½ hours. If the slightest quantity of soap remain on the fibres they will have a greyish tint. Careful testing and observation show that the employment of soap gives a more favourable result with regard to the strength than soda, while the latter produces a fibre with greater elasticity. Where strength and elasticity have to be considered a mixture of soap and soda is therefore preferable. A solution of sodium hydrate, 6 to 8 per cent. of the weight

of silk, removes the bast in about half an hour at 60° C. The fibre, after boiling, has a high gloss, and is only slightly affected in strength and elasticity, but it has to be treated quickly and with care, otherwise the lye has a deleterious effect. The addition of soap to the lye is not so advantageous as to submit to soaping after boiling. The soap used for thus removing the bast, is 3 to 5 per cent. of the weight of silk, allowing for the hardness of the water employed, and the time, about half an hour at 35° to 40° C.; after this the material is well washed.

The accompanying illustrations represent microscopical views of the fibres of real silk (Fig. 1), of Indian (Fig. 2), and China tussah (Fig. 3). In each *a* indicates the raw fibre covered with the bast, *b* indicates the cleaned fibre, *c* a section of the raw, and *d* of the silk cleansed from bast.

**The Samingor Acid Treatment.**—This operation consists in treating the tussah in an acidified water bath mainly to remove any mineral matter remaining.

The bath is used at about 30° to 40° C. for soaped tussah, and at boiling temperature with 10 per cent. fuming hydrochloric acid for fibre that has been treated with soda. The strength of the fibres is weakened somewhat by this operation, while the elasticity is increased about 10 per cent. China tussah of European spinning (China Filatur) is by the boiling acid treatment improved both as to strength and elasticity, probably because it contains comparatively little inorganic compounds, while Indian and ordinary China tussah contain a larger percentage. The latter silk by treatment with hydrochloric acid is freed from these mineral ingredients, and naturally becomes weaker as to textile strength, though the elasticity is increased by the removal of these brittle salts.

The bleaching of tussah silk, like that of other textile fibres, consists in oxidising the natural colours with the formation of colourless derivatives.

Fig. 1.

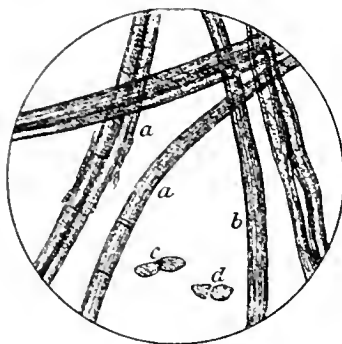


Fig. 2.

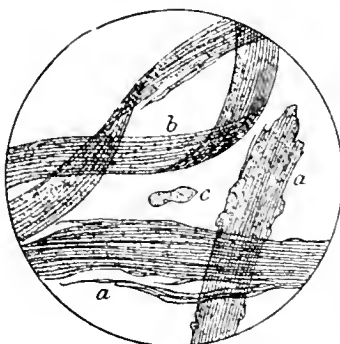
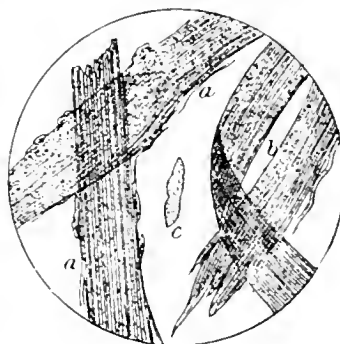


Fig. 3.



The results obtained by all previously devised and tedious operations were no better than the present method of using simply hydrogen peroxide. To hasten the bleaching it was proposed to saturate the fibre with turpentine oil and expose it to the light for the generation of ozone.

An English method (Eng. Pat. 1414, 1878), consisting of the use of hydrogen peroxide mixed with ammonia and ammonium hypochlorite, confirms the advisability of using a hypochlorite salt. A chlorate salt has also been recommended. For 100 kilos. tussah there is prepared, for example, a bath of 500 litres of 3-volume hydrogen peroxide, 5 kilos. ammonium carbonate, 5 kilos. potassium chlorate, and 3 kilos. sodium nitrite. The fibre is left in the bath 12 hours, dried by a current of air at 20° to 25° C., returned to the same bath another 12 hours, and then dried.

The present methods of employing hydrogen peroxide with the addition of water-glass, soda, ammonia, &c., are worth mentioning. For 100 kilos. tussah a bath is prepared of 500 litres of 3-volume hydrogen peroxide (or, for example, 150 litres of 10-volume hydrogen peroxide), 20 litres ammonia of 22°, and 2 to 3 kilos. sodium hydrate. The treatment takes 24 hours; the material is then washed, and, if

necessary, returned to a warm bath. The quantity of caustic employed is in proportion to the acidity of the commercial hydrogen peroxide.

Another method is to use 150 to 160 litres of 10-volume hydrogen peroxide, 3 kilos. potash, 5 kilos. ammonium carbonate, and a quantity of ammonium hypochlorite obtained by decomposing 2 kilos. ammonium sulphate with calcium hypochlorite ( $\text{CaCl}_2 \cdot 2\text{Ca}(\text{ClO})_2$ ), from which the calcium chloride is removed by Glauber's salt.

In some works the bleaching is first performed with peroxide of hydrogen with the addition of water-glass, followed by an aqueous solution of sulphurous acid, or the gas itself.

The bleaching action of the peroxide of hydrogen is increased if the tussah, without being washed, is hung up in the air for several hours, whereby the evaporation concentrates the bleaching liquor, making it more effective, and the air and light give rise to the formation of ozone.

A peculiar but effective operation consists in bringing the peroxide of hydrogen and alkali together upon the fibres while suspended in the air. This is done by impregnating the fibres with hydrogen peroxide and then suspending them in a room hermetically sealed, and upon the floor of which vessels are placed containing ammonia. The material is

hung for 12 to 24 hours and the operation repeated until sufficiently bleached. The fibres appear to be attacked less by this treatment than by bleaching in a bath.

Another process consists in impregnating the fibre with concentrated peroxide of hydrogen and then steaming under pressure, by which means the bleaching is effected in an hour or two. Zinc-dust or a weak electric current is occasionally employed to hasten the bleaching, and powdered sodium peroxide instead of hydrogen peroxide has been recommended, and will probably be more generally adopted. (Compare *Färber Zeit.* 1891-92, 263; this *Journal*, 1892, 814; 1893, 329.) Cheaper oxidising agents than hydrogen peroxide, such as potassium permanganate, bromine water, &c., do not give satisfactory results, besides attacking the fibres somewhat strongly.

Electrolytic and electro-ozone processes may possibly have a great future before them as a means for bleaching tussah silk. Between tussah and genuine silk there are two important points of difference, which largely influence the dyeing of the fibres. Tussah has a much smaller affinity for colours than genuine silk, and differs considerably in its physical structure. Whilst real silk takes the dye very easily, the tussah shows a much less power of absorbing it—a property in which it resembles most vegetable fibres. The difference is also seen in its solubility in cuprammonium solutions, zinc chloride, hydrochloric acid, &c., as compared with the mulberry silk; and also in its power of resisting chemical agents in general. To dye tussah fibre it is necessary that the bath should be hotter and contain more acid than with real silk; it should be introduced at 60° to 70° C., and finished at the boiling temperature. Permanent colours are used when possible, and azo colours, diamine colours, &c., have recently been introduced for dyeing tussah, with success. With diamine colours the dyeing is performed in pure water with the addition of common salt and Glauber's salt, while soda and soap are employed for the diphenyl colours. Light and clear shades are generally produced with the usual colours; all the rose shades, from the most yellow to the most blue, in a weak soap bath with rhodamine and phosphine, or rhodamine with alkali violet; all the light blues with alkali blue, &c.

Other kinds of tussah silk, as the Indian *Eria* silk of *Attacus ricini* behave in the dyeing operation similar to tussah, while some, as the Moonga silk of *Antheraea assama* behave like genuine silk. The most difficult colours to dye tussah fibre are the lightest and darkest. Although both the raw and the cleansed fibre appear to be uniform in texture, its irregular and uneven structure shows itself after bleaching. Even in the same skein different parts are bleached and dyed with varying degrees of intensity. The light shade which the tussah has after bleaching is due to oxidised derivatives of the natural colouring matter, which cannot be removed with a stronger bleach without injuring the fibres. Sometimes a short, energetic, hot boiling in a soap bath after the bleaching, has a good effect, but this soaping affects the fibres, and should not be continued more than 10 to 15 minutes. With light colours, as white, sky-blue, &c., there is a tendency to become striated, but with cream and pink shades it is not so to the same extent, as the yellowish shade of the bleached tussah has less effect upon creams and pinks. It is therefore preferable to employ a soap bath in dyeing light shades, of course using basic dyes and the alkali colours, such as may be employed with neutral or alkaline soap, alkali blue, neutral diamine dyes, or the more alkaline, substantive tetrazo colours.

A difficulty in dyeing tussah lies in the fact that the fibres never take the darkest shades—as marine blue—sufficiently deep, owing to the great resistance the fibre offers to colouring matter. The fibres of tussah silk are not cylindrical like genuine silk, but are flattened, and the substance of which they are composed is not homogeneous like the structureless fibre of the mulberry silk. Tussah silk consists of a number (15 to 25) of fine fibrils running parallel and bound together in bundles, a structure which is distinguished under the microscope, and gives the surface of the tussah an uneven striated appearance. The beautiful gloss peculiar to genuine silk is absent in tussah. The method of dyeing tussah black is similar to that em-

ployed for real silk, and being rather important and difficult a few details may be given. The tussah fibre is treated for several hours with basic sulphate of iron mordant at 30° B., washed twice, and then with a catechu bath (20 to 25 per cent. catechu), to which is added some fustic extract. A catechu bath is very beneficial to genuine silk, but great care must be observed with tussah that the temperature does not exceed 65° to 70° C., otherwise the catechu leaves the fibres and returns to the bath. This temperature must be retained for about three hours. After washing, it is dyed with logwood and soap at 55° to 70° C. for two to three hours, with the addition of gelatin, and cleared with acetic acid. Another method for blue-black is to treat the fibre for an hour with iron mordant, rinsing, mordanting for two hours in an alum solution of 4° to 5° Baumé, rinsing, and dyeing in a bath (for 10 kilos. tussah) of 6 to 7 kilos. logwood 300 grms. fustic, and 2 kilos. soap.

For a deep, full black, although more costly, the following method may be used:—Mordant during six hours in an iron mordant of 30° Baumé, soaping, blueing (with 12 per cent. yellow prussiate of potash and 9 per cent. hydrochloric acid), washing, treating in an alum bath of 5° Baumé, washing, treating with ferrous sulphate 24 per cent., and dyeing in a boiling vat containing 60 per cent. logwood, 15 to 25 per cent. fustic (according to the shade), and 25 per cent. soap. To finish, one can employ with success the method adopted for *seru* silk, that is, alternately treating with chestnut extract and iron pyrolignite until the desired depth of blue colour is obtained.

With each of these methods it is an advantage to treat the tussah before dyeing with a weak solution (according to the strength of the fibre, 3° to 1½° Baume) of hydrofluoric acid. Hydrochloric acid may be used, but it gives inferior results. This treatment removes the inorganic compounds of the tussah which resist the mordant. The finished dyed tussah, like Schappe and organzine, is brightened and cleared with oil—olive oil for fine tussah fibre, or cheaper vegetable oils may be used if made into an emulsion (by well shaking with carbonate of potash lye) and added to a tepid acidified clearing bath. The bath is well agitated and the tussah drawn quickly through for about five minutes, and, after winding, freed from water in a centrifugal machine. The quantity of oil used, is according to the silk and the purpose for which it is to be employed; more is used for throwu silk than for *tram*, generally 5 to 8 per cent.; whereas for *grège* rarely more than 3 per cent.

—S. P. E.

#### *The Use of Benzidine Colours in Discharging Indigo.* G. Stein. *Färber Zeit.* 5, 1893-94, 269.

MANY of the benzidine colours can be used in producing coloured discharge prints on dyed indigo cloth, both by the chromate and acid process and by the ferriyanide and alkali process, the latter giving the better results. Brilliant Geranine B, Chloramine yellow, and Chrysophenine may be applied in the acid way; whilst in the alkaline process Chloramine orange, Brilliant Geranine 3 B, Heliotrope 2 B, the Congo and Mikado oranges, and the Benzo-browns may also be used. In applying the alkaline process, the colour may be added to a discharge paste containing potassium ferriyanide, and the discharge be effected by a passage through caustic soda solution; or the colour may be mixed with the ferriyanide and carbonate of magnesia, and the discharge effected by one hour's steaming. The process may be varied by dyeing all over with indigo and a benzidine colour and discharging in the acid or alkaline way. Thus with Chloramine orange an orange print on a black ground is produced; with Congo orange G an orange figure on a bronze ground, &c.—W. E. K.

#### *Resists with Tannin Mordants.* Ch. Casanovas. *Färber Zeit.* 5, 1894, 233-234.

THE following operations are suitable for this method of treatment:—(1) Printing the resists and drying; (2) blocking in the tannin and drying; (3) fixing the tartar emetic and washing; (4) dyeing with basic coal-tar colours,

washing, bleaching, and drying. The best means for resisting appears to be tartar emetic (potassium antimonious tartrate), but the tannin-antimony lake fixes itself more or less upon the fibres and afterwards colours them. Owing to the tannin, after blocking, penetrating the fibres before the tartar emetic has time to act as a resist, the parts which should remain white take the dye, and the subsequent soaping and bleaching is ineffectual to make them perfectly colourless. The resist of Prud'homme for steam aniline dyeing, by adding common salt to the resist to obtain a more concentrated tartar emetic, does not give a better result, besides being very hygroscopic, and in the tannin blocking running and not remaining clear and distinct, especially when treated in the "hot-blue." The easily-soluble resist thus gets on the crown of the printing roller, and ultimately comes off.

Magnesium acetate does not give a good result; the resisting parts are, after dyeing, very much coloured. Basic antimony oxalate used as a resist gives a better result than the means proposed by Jaquet, viz., to mix directly with tannin steam-dyes. Freshly precipitated and concentrated antimony hydroxide does not resist. Kertesz proposed to fix the tannin steam-dyes by means of freshly-precipitated antimony oxide. Zinc salt does not appear to resist for steam aniline colours any better than tartar emetic. More suitable than zinc salt and tartar emetic is the antimony salt of de Haen, which, together with tannin, could be used for cloth and calico were it not so expensive.

A process devised by Lamy and Kopp consists in the treatment with iron mordant, dunging with cow-dung, then treatment in sumac, and afterwards dyeing with basic coal-tar colours. This process can also be used as a resist for printing under or upon an iron mordant; the parts thus treated for resisting subsequently take no colour when dyed with aniline colours; the treatment is well known and satisfactory.

Alumina throws down the tannic acid, and with this mordant lighter shades can be dyed, such as lilac, rose, &c.; the iron, however, produces cloudy effects by forming a lake with the tannic acid. It is said that English goods, although dyed twice, are cheap, but they are not all one could desire in a genuine article.

Blocking in the tannin (20 grms. per litre), treating with tartar emetic (5 grms. per litre), and dyeing in a bath with an iron salt would give the same shade. The result of this method of resisting is satisfactory, and if in the dyeing with tannic acid a little is fixed upon the white parts, these will be restored to their natural whiteness by the chlorine treatment after the second dyeing. Treating with tartar emetic or steaming after dyeing does not improve the result. Owing to the difficulty of producing white with tartar emetic resist, the method of Koehlin frères with caustic soda was introduced, by means of which a beautiful product is obtained. Still the strong caustic lye which is employed renders the fibre liable to the danger of acquiring affinity for the dye and producing an imperfect white. Relying upon the Davy method of estimating tannin quantitatively, and upon the improved modification of Ichberg for titration of the tannic acid, the author has proposed the following as a suitable resisting agent:—900 cc. of thickening of British gum 20 grms. of gelatin, dissolved in 100 cc. of water and added to the thickening, to which are added, cold, 80 cc. of nitrate of alumina of 15 Baumé.

Stamping, drying, blocking in a somewhat concentrated solution of tannin, fixing with tartar emetic, and dyeing with aniline colours and washing follow. (More gelatin may be used, but it is better to boil the gelatin until it ceases to run when cold; it precipitates the tannin.)

In this manner a perfect result is obtained, and the same as with aniline colours that are fixed with tannin and tartar emetic.—S. P. E.

*Cotton-dyeing with Benzidine Colours.* J. Mullerus. *Färber Zeit.* 5, 1894, 234.

In the *Chem. Zeit.* 31, 1894 (this Journal, 1894, 124—125), an interesting article appeared in which Dr. C. O. Weber speaks of the possibility of obtaining an organic lake-forming compound which would furnish a means for

dyeing with benzidine colours upon cotton fibres as with wool. Weber says he has not succeeded, after many attempts, in obtaining with egg albumin or blood albumin lanugine acid, or with an insoluble salt of the sulphonic acid of a dye, the desired lake-forming amine or amido-acid. Weber does not appear to have experimented with albumin alone; he only mentions that the blood albumin, at least in a coagulated form, has the property of fixing sulphonic acid colours. As the fastness in cotton-printing with albumin-fixed benzidine and diamine colours is so striking, the idea that the function of the albumin is only that of an indifferent mordanting agent, is conceivable. Weber's supposition seems to endorse this view. For three years the author has employed blood albumin with sulphonic dyes for the production of colours resistant to light, with "Rouge de St. Denis." Before dyeing he prepares the cotton in a bath containing 10 per cent. of blood albumin 1:1 and 10 per cent. Turkey-red oil of 60 per cent., drying and dyeing as usual. The stuff thus prepared takes the colour better and is fuller, and stands water and light well. It is noticeable that by the use of albumin solution alone, even with double the quantity, an inferior result is obtained.—S. P. E.

*The Influence of Weighting on the Specific Gravity of Silk.* H. Silbermann. *Chem. Zeit.* 18, 1894, 714.

See under XXIII., page 907.

#### PATENTS.

*Improvements in Apparatus for Dyeing Slubbing or Slivers of Fibrous Material.* J. Rhodes and A. E. Perkins, Bradford, Yorks. Eng. Pat. 13,379, July 13, 1893.

THE object of this invention is to uniformly expose the material to the dyeing liquor, without treatment liable to derange the fibres. The apparatus consists of a tube or series of tubes, each tube being as long as the height of the building will allow. The tubes are arranged in pairs over vats containing guide rollers, and the dye liquor, as well as the material, passes down one tube into the vat, and rises in the second tube, at the top of which the slivers pass between squeezing rollers, and may then be carried through one or more further pairs of tubes, similarly supplied with dyeing liquor, to complete the dyeing operation. The slivers are attached to an endless flexible apron. The flow of the liquid through the tubes may be either in the same or the reverse direction to that of the material; or the liquid may be stationary in the tubes during the passage of the material.—R. B. B.

*Improvements in Machinery for Washing and Scouring Wool, Worsted, Yarn, Slubbing, and other Fibres.* Brintons, Limited, and L. Hanson, Kidderminster. Eng. Pat. 14,865, August 3, 1893.

THE object of the invention is to construct a self-acting washing and scouring machine without the use of reciprocating forks. The materials are carried along on a series of endless aprons; below each apron is a vessel containing the washing liquid, which is pumped from here into a shallow vessel, with a perforated bottom, above the apron, and thence showered down upon the material. A series of four or more such machines should be in use together, the wool being treated with the dirtier scouring liquor from previous lots in the first machine, and with fresh clean water in the last. At the delivery end of each apron the wool passes between squeezing rollers.—R. B. B.

*Improvements in the Process for Producing Fast Colours from Direct Dyes on Cotton.* O. Imray, London. From The Farbwerke vormals Meister, Lucius, and Brünig, Höchst-am-Main, Germany. Eng. Pat. 15,326, August 11, 1893.

A GREAT defect of many "direct cotton" dyes is their want of fastness to washing, and the readiness with which they



bleed on to the undyed portions of the fibre. According to this patent, the colours are made fast to soap by treatment of the fibre, after dyeing, with a solution of a chromium salt, preferably at the boil. The colouring matters most suitable for this treatment are those simple and mixed azo colouring matters which dye directly on cotton, and which contain as components salicylic acid, ortho- and meta-cresotic acid (hydroxy-toluic acid),  $\alpha$ -hydroxy-naphthoic acid, or dihydroxy-naphthalene-mono- and disulphonic acids derived from 1,8-dihydroxy-naphthalene.

Basic chromium salts are best suited for this purpose. A nearly complete formation of the chrome-lake will be obtained, for example, by passing the goods for a few minutes through a boiling solution containing 0.5 gram. of basic chromium chloride per litre. The following is an example of the manner of carrying out the invention:—

*Fast Yellow on 20 kilos. of Cotton Thread.*—The previously well-boiled thread is boiled for three-quarters of an hour in a bath containing:—

0.8 kilo. cotton yellow G.  
2 „ sodium phosphate.  
0.2 „ soap.  
2 „ common salt.  
600 litres water.

It is then taken up, left to cool, well wrung, and worked for half an hour at the boil in—

600 litres of water,  
0.5 kilo. basic chromium chloride;

after which it is slightly rinsed and, if necessary, soaped.

—R. B. B.

*Improvements in Apparatus for Treating Fibrous Materials, Yarns, or Threads with Mordanting, Preparing, Dyeing, or other Liquids.* T. P. Miller, Cambuslang, Lanark. Eng. Pat. 16,038, August 25, 1893.

THE fibrous materials in an unspun condition are placed on an endless web of cotton cloth, and carried through a long trough containing the liquid. At the middle part of the trough there are a number of beetles, which are lifted by reciprocating metal pieces, but drop by their own weight on to the materials. By means of a ratchet-wheel feed, the endless web, with the materials on it, is moved forward at each ascent of the beetles, and, after passing under these, carries the materials between a pair of squeezing rollers at the delivery end of the trough.—R. B. B.

*Improvements in Dyeing Wool with Sulphonic Acids of Anthraquinone Colouring Matters.* O. Imray, London.

From The Farbwerke vorm. Meister, Lucius, and Brüning, Höchst-am-Main, Germany. Eng. Pat. 17,544, September 18, 1893.

A FURTHER application of the process described in Eng. Pat. 15,310 of 1892 (this Journal, 1893, 825). The wool is dyed in an acid bath with the sulphonic acid, and subsequently converted into colour-lake by treatment with metallic salts. The sulphonic acids employed may be those not only of alizarin, but also of anthraquinone colouring matters containing more hydroxy-groups, and of their nitro-, amido-, &c., derivatives. The method of dyeing is fully described in the previous patent.

The advantages claimed are: (1) only one bath is required; (2) greater uniformity of shade, more thorough dyeing, and shades faster against rubbing; (3) the possibility of employing the alizarin dyes in new branches of the industry.—R. B. B.

*Improvements in Rotary Washing and Dyeing Machines.*

J. W. Crabtree, Bradford, Yorks. Eng. Pat. 19,769, October 20, 1893.

THE machine consists of a fixed outer cylinder and an inner cylindrical cage, which is revolved alternately in opposite directions, and which is partly solid and partly perforated, the perforations being narrow slots cut in the

direction of rotation. Within the inner cage are a number of U-shaped longitudinal chambers, one half of the U being perforated, the other half solid. During the rotation of the cage these chambers carry liquid from the bottom, and when near the top discharge it in a spray on the fabrics. The chambers are placed with the perforations in opposite directions, so that half of them work in each direction of rotation. Inside the cage also, a number of perforated pipes are arranged for the introduction of steam in a series of fine sprays.—R. B. B.

*Improvements relating to Dyeing Wool with Chromium Lakes.* E. Hoffmann, Leipzig-Lindenau, Germany.

Eng. Pat. 11,077, June 7, 1894.

THE dyestuffs are applied in a single bath, together with the chromium salt of an organic acid, which may be tartaric, citric, oxalic, succinic, lactic, benzoic, salicylic, or malic acid. Or the organic salt is mixed with some other chromium salt, e.g., the chloride, sulphate, nitrate, fluoride, acetate, &c.—R. B. B.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Notes on the Manufacture of Sulphuric Acid in America.* G. Lunge. Zeits. angew. Chem. 1894, 133—137.

THE growing demand for superphosphates has favourably reacted on the American sulphuric acid industry, and it is stated that the yearly output of acid is represented by a money value of some 6½ million dollars. The works producing the same are as a rule well erected and compare advantageously with similar European establishments.

Very little can be said about the construction of the chambers. At one of the works the author noticed a set of 12 chambers each only 8.2 metres long, and here the chamber space required and the nitre consumption were said to be very low. At another works a set of three chambers was erected, 18, 15, and 12 metres long respectively, with a plate tower inserted between each chamber. This arrangement is, however, not at work yet, and no details can be given as regards the efficiency of the plate tower in this position.

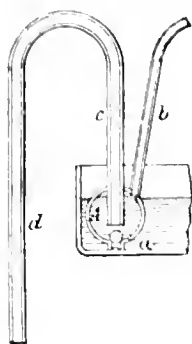
Among pyrites-kilos that of Speuce, although labour-saving, is said to be too troublesome to work. Johnson's kiln (similar to McDougall's) receives greater praise. But in the author's opinion the Frasch kiln is the best apparatus hitherto constructed for roasting pyrites. Falding's improvement (this Journal, 1893, 1032) is also remarkable; here the bricks are hollow and form channels for the circulation of air, which keeps the brickwork cool, and the heated air may be afterwards passed below the bars.

The draught is produced by means of fans of hard lead, or of wood covered with lead, fixed on rotating iron shafts, which run in lead bearings, without stuffing-boxes. They are driven by electromotors and are greatly to be recommended.

A siphon of very practical construction may be mentioned here. Its shorter leg *c* opens into a bulb *A*, which is provided on the bottom with a short exit-tube closed by means of a glass ball *a*. When setting the siphon, air is blown into the tube *b*, the acid rises into the tube *c*, and overflows from *d*. From this moment the siphon becomes self-acting and the acid enters through *a*.

The concentration of sulphuric acid is chiefly effected in platinum vessels, often in combination with cast-iron vessels, which latter are either retort-shaped or provided with a leaden lid cooled from outside (as proposed by Faure and Kessler). The gold-lined platinum vessels of Heraeus are extensively used, and are said to be a success when working with not too impure an acid. Where first cost is a consideration, cast-iron retorts are used, either wholly or in combination with platinum vessels. Thus, at one of the works the acid is concentrated up to 93.5 per cent. monohydrate in platinum vessels, and from this point up to

98 per cent. in round iron vessels of about 0.9 metre diameter and made of 5 cm. metal. The life of such a pan is about two months, and but little iron goes into solution.



Another evaporating plant consists of two platinum pans for concentrating up to 166° Tw., and of an iron pan for 169° Tw. acid. This acid is consequently somewhat impure, and is used for purifying oils. The heating is effected by means of liquid fuel. The waste heat passes over other pans and concentrates there acid from 116° Tw. to 146° Tw. It is stated that 100 kilos. of 169° Tw. vitriol require 7.5 kilos. of oil, costing about 2½d., whilst platinum stills require for a similar quantity 15–20 kilos. of coal. The iron pans last for three months.

Another combination, yielding a 98 per cent. vitriol, consists of two platinum vessels with lead covers, of which the upper vessel containing the weak acid is heated direct, and of two separately heated iron retorts provided with lids. The whole is completely surrounded with fire, which passes first below the pans and returns over the top lids.

At the California Powder Works, Pinole, California, a cast-iron pan is in use measuring 1.68 m. × 0.46 m. × 0.254 m. deep, and divided by means of partition walls into three channels, each 0.127 m. wide, for the circulation of the acid. The acid is finally delivered into Johnson and Matthey's platinum cooling apparatus, from which it is obtained perfectly clear. The vitriol resulting from 6,000 cub. metres chamber space passes through two Delplace apparatus into the iron pan, which is heated by means of mineral oil. The pans last about 4–6 months, and the resulting acid is remarkably free from iron.—H. A.

#### *The Electrolytic Production of Potassium Chlorate.* C. Häussermann and W. Naschold. Chem. Zeit. 18, 1894, 857–858.

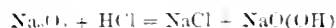
EXPERIMENTS were made to determine the conditions most favourable to the production of chlorates, the apparatus used consisting of a quadrangular vessel of sheet iron, which served also as cathode, the anode being of retort carbon or of platinum enclosed in a porous clay cell. The capacity of the anode cell was 0.5 litre and that of the cathode compartment 1 litre. The current was produced from a dynamo with a voltage of 110, the strength being maintained at 5 amperes by means of added resistances. The duration of each experiment being three hours, the current value was equal to 15 ampère hours; the temperature was regulated by immersing the apparatus in a water-bath. After every experiment the volumes of the anode and cathode solutions were measured and the liquids analysed. Of the cathode solution 100 cc. were boiled and diluted to 500 cc.; one part was then titrated with semi-normal hydrochloric acid, using methyl orange as an indicator, whilst another part, just acidified with dilute sulphuric acid and neutralised by digestion with zinc oxide, was used after filtration for the estimation of chlorine by decinormal silver solution. In the anode solution the total chlorine was determined as above; to determine the chlorate a part of the boiled and then diluted solution was mixed with excess of decinormal ferrous salt and dilute sulphuric acid and heated, the excess of ferrous salt being titrated back with potassium perman-

ganate solution. 1 ampère hour should yield theoretically 0.55 g. 0.75 gm.  $\text{KClO}_3$ , or 0.208 gm.  $\text{KOH}$ . In the first experiment a 10 per cent. solution of  $\text{KCl}$  at 15° C. was used alike for the anode and cathode cells, the effective carbon anode surface was 150 sq. cm., and the current density 0.033 ampère per sq. cm., while the voltage ranged from 1.5 to 5. But after the three hours only 0.9 gm.  $\text{KClO}_3$  was found in the anode solution, equal to 0.06 gm. per ampère hour, or 8 per cent. of the theoretical yield. In a second experiment, otherwise similar to the first, 100 grms. of a 30 per cent. potash solution were run into the anode cell, charged as before, at such a rate that free chlorine was always distinctly present. In spite of the loss of chlorine 0.38 gm. of  $\text{KClO}_3$  per ampère hour were obtained, or 50 per cent. of the theoretical yield. In a third experiment the 100 grms. of potash solution were so added that the reaction throughout was slightly alkaline. The anode was of platinum sheet (210 sq. cm. in effective surface), and the voltage averaged four. The result was better, 0.5 gm.  $\text{KClO}_3$  per ampère hour (= 67 per cent. of theoretical) being formed at the anode. By substituting potassium hydroxide for the potassium chloride in the anode cell only 33.5 per cent. of the theoretical result was obtained, which accords with the observation that the chlorate is produced by a secondary action of chlorine on caustic potash, and not by that of electrolytic oxygen on potassium chloride. By substituting platinum wire for sheet a greater current density per sq. cm. of anode surface was obtained, and a distinct smell of ozone was observed; yet in this experiment, and in others in which a higher temperature (up to 85° C.) or a stronger solution of potassium chloride were used, no appreciable alteration in the result was observable, nor was there any change in the yield of chlorate when potassium hydroxide was substituted for the chloride in the cathode cell. From these experiments it would seem that the highest yield of chlorate is obtained when the anode solution is kept faintly alkaline, while it is lower if a weak acid or strongly alkaline solution be used; on the other hand, it is not greatly affected by a moderate alteration in temperature or concentration. Nevertheless, a stronger solution and a higher temperature are more conductive and therefore more economical. On a large scale, therefore, the solution should be concentrated and warm (80°); platinum is the best material for the anode, but lead or lead peroxide might possibly be substituted. In practice, instead of using fresh caustic alkali, the cathode liquid might be caused to flow at a suitable rate into the anode cells. The chlorate is obtained from the anode liquid by crystallisation, followed by washing and recrystallisation. It is still an open question whether the direct production of chlorate in this way is more economical than the process in which electrolytic chlorine is employed. Much depends upon the possibility of obtaining a diaphragm of low resistance capable of resisting the action of chlorine and of alkalis. Sodium chlorate may be formed in like manner to the potassium salt.—W. G. M.

#### *The Action of Sodium Peroxide upon Acids in Alcoholic Solution.* J. Tafel. Ber. 27, 816.

COMMERCIAL sodium peroxide, although acting violently upon many organic bodies, does not seem to react with alcohol. With very dilute alcohol, and even with acids in alcoholic solution, little or no oxygen seems to be evolved. In the case of concentrated alcoholic hydrochloric acid, although there is no visible evolution of gas, a crystalline powder is deposited.

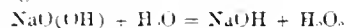
The product must be either a mixture or a combination of sodium chloride with a body of the composition  $\text{NaIO}_2$ . The following equations probably represent the reactions:—



By action of heat—



On treating with water—





The equations only generally represent the reactions taking place.

Sulphuric acid and nitric acid act in a similar way with commercial sodium peroxide.

The hydrate  $\text{NaO}(\text{OH})$  does not seem to have the property of forming salts with either hydrochloric, sulphuric, or nitric acid.

With acetic acid, sodium peroxide behaves quite differently. If the peroxide of sodium be thrown into acetic acid, the latter immediately bursts into flame. But if it be added, with certain precautions, to a solution of acetic acid in alcohol, it gives a clear solution, from which a colourless salt crystallises out. This salt gives off oxygen at a low temperature, and with water gives a solution which behaves in many respects like a solution of hydrogen peroxide, and which is neutral.

On heating this product, oxygen and water are given off, and the residue is nothing but sodium acetate. It is therefore possible that by the action of sodium peroxide upon acetic acid, sodium acetate is formed with hydrogen peroxide in the form of water of crystallisation. This would explain the behaviour with heat and with water. But the author thinks there must be a similarity in the reaction which takes place when acetic acid, and when mineral acids act upon sodium peroxide. The explanation above given cannot apply to the product of the action of the peroxide on hydrochloric acid, since, according to analysis, this product evolves too little water to correspond with the amount of oxygen, if both were present in the state of  $\text{H}_2\text{O}_2$ . He thinks it more probable that the following reaction takes place—



Thus the hydrate  $\text{NaO}(\text{OH})$  seems to form a salt with acetic acid, but not with mineral acids.

In conclusion, the author has found that commercial sodium peroxide is changed by pouring alcohol upon it. Part of the substance goes into solution, from which, concentrated hydrochloric acid precipitates nothing but sodium chloride. The remainder, which is insoluble in alcohol, forms with alcoholic hydrochloric acid a salt which in appearance cannot be distinguished from the salt obtained directly from commercial peroxide, but which, according to analysis, contains sodium chloride and the hydrate, in the proportion of approximately 1:1.—H. M.

*The Behaviour of the Woods of Conifera to Solutions of the Chlorides of Copper.* E. Jensch. Zeits. angew. Chem. 1894, 153—155.

WHEN extracting copper from its ores according to Hoepfner's process (this Journal, 1889, 401), great difficulties have been encountered owing to the carbonising action of the hot cuprous chloride solutions on the timber of the conical rotating drums such as are used in the extracting process. After 22 days' work the fir staves were found to be carbonised to a depth of 4—5 mm., and the pitch-pine staves to a depth of 12—16 mm. It was, in fact, quite impossible to keep the apparatus liquor-tight. Further experiments proved that slowly-grown Bavarian fir shows greater powers of resistance than pitch-pine to the action of hot cuprous and cupric chloride solutions.—H. A.

*Volumetric Estimation of Sulphuric Acid in Crude Commercial Hydrochloric Acid.* L. Rump. Chem. Zeit. 18, 1894, 225.

See under XXIII., page 904.

*Note on the Valuation of Potashes.* O. Bernheimer. Chem. Zeit. 18, 1894, 742.

See under XXIII., page 905.

*The Estimation of Sulphur in Sulphides, with Simultaneous Determination of Arsenic.* P. Jannasch. Zeits. anorg. Chem. 6, 1894, 303.

See under XXIII., page 905.

## PATENTS.

*Improvements in or relating to the Treatment of Sodium Chloride, and to the Method of Utilising the Products in the Manufacture of White Lead, and of Soda, Chlorine, and their Derivatives.* R. Haddon, London. From Compagnie Electro-Chimique de St. Bérn, Paris, France. Eng. Pat. 13,406, July 10, 1893.

THESE improvements relate to methods of dealing with the products set free in the electrolysis of salt solution; the chlorine and soda solution being brought together outside the electrolytic apparatus for the production of hypochlorite of soda; or the chlorine being given off is converted into useful derivatives, and the caustic soda solution, containing 120 grms. of caustic soda per litre, brought into contact with litharge in a digester, mechanically agitated, the solution of the litharge being assisted by heat, and the hot solution carbonated in a series of carbonators, insoluble white lead being precipitated and separated by a filter-press; the alkaline liquid being further carbonated for production of insoluble bicarbonate in solution of sodium chloride, which mother-liquor is returned to the electrolyser. The same process and apparatus may be employed for carbonating the alkaline liquid direct from the electrolyser, the chlorine being employed for production of chloride of lime.—J. C. R.

*Improvements in the Electrolysis of Iodides, Bromides, Nitrates, and other Salts.* J. Hargreaves, Farnworth-in-Widnes, and T. Bird, Cressington, Liverpool. Eng. Pat. 14,130, July 21, 1893.

THIS invention relates to the decomposition of iodides, bromides, nitrates, and other salts by the electrolytic method, and consists in the employment of apparatus of the kind described in the specification of Eng. Pat. 18,871 of 1892 (this Journal, 1894, 255, and 528) for use in the manufacture of alkali. The inventors find that electrolytic apparatus of the kind referred to is applicable for use in the decomposition of various substances besides sodium chloride, such salts as above mentioned, yielding iodine, bromine, and nitric acid next the anode respectively.—J. C. R.

*Improvements in the Electrolysis of Chlorides, Iodides, Bromides, Nitrates, and other Salts, and in Apparatus therefor.* J. Hargreaves, Farnworth-in-Widnes, and T. Bird, Cressington, Liverpool. Eng. Pat. 14,131, July 21, 1893.

THESE improvements relate to developments of the process contained in Eng. Pats. 18,871 of 1892, 5197 and 5198 of 1893 (this Journal, 250, 255, and 528 of 1894), wherein a cathode is employed in contact with, or forming part of, a porous partition in apparatus for the electrolysis of alkali salts. The present objects are to secure a more efficient and convenient construction of apparatus in the direction of modifications of the methods employed in forming the porous diaphragm, of lime and asbestos on wire gauze, dried, and steeped in phosphate of soda, &c. solution, thus converting the lime or baryta into insoluble phosphate, the soda being "left free." In some cases the alkaline earth is carbonated before being treated with phosphate or silicate, and the density of the diaphragm may be varied by treatment with solution of lime, baryta, or magnesia, drying, and again treating with phosphates or silicates.—J. C. R.

*Improved Process and Apparatus for the Manufacture of Cyanides.* A. Gordon and J. Gordon, Loanhead, Midlothian. Eng. Pat. 16,750, September 6, 1893.

THE blast furnace in which the process is conducted, has its lower part shaped like an inverted truncated cone, formed as an iron water-jacket, several inches wide, through which

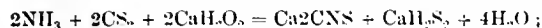
water is kept flowing, the tuyère openings being into the lower part. The jacket may be continued up the barrel of the furnace, which has an outlet a short distance above the hottest part. The fumes are drawn through this outlet by an exhausting apparatus so as to traverse a series of metal pipes, to which water may be applied externally. There are bottom boxes to the pipes that can be heated from fireplaces when it is desired to draw off the fumes in a fused state. There is also a wet fume condenser, similar to that described in Eng. Pat. 5239 of 1878. The top of the furnace is provided with an outer iron cup and cone; the cup of the usual description; but the cone has an aperture above, closed by an iron ball, actuated by a hand lever, opening out to a box or hopper, into which the alkaline materials for producing cyanides are placed. When the cone with its hopper is depressed, the fuel falls from the larger hopper, about the circumference of the furnace, leaving the inner part vacant for the materials, which are introduced to the centre from the cone. By this arrangement injurious action on the brickwork walls of the furnace is obviated.

In manufacturing potassium cyanide, for instance, potassium carbonate is intimately mixed with carbonaceous matter, preferably nitrogenous, such as animal substances, coal, pitch from shale, and the like, for introduction to the furnace interior. Ammonia may be passed into the furnace along with the blast. The heat maintained is high enough to volatilise the cyanide as it forms, so that it is conducted as fume into the condensing apparatus.—E. S.

#### *Improvements in the Manufacture of Sulphocyanides.*

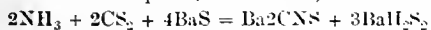
H. W. Crowther, West Bromwich, and E. C. Rossiter, Smethwick. Eng. Pat. 17,846, September 22, 1893.

CALCIUM sulphocyanide is obtained by digesting together ammonia, carbon bisulphide, and lime, preferably approximately in the proportions indicated by the equation—



but a slight excess of both ammonia and lime is used. The ammonia is taken at 7 to 15 per cent. aqueous solution, which is charged into an iron autoclave, fitted with stirrer and steam-jacket, the latter admitting of cold water being run in for rapid cooling. The lime solution is then added, and when these are well mixed, the carbon bisulphide. The pressure is allowed to rise to 15 to 25 lb. per square inch before shutting off steam, after which the pressure may rise to 60 to 90 lb. When the pressure has fallen, the vessel may be again heated. Other bases may be substituted for lime; namely, baryta, strontia, magnesia, and the caustic alkalis. The solution of calcium sulphocyanide and sulphhydrate obtained, is run into a series of vessels described in Eng. Pat. 8666 of 1887, for treatment with carbonic acid, preferably limekiln gases, to convert the sulphhydrate into carbonate or bicarbonate. The hydrogen sulphide evolved may be utilised in making sulphuric acid or sulphur. The calcium carbonate is filtered off. When alkali sulphocyanides are wanted, it is preferred to obtain these from the calcium sulphocyanide by means of the alkali carbonate or sulphate.

Sulphides may be substituted for hydroxides in the process. The following equation represents the proportions used when barium sulphide, for instance, is taken:—



Reference is made to Eng. Pat. 5354 of 1891, according to which manganese dioxide or ferric hydroxide is used in the manufacture of sulphocyanides.—E. S.

#### *Improvements in the Process of Concentrating Sulphuric Acid in Lead Vessels.*

W. Wolters, Borowitzchi, Russia. Eng. Pat. 18,831, October 7, 1893.

LEAD sulphate is added to the sulphuric acid to be concentrated, and is kept in suspension by suitable agitation, so that the material of the vessel is protected from corrosion by the acid. The vapour given off is taken to leaden

chambers for condensation. Most of the lead sulphate dissolved by the acid, separates on cooling and may be used again.—E. S.

#### *Improvements in the Elimination of the Oxides of Zinc, Iron, and Copper from Cobalt Oxide, and Apparatus therefor.*

E. A. E. Weinberg, Dornafvet, and R. Curriek and J. G. Johanson, both of Gelle, Sweden. Eng. Pat. 24,900, December 27, 1893.

THIS invention relates to the elimination of the oxides of zinc, iron, and copper from cobalt oxide by the use of hydrochloric acid. The impure oxide having been washed in boiling water to free it from alkaline salts, is dried, crushed, and sifted through a 50-mesh sieve. It is then mixed with 10 per cent. of crude hydrochloric acid, and the paste thus produced is placed in a fire-brick muffle or oven and heated to about 900 °C., care being taken that the atmosphere has free ingress and egress, and that the mixture is well stirred. The oxides are converted into chlorides, all of which being volatile with the exception of the chloride of cobalt, pass off, leaving the latter behind. The temperature is then increased to about 1,150 °C., when the chloride is transformed into the dark blue cobalt oxide, which on being washed in boiling water, dried, and pulverised, is ready for the market.—A. W.

#### *Improved Apparatus and Process for Electro-Chemical Decompositions.*

H. Carnichael, Malden, Mass., U.S.A. Eng. Pat. 8061, April 24, 1894.

See under X1., page 891.

#### *Improvements in Methods of Treating Lead Ores.*

A. G. Fell, New York, U.S.A. Eng. Pat. 9536, May 15, 1894.

See under X., page 889.

#### *An Improved Method, Means, or Process of Producing Caustic Soda and Chlorine Gas or Caustic Potash and Chlorine Gas by the Electrolysis of Common Salt in Solution or Chloride of Potassium in Solution.*

T. Drake, Huddersfield. Eng. Pat. 11,661, June 16, 1894.

THESE improvements consist in wrapping metallic gauze or perforated metal sheeting round a porous pot, and making cathode connection direct thereto. This gauze, preferably of copper, is treated with a salt of mercury sometimes, and so becomes amalgamated; but whether amalgamated or not is intended for use as a cathode in the electrolysis of salt, &c., the salt solution being placed in the porous pot, and water in an outer iron vessel to collect the soda from the cathode. In other respects the apparatus resembles that described in Eng. Pat. 7985, 1894 (this Journal, 1894, 744), the gauze replacing the bath of mercury.—J. C. R.

#### *Improvements in Apparatus for Obtaining Ammonia and Ammonia-Salts from Nitrogenous Organic Matters.*

H. H. Lake, London. From L. Sternberg, Jersey City, U.S.A. Eng. Pat. 12,881, July 3, 1894.

THE retorts are vertical, and in groups of four, each group having a common feed chamber at top. There is a feed pipe to each retort, having two slide-valves, by use of which the feed can be effected with the minimum admission of air. There is also a common discharge chamber to each group, also provided with two valves. Each retort has a vertical screw of coarse pitch, the thread of which is secured to an elongated drum mounted upon a hollow shaft, for admission of non-oxidising gases and steam, which enter by a hollow stuffing-box, and serve to cool the shaft, and so to protect the screw from overheating. The shafts are kept revolving. A fireplace is provided, but is chiefly used on starting, the necessary heat being maintained by combustion of the gas produced in the process. The materials for the production of the required products having been charged into the retorts, and duly heated, the effluent gases are led from the

receiving chamber below into an absorber, where ammonia is withdrawn by sulphuric acid, and the ammonia-freed gases are pumped into a gas service pipe, from which, by suitable valves, they are led, either for storage of excess, into a gas meter, or directly to a series of branched pipes, through which the gases pass, partly, as already described, into the hollow shafts, and partly into the heating chambers surrounding the retorts, where their combustion maintains the required temperature. Provision is made for the injection of steam as required. There is reference to a former patent, 21,723, 1892 (this Journal, 1893, 263).—E. S.

bottom of the seggar is cut away, thereby reducing its weight and enabling such articles as basins and dishes to be placed so as to protrude through the bottom of the seggar, or, if inverted, through the bottom of the seggar in the row above. Either arrangement effects an economy of space in the kiln.—V. C.

*Improvements relating to the Manufacture of Filters.* H. H. Lake, London. From Bittel and Co., Worms-on-the-Rhine. Eng. Pat. 19,873, October 21, 1893.

See under 1., page 872.

## VIII.—GLASS, POTTERY, AND ENAMELS.

*The Manufacture of Porous Cells.* D. Titaner. Central-Auzeiger f. Ziegel- u. Kalkind. 1894, 13, 47.

THE author discusses the composition and preparation of the mixtures employed in the manufacture of porous galvanic-battery cells. The addition of chalk to the mass results—owing to the escape of carbonic acid during firing—in a high degree of porosity. Such cells are, however, not proof against the action of strong acids. For this purpose, lime must be absent.—H. T. P.

*Transparent Cement for Porcelain.* Deutsch. Töpfer u. Ziegler Zeit. 25, 1894, 220.

CAOТСНОУ (75 grms.) is dissolved in 60 grms. of chloroform and 15 grms. of mastic added. The mixture is allowed to remain in a closed flask until solution is complete.

—A. R. L.

## PATENTS.

*Improved Means or Process for Operating upon Glass for General Ornamentation and Embellishment Purposes.* A. A. Gibbs, S. A. Gibbs, and W. T. Gibbs, London. Eng. Pat. 15,341, August 11, 1893.

THE following is an example of the means of carrying out the inventors' process:—A coat of acid resist is laid upon the glass; from the parts forming the background to the design the "resist" is removed with a stencil; soda and hydrofluoric acid are then poured upon the surface. Hydrofluoric acid is next applied; the resist is then removed and the glass is cleaned. The glass is next coated with stain, and by means of a stencil the ornament is freed from the stain, which remains as a protection for the background. The stain is then burnt into the glass. The glass is then taken from the kiln, cleaned, and the required outline traced upon the glass, the background being filled with acid resist. The solution of soda and hydrofluoric acid is again poured on so as to leave a white "mat" on the whole ornament, leaving the outline, which is protected by the resist, clear. The shading-in is then done according to the ordinary process of the trade.—V. C.

*Improved Seggars for Use in Enamelling, Hardening on, Lustre and Majolica Kilns.* C. Copeland, Tunstall, Staffordshire. Eng. Pat. 18,388, October 2, 1893.

THE object of this invention is to make seggars which will hold a larger quantity of ware than those of the ordinary pattern. Each seggar is provided with upright pillars at suitable distances near the outer periphery, the pillars being furnished with grooves on their inner surface to serve as supports for various articles. A portion of the

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### PATENTS.

*Improvements relating to the Manufacture of Artificial Stone.* A. McLean and C. W. Wilson, London. Eng. Pat. 17,322, September 14, 1893.

THE clinker from refuse-destroyers is ground to the size of small shot, mixed with a binding material, such as Portland cement, and water added; the proportions being approximately three parts clinker to one part Portland cement. The mixed materials are then subjected to a pressure of from 250 to 500 tons to the square inch. The material obtained has the advantage of being non-slipping.

—V. C.

*Artificial Plaster of Paris made from the Lime Residue resulting from the Reduction of Sulphate of Ammonia.* J. L. Kerr, Manchester. Eng. Pat. 8475, April 28, 1894.

THE hydrated sulphate of lime formed as a by-product in the above process is dried by waste heat in suitable kilns and heated for a considerable time to a temperature of 300° F. until all the water of crystallisation is driven off. It is then ground to a fine powder and stored in air-tight barrels. It is claimed that the plaster of Paris thus made is a distinct novelty, and is equal to that now obtained from gypsum.—V. C.

*Improved Stoneware for Building Blocks, Tiles, Ornaments, and other Articles and Objects.* J. C. Sellars. Birkenhead, and C. A. Ropes, Liverpool. Eng. Pat. 9241, May 10, 1894.

THE ware comprises, first, a granular substance, such as sand; secondly, the silicate or borate of soda or potash; and lastly, a compound of aluminium, preferably alum. The solution of silicate or borate is mixed with the granular material to a plastic consistency, the plastic material being then placed in moulds, from which it is readily removed when required. The moulded article is then dried and soaked with solution of alum.

THE ware thus produced will resist atmospheric action and temperatures such as are reached in an ordinary fire-grate. If desired, the articles thus made may be coated with cement or enamel. V. C.

## X.—METALLURGY.

*The Tortimolite Process.* Iron and Coal Trades Rev.  
1894, 238.

THIS is a new process, for improving pig-iron after it is cast, now being tested at the Little Bell furnace in Bessemer, U.S.A. It is called the Tortimolite process, and consists in running pig metal into chills heavily coated with a wash of impure graphite, apparently containing about 75·11 per cent. of carbon. The metal is supposed to absorb some of the carbon and become stronger and softer, probably somewhat on the principle of cementation. It makes a very smooth, clean pig, and shows slightly less chill than untreated metal in chills. Tests so far made seem to indicate a gain of strength in test bar and in pig, and a slight increase of carbon in the metal as well as slight changes in the other chemical constituents. Tests made at the Howard Harrison Pipe Works of this metal, as compared with untreated pig of the same cast, are said to have shown decided superiority in fluidity and strength for the Tortimolite metal.

*The Variations in the Silicon Percentage of different Samples of Pig-Iron from the same Tap.* H. Rubricius.  
Chem. Zeit. 18, 1894, 1005.

A SERIES of tests shows that the percentage of silicon in pig-iron is lowest in that part of the metal which is nearest to the bottom of the blast furnace, and increases in the same charge as the upper portion nearest the slag hole is approached—sometimes to the extent of 1 per cent. But it is not yet possible to correlate the initial silicon percentage and the rate of increase, as it often happens that a sample in which the lowest portion contains but little silicon is unusually rich in that element in the upper part. The difference cannot be accounted for by the lower specific gravity of the silicon, yet the phosphorus and sulphur do not show the same variation. The results of eight tests are quoted below:—

Test.	Percentages of Si from below upward.						
	1.	2.	3.	4.	5.	6.	7.
1	1·13	1·15	1·15	1·19	1·33	1·40	1·42
2	1·38	1·44	1·45	1·60	1·63	1·72	1·79
3	1·15	1·34	1·43	1·57	2·17	2·18	2·20
4	1·40	1·50	1·54	1·66	1·82	1·84	1·88
5	1·95	2·09	2·13	2·45	2·70	2·72	2·76
6	1·81	1·83	1·84	1·86	1·89	2·16	2·20
7	2·72	2·74	2·77	2·79	2·85	2·88	2·89
8	2·46	2·48	2·50	2·53	2·54	2·58	2·60

—W. G. M.

*The Ligation of Silver Copper Alloys.* E. Matthey.  
Proc. Roy. Soc. 55, 265—269.

THE author refers to the difficulty of obtaining a plate of standard silver (925 parts of silver in 1,000) of uniform standard, and, as a result of many trials, he concludes that the best means to ensure this is to cast the alloy in very thin plates and subject them to slow and uniform cooling. Thick plates by rapid or slow cooling are sure to have points of local richness. A bar was cast into a "skillet" mould to produce a casting 30 cm. in length, 13 cm. in width, and 6 mm. in thickness, weighing 5 kilos. Assays of various parts of this plate, after slow and uniform cooling, showed a very slight tendency for the silver to liquefy to the centre. Another bar in the same mould cast at a higher temperature showed a depreciation of silver at the centre.

The mould was ultimately reduced to 1 mm. in thickness, and the results then obtained showed that liquation, although not entirely prevented, had practically disappeared.

—A. W.

*The Pneumatic Pyrometer.* H. Motton. Engineering,  
1894, 251.

See under I., page 869.

## PATENTS.

*Improvements in the Treatment or Preparation of Pulverised or Finely-divided Ores, or the like Material containing Iron, for Use in the Blast Furnace for the Production of Iron.* W. Thonkinson, West Hartlepool.  
Eng. Pat. 13,769, July 15, 1893.

THE materials, after pulverisation when necessary, are consolidated into lumps by incorporation with a pulp of finely-ground slag or other suitable matter and subsequent drying or setting.—J. H. C.

*Improved Method of and Apparatus for Pickling Metal Articles.* S. O. Cowper-Coles, London. Eng. Pat.  
15,507, August 15, 1893.

WEAK acid pickling solutions are used in combination with currents of electricity, which are frequently reversed so as to loosen and detach the scale; heat being used when necessary, as also magnets variously applied. The scale is removed by continuous pumping and filtering or other convenient method.—J. H. C.

*An Improved Process and Means used therein for the Disintegration of Agglomerated Minerals.* W. S. Lockhart, Blackheath. Eng. Pat. 16,724, September 5, 1893.

THE agglomerations are treated by water or other liquid under pressure, either by jets applied *in situ* or in closed stationary or revolving chambers. The disintegration may be aided by alternate freezing and thawing in some cases.

—J. H. C.

*Improvements in the Manufacture or Production of Metals from their Ores, and in Plant or Apparatus therefor.* W. F. Berner, St. Petersburg. Eng. Pat. 19,375, October 14, 1893.

THIS patent is for a complicated furnace, having for its object the smelting and refining of ores. In the case of iron, the furnace consists of two shafts having a common hearth and a refining furnace adapted to be placed in communication with such hearth, one half of the furnace being first worked as a blast or reducing furnace, whilst the other half is worked as a decarburising furnace by the hot gases escaping from the first half. The regenerative principle is brought into play so that the relative functions of the two halves of the furnace can be reversed. Various devices are described for regulating the supply of air, gases, &c., according to the requirements of the furnace, so that almost any emergency can be met and provision made for smelting almost any ore, the whole smelting and refining being carried out in one operation, and in one furnace.—A. W.

*Improvements in Methods of Treating Lead Ores.* A. G. Fell, New York, U.S.A. Eng. Pat. 9536, May 15, 1894.

GALENA or other ore is crushed to a fine powder and treated with water containing a mixture of sulphuric and hydrochloric acids, or sulphuric acid and a chloride. Heat is applied for from 2 to 6 hours, the operation terminating about 30 minutes after the evolution of sulphuretted hydrogen has ceased, when more water is added and the mass stirred and allowed to settle. The sedimentary lead compound is washed free from acid and is then agitated

with a solution of monocarbonate of soda and moderately heated. After settling, the solution of soda salts is drawn off, and the resulting lead compound is then treated according to the commercial product required; for instance, with nitric acid for the nitrate, acetic acid for the acetate, and so on. If the oxide is required it is mixed with a small quantity of nitric acid, dried, and heated with varying intensity, according to the kind of oxide required.

If silver be present in the galena it is extracted by treating the compound after the soda treatment with a hot solution of sodium chloride previous to the final treatment for the production of the lead salt or oxide.—A. W.

*An Improved Flux for Use in Soldering Aluminium and other Metals.* W. P. Thompson, Liverpool. From O. Nicolai, Wiesbaden, Germany. Eng. Pat. 11,148, June 8, 1894.

THE object is to solder aluminium to itself or other metals with a suitable solder, such as zinc, tin, or cadmium, or an alloy of these or similar metals, and with the use of a flux consisting of a cadmium salt, preferably one of the haloid salts.—A. W.

*A Means or Apparatus for Producing Pure Castings.* H. R. Müller and F. O. Müller, Löbtau, Saxony. Eng. Pat. 11,574, June 14, 1894.

THE apparatus consists of a peculiarly arranged inlet and collector for separating and retaining all mechanical impurities, such as slag, &c., so that no portion of it can enter the mould.—J. H. C.

*Improvement in the Method or Process of Treating Gold and Silver Ores, and a Composition or Matter for the same Purpose.* E. D. Kendall, Brooklyn, U.S.A. Eng. Pat. 11,797, June 18, 1894.

THE pulverised ores are treated with sodium dioxide and potassium or other suitable cyanide, generally in the form of solutions, and previously mixed in suitable proportions. They are then leached and filtered in the ordinary way, and the metals precipitated by electrolysis or other suitable means.—J. H. C.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

*The Manufacture of Porous Cells.* D. Titaner, Central-Anzeiger f. Ziegel- und Kalkind. 1894, 13, 47.

See under VIII., page 888.

*The Electrolytic Production of Potassium Chlorate.* C. Haussermann and W. Naschold. Chem. Zeit. 18, 1894, 857.

See under VII., page 885.

### PATENTS.

*Improvements in or relating to the Treatment of Sodium Chloride and to the Method of Utilising the Products in the Manufacture of White Lead and of Soda, Chlorine, and their Derivatives.* R. Haddon, London. From Compagnie Electro-Chimique de St. B'ron, Paris, France. Eng. Pat. 13,406, July 10, 1893.

See under VII., page 886.

*Improvements in Electrolytes for Primary Batteries.* W. H. Longsdorf, London. From G. Hewett, New York, U.S.A. Eng. Pat. 14,056, July 20, 1893.

THE following formulae are given for producing two kinds of powder for use in primary batteries: the first for use in single fluid batteries or next the carbon in batteries having porous cells; the second for use as an excitant in the compartment containing the zinc element in porous cell batteries:—

I. 8 fluid oz. commercial nitric acid, 36° B.;  
3 pints commercial sulphuric acid, 66° B.; the mixture heated to 136° F.; and  
8 lb dry bichromate of soda dissolved therein. The compound on allowing to cool solidifies, and can then be ground to powder. The mass should be cooled rapidly, protected from the atmosphere, and subsequent exposure avoided.

H. 15 oz. powdered chloride of sodium;  
1 oz. bisulphate of mercury; and mix thoroughly.

The powders are for use in solution, being readily soluble.  
—J. C. R.

*Improvements relating to the Manufacture of Paris Blue or Berlin Blue, and of Berlin Green, by Electrolysis.* H. Goebel, Garzweiler, Germany. Eng. Pat. 14,089, July 20, 1893.

See under IV., page 876.

*Improvements in the Electrolysis of Iodides, Bromides, Nitrates, and other Salts.* J. Hargreaves, Farnworth-in-Widnes, and T. Bird, Cressington, Liverpool. Eng. Pat. 14,130, July 21, 1893.

See under VII., page 886.

*Improvements in the Electrolysis of Chlorides, Iodides, Bromides, Nitrates, and other Salts, and in Apparatus therefor.* J. Hargreaves, Farnworth-in-Widnes, and T. Bird, Cressington, Liverpool. Eng. Pat. 14,131, July 21, 1893.

See under VII., page 886.

*Improvements relating to Thermo-Electric Batteries, and to Apparatus for Use in the Manufacture of the Same.* J. B. C. Dion, Paris, France. Eng. Pat. 17,493, September 16, 1893.

THESE improvements relate to a thermo-electric battery, consisting of a series of elements composed of antimony, zinc, and a small quantity of tungsten and arsenical cobalt (100 parts antimony, 50 to 70 parts zinc, to which is added the tungsten and arsenide of cobalt), obtained by casting the metal into moulds. These elements are connected with each other by metallic strips, and placed in superposed crowns or in spirals round a furnace, the temperature of which is kept constant in an automatic manner by a thermometer arranged so that the mercury arriving at a certain height closes a circuit which electromagnetically works a lever regulating the admission of air to the furnace. Each crown is insulated from the others by asbestos discs, sunk into a suitable cement which covers about one-half of the elements. The other extremity is free, in order that a current of air may cool this outer portion, while the inner extremity of these same elements is heated to the highest degree.—J. C. R.

*An Improved Manufacture of Electric Accumulators.* L. W. and G. E. W. Schöffer, Gelnhausen, Germany. Eng. Pat. 17,866, September 22, 1893.

THESE improvements relate to the manufacture of electric accumulators, in which the active covering material of the positive electrode consists of a mixture of metallic lead

finely pulverised, with an electrically indifferent substance in the form of powder. This is either (1) separated by the acid in proportion as the conversion of the lead into peroxide progresses, and thus gives room for the increase of volume. Such substances are zinc-dust, starch, and the like. (2) Or the proposed substance is or becomes sufficiently yielding to permit of the expansion of the particles of lead in their conversion into peroxide of lead, and to receive the pressure produced. Such substances are pumice stone, blast-furnace slag, glass, cork, wood, &c., in the form of powder.—J. C. R.

*Improved Apparatus and Process for Electro-Chemical Decompositions.* H. Carmichael, Malden, Mass., U.S.A. Eng. Pat. 8061, April 24, 1894.

This specification deals rather extensively with the subject of the electrolysis of alkali salts, particularly chloride of sodium solution, for obtaining chlorine and soda, and aims at the better separation of the ions set free at the electrodes, partly (1) by the employment of porous partitions, arranged generally horizontally or on the slant; (2) the introduction of fresh electrolyte at a point or zone to prevent diffusion upwards of soda towards the products of the anode at the top, *i.e.* just below the anode; and (3) maintaining a lower temperature at the bottom than at the top of the electrolyte, and generally working the apparatus to obtain efficiency. Several forms of apparatus and their working are explained. The anodes of carbon may rest upon platinum wires or platinum pins connected to insulated mains. The electrolysis of sulphate of soda is also dealt with. There are 30 claims.—J. C. R.

*An Improved Method or Means or Process of Producing Caustic Soda and Chlorine Gas or Caustic Potash and Chlorine Gas by the Electrolysis of Common Salt in Solution, or Chloride of Potassium in Solution.* T. Drake, Huddersfield. Eng. Pat. 11,661, June 16, 1894.

See under VII., page 887.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*Ekenberg's Process of Refining Fats, Oils, &c.* R. Benedikt. Zeits. angew. Chem. 1893, 672—676.

In Ekenberg's process for refining fats, oils, &c., a centrifugal emulsor of the Separator Co. (of Stockholm) is combined with a Laval separator to form a battery or a number of batteries through which the fats and oils are passed, being alternately intermixed intimately in the centrifugal emulsor with the reagents (or wash-water), and subsequently separated in the Laval separator.

The author has studied this process both in the laboratory of the Separator Co. and in his own, with apparatus supplied by the company, and now gives the following description of process:—

The combination of one emulsor with one separator is termed an "element." In case the emulsion obtained in the former separates very easily a Laval apparatus may be dispensed with, and conveniently replaced by a Florentine receiver. The number of "elements" necessary to form a "battery" naturally depends on the number of washes required in each individual case.

As a rule, the emulsion formed in the emulsor may be run at once into the separator, the intermixture with the reagents being so intimate that refining is effected instantaneously. In case longer interaction be required, suitable receivers are interposed to alternately receive the mixtures.

The mixtures are heated or cooled, as the case may be, by a plate-warmer or a plate-cooler, and are moved from one element to the following by means of centrifugal pumps driven from the same shafting as the elements themselves.

The plates of the emulsors and the drums of the separators are made of "acid-steel" ("Saure-stahl"), resisting the attack of concentrated sulphuric acid and of alkalis. For dilute acids brass plates are used, and drums covered with an alloy of lead and antimony.

A battery, consisting of emulsors and separators of ordinary size, is able to work 3 to 6 tons *per diem*, the quantity depending on the nature of the oil, &c. In the case, however, of oils containing large proportions of free fatty acids, say up to 25 per cent., the output is smaller.

The following examples will serve to illustrate the manner in which the process is worked:—

*I. Refining of Vaseline Oils and Heavy Mineral Oils.*

—These oils are refined in the usual manner by washing with 8—25 per cent. of concentrated sulphuric acid, and washed subsequently with dilute alkali, and finally with water.

The crude oil is washed with once-used acid in the emulsor and separated, the now twice-used acid running into a tank. The chief object of this washing with acid, which has become reduced by the preceding operation to from 1.5 to 1.6 sp. gr., is to dehydrate the crude oil. The dried oil is next emulsified with fresh acid, the emulsion separated, and the separated acid conducted to the first separator. The oil is then washed with either already used or fresh caustic soda lye, as may be necessary, and finally with water; the emulsion is then allowed to separate in a Florentine receiver.

The empyreumatic resins do not separate, as in the ordinary processes, in the shape of large, hard lumps, but form a homogeneous emulsion with the acid. The waste acid is worked up in the usual manner by dilution with water and conversion into ammonium sulphate. If the separators are properly adjusted, rarely more than 0.1 per cent. of oil will be found in the wash-water.

*II. Refining of Fatty Oils.*—For refining fatty oils with sulphuric acid, as in the case of rape oil, only three elements are required, the oil being treated with acid in the first, and washed in the second and third with cold and warm water.

*III. Preparation of Neutral Oils.*—A plant for the preparation of neutral cocoa-nut or olive oil (even containing as much as 25 per cent. of free fatty acids) is described. It consists of six "elements," in three of which Florentine receivers have been substituted for centrifugal separators. The oil is washed first with alkali, then with water, next with alkali for the second time, afterwards with dilute alkali, then with very dilute acid, and finally with warm water.

As the emulsor works in a very satisfactory manner the emulsions are not always completely separated in the separator. This may not only be owing to faulty adjustment, but also to other circumstances peculiar to the nature of the emulsions, and it may happen that emulsions are formed which cannot be separated by the separator. Such emulsions, termed "critical" by Ekenberg, may be resolved into two layers by altering the concentration of the reagents, by addition of common salt, &c. From this it will be evident that a battery erected for a certain oil cannot be used for another without paying due regard to the peculiarities of the case.

The following advantages, besides continuous working, are claimed for the Ekenberg system:—The crude oil may be freed from tissues, &c. by a preliminary wash with water. In refining processes by means of acids there is a saving of from 40 to 60 per cent., considerably less oil being saponified. The acid is separated down to 0.2 per cent., and the consumption of alkali is, consequently, very small.

—J. L.

*Detection of Disguising and Perfuming Media in Oils and Fats.* D. Holde. Mittheilungen aus den konigl. tech. Versuchsanst. zu Berlin. 1894, 31.

See under XXIII., page 906.

*Properties of Sunflower Oil.* D. Holde. Mitt. aus den k. k. techn. Versuchsanst. zu Berlin, 1894, 36.

In the purpose of examination the oil was prepared in the laboratory from sunflower seed by extraction in a Soxhlet

apparatus with petroleum ether, boiling at 50° C. The following table, giving the properties of the oil, shows its great similarity to poppy oil, which has almost identically the same characteristics as those here given for sunflower oil:—

Observer.	Percentage of Oil in the Seeds.		Appearance. Smell and Taste.	I <sup>o</sup> = Viscosity Degree according to Engler (Water at 20° C. = 1).		Behaviour on Cooling.	Specific Gravity at 15° C.
	Expressed from Seeds after Removal of Husk.	Extracted by Solvent from whole Seed.					
Schadler	German seed, 33.5. Russian seed, 34.3.	26—27	Clear, pale yellow to bright yellow; almost tasteless; peculiar mild taste.	..	.. ..	Thickens on cooling; solidifies at - 16°	0.9260
Berlin Versuchs- anstalt.	.. .. .	26—28	Wine yellow; clear; pleasant taste.	6° 20 30 40 50	I <sup>o</sup> 8.2 5.5 3.9 3.1	Clear at - 5° C., turbid at - 10° C., but flowing easily; partially solid at - 17° C.	0.9249
Benedikt	.. .. .	.. .. .	Clear, bright yellow; agreeable smell; mild taste.	..	.. ..	Solidifies at - 16°	0.9262
G. de Negri and G. Fabris.	.. .. .	.. .. .	Clear, bright yellow; almost tasteless.	..	.. ..	.. .. .	0.9260

Observer.	Iodine Number.	Saponification Number.	Bechli's Test.	Millian Reaction.	Drying in thin Layer.	Acidity Per Cent. Oleic Acid.	Coefficient of Expansion between 20° and 78° C. $\alpha \times 1,000,000$ .	
Schadler	128—130	193	.. .. .	.. .. .	Moderately drying.	.. .. .	..	..
Berlin Versuchs- anstalt.	135	193	Negative	Yellow colour with brownish shimmer.	Became in part hard after 5 days at the temperature of the room.	5.6	746 to 763	
Benedikt	129—133 (after Spuller and Dietrich).	193	.. .. .	.. .. .	Slow drying	Free from acid.	..	..
G. de Negri and G. Fabris.	120	188—199 (expressed and extracted).	Negative	Negative	.. .. .	.. .. .	..	..

—H. S. P.

*The Examination of Linseed Oil.* F. Filsinger.  
Chem. Zeit. 18, 1894, 1005—1006.

SINCE the specific gravity, iodine number, and saponification equivalent of linseed oil are liable to vary even in unadulterated samples, the author has experimented with the optical test proposed by Aignan (Comptes rend. 1890, 110, 1273; this Journal, 1890, 903), as this indicates the

presence of rosin oils, which are dextro-rotatory, and which are the most likely adulterants, owing to their cheapness and the difficulty in detecting their presence. In the following table the author quotes the results of testing two samples of known purity expressed from German and Indian seeds, and of other commercial samples. In estimating the iodine number, a large excess of iodine was allowed to act upon the oil for from 18 to 20 hours:—

	German Seed.		Dutch Oil.		English Oil.	
	Oil for Food.	Indian Seed, Oil for Varnish.	1.	2.	1.	2.
Specific gravity at 17.5° C.....	0.9313	0.9313	0.9370	0.9370	0.9305	0.9310
Iodine number .....	182.9	181.8	159.9	164.3	185.3	175.4
	182.5	179.5	159.2	163.4	..	..
	182.4	178.1	..	..	..	..
Saponification equivalent .....	192.4	189.2	..	..	..	187.6
	192.1	188.5	..	..	..	187.9
Polarisation apparatus of Schmidt and Hansch, 200 mm.	±0°	±6°	+25°	+24°	±0°	±0°

—W. G. M.

*Estimation of the Titre of Tallow.* F. Wolfbauer.  
Chem. Zeit. Rep. 18, 1894, 28.

See under XXIII., page 908.

*Evaluation of Crude Glycerol.* O. Heller. Ber. der Pharm.  
Ges. 4, 1894, 17.

See under XXIII., page 908.

*Refined Fish Tallow.* W. Eitner. Leather Trades  
Circular and Review, 1894, 1035.

See under XIV., page 894.

### PATENTS.

*Improvements in extracting Oil from Oil-containing Plants and Fruits, and in the Production of Fodder from the Residual Matter.* W. Spindler and C. Stautz, Darmstadt. Eng. Pat. 16,987, September 9, 1893.

THE raw materials containing oil are mixed with a solution of salt, containing 2 to 5 per cent., and boiled for a short time. Instead of a saline solution, a solution of sugar or syrup, &c., might be used, but it is too expensive, and the fodder produced is inferior. The mass is placed in a press, and a slight pressure applied, when the saline solution flows at first of a milky colour, together with almost all the soluble portion of the material. Upon increasing the pressure, the oil is expressed, which may be allowed to float upon the saline solution, and separated in the usual way. The compressed material is then allowed to dry for use as fodder.

—J. J. K.

*Improvements in Apparatus to be Employed in the Clewing of Cotton Seeds.* W. A. Tattersall and W. E. Pearson, Cleckheaton. Eng. Pat. 17,808, September 22, 1893.

THE object of this invention is to remove all adhering cotton or floss from the seeds by special apparatus, preparatory to their being crushed for the extraction of oil, &c. The improved apparatus is provided with a hopper for the reception of the raw seeds, which pass therefrom into a concave tapered dish, perforated from the outside, so as to leave projecting burr edges in the interior, which serve as a holding surface for the seeds whilst they are acted upon by a taper roller covered with card teeth. The revolution of the card-covered roller serves to comb or card the cotton from the seeds, and the roughened interior of the dish prevents too much freedom in the motion of the seeds. The dish and taper roller are set at a slight incline, so that the seeds gradually fall towards the lower edge, where they are discharged.—J. J. K.

## XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

### (A.)—PIGMENTS, PAINTS.

#### PATENTS.

*An Improvement in the Manufacture of Sulphate of Lead Pigment.* A. Macdonald, Notting Hill. Eng. Pat. 11,972, June 17, 1893.

THE fumes of lead sulphate, if condensed in water, are filter-pressed, and then, wet or dry, are heated in the presence of air to a temperature of about 230° C. to destroy impurities.—F. H. L.

*Improvements in the Manufacture of Oil Paints and Compositions from Carbonised or Calcined Ironstone or Iron-ore.* A. Angell, Southampton. Eng. Pat. 7465, April 14, 1893.

THE pigment is heated to a temperature of about 150° C. after a preliminary grinding and before mixing with the oil, in order to remove moisture and as much air as possible from its capillary spaces. In the case of black "polarite," the heat should not be allowed to exceed 145°, and the mixture with the oil should take place at about 40° C.

—F. H. L.

*Manufacture of Coated Metallic Powders.* O. Inray, London. From J. Sachs, New York. Eng. Pat. 12,382, June 26, 1894.

"THIS invention relates to the manufacture of metallic powder, each particle of which consists of a core of one metal and a shell of another, the powder thus produced being applicable to purposes to which bronze powders, flitters or flakes, and metallics or large granules are applied, but at a less cost." Ordinary zinc powder is cleaned and immersed in a solution of zinc and copper in potassium cyanide and ammonia, with continual agitation until it is uniformly coated. The proportions of the copper and zinc may be varied according to the colour, &c. desired, or other metals may be employed, with or without heat and an electric current.—F. H. L.

### (B.)—RESINS, VARNISHES.

#### PATENTS.

*Improvements in Covering Lead Surfaces to Protect Water and other Potable Liquids in Contact therewith from Contamination.* A. McDougall, Southport. Eng. Pat. 14,586, July 29, 1893.

THE surfaces are coated with a mixture of equal parts of cotton-seed pitch dissolved in carbon bisulphide, and caoutchouc dissolved in naphtha.—F. H. L.

*Improvements in the Manufacture of a Drying Medium for Paints, and of Oil Varnish.* J. E. and C. S. Bedford, Leeds. Eng. Pat. 17,581, September 19, 1893.

THE fatty acids obtained from the hydrolysis of linseed oil or the mixture of such acids and unaltered oil produced by treating the latter with alkali insufficient in amount to saponify it completely, are submitted, in the presence of a small quantity of litharge, to the action of air or oxygen at a temperature not exceeding 70° C. in an apparatus such as that described in Eng. Pat. 18,628, 1889 (this Journal, 1890, 1046). Treated in this manner, they gain considerably in viscosity, and finally become almost insoluble in turpentine and petroleum spirit, but still remain miscible with oxidised or boiled linseed oil. To prepare a quick-drying varnish for use in floorcloth manufacture, the acids themselves, after oxidation, are diluted with 10 per cent. of turpentine or other solvent, heated to 70°, 5 to 15 per cent. of the moist hydrate of barium, strontium, calcium, or magnesium added, and the whole stirred vigorously till the soap has dissolved. Such a product when spread upon oil-cloth, with or without pigments, and placed in a stove heated to 60°–70° will dry in 12 hours without tackiness. To make a thinner varnish the mixture of oil and fatty acids, after oxidation, is cooled, and 50 per cent. of turpentine, &c., together with 3–7 per cent. of calcined magnesia added, and the whole thoroughly mixed.

One advantage claimed for these varnishes is that they yield smooth, glassy films free from wrinkles.—F. H. L.

*A New or Improved Furniture Polish.* A. P. Horn, Hamburg. Eng. Pat. 12,305, June 25, 1894.

THE inventor replaces the methylated spirit usually employed in the final operation of French polishing—know



as "spiriting-off"—by the following mixture:—8 kilos. of gum-benzoin and 16 of sandarac are dissolved in a mixture of 240 kilos. of alcohol and 120 of acetone, and, after filtering, 440 kilos. of "benzine" are added.—F. H. L.

#### (C).—INDIA-RUBBER, &c.

##### PATENT.

*Improvements in Apparatus for Separating Foreign Substances from Waste Rubber, Bark, Grain, and other Materials.* J. Anderson, jun., Manchester. Eng. Pat. 17,477, September 16, 1893.

See under L, page 871.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

*Refined Fish Tallow.* W. Eitner. Leather Trades Circular and Review, 1894, 1035.

Fish tallow is the solid grease which is separated at a low temperature from different kinds of fish oil; being a cheap and suitable material it is used in many tanneries, chiefly as "dégas." The fish tallow obtained from whale-blubber does not require much refining, and is as good for leather-dressing as is ox tallow; that from Japanese train oil contains a substance which has been termed fish glue, but is, in reality, closely allied to resin and india-rubber. This foreign matter is fatal to the successful employment of the fish tallow as imported in cakes from Japan; it prevents the grease from penetrating the skin, and remains as a sticky mass on the surface of the leather when the dubbing is applied on the table, although in the stuffing drum the effects of this resinous matter are not so serious. The refined fish tallow now placed on the market is free from this objectionable constituent, and is of a much brighter colour than the crude substance. It melts at 42° and is not so stiff as ox tallow. The market form of the refined fish tallow is that of pressed cakes, having an odour similar to that of chamois-dégas. It is much extolled by the author as a substitute for ox tallow.—A. G. B.

*Canaigre as a Substitute for Bark in Tanning.* Board of Trade Journal, September 1894, 262.

As supplementing the information already available on the subject of the use of canaigre as a substitute for bark in tanning (see also this Journal, May 1894, 650), the United States Vice-Consul-General at Vienna, in a recent report to his Government, supplies the following additional particulars:—

Eitner, director of the experimental station for the leather industry in Vienna, has made numerous experiments with the newly introduced American tanning material canaigre, and states that it has the property of tanning leather in a short time, and of giving it a brighter, clearer orange colour than has been obtained by any other tanning material. The leather is soft, neither shrinks nor swells, and although rapidly dyed, is strong, and not at all brittle.

Canaigre has also been used with good results at the Levinische Fabrik, Trieste, in the tanning of sole leather, particularly sole leather for the finer kinds of ladies' shoes. In the treatment of leather for ladies' shoes, the canaigre is added to pine bark. It affects the leather first, and causes the pine to act more quickly and evenly than it would otherwise do. In the Levinische Fabrik, in the final tanning process of skins which are to be used for sole leather, the skins are treated with a powerful mixture of extracts. In the first bath, to which the skins are subjected for 16 days, 7 German lb. of oak bark and 2 lb. of canaigre

or valonia are used. In the second bath, which lasts 24 days, there are 6 lb. of oak bark and 3 lb. canaigre; and in the third, which lasts 30 days, 5 lb. of oak bark and 3 lb. of canaigre or valonia. Very strong skins sometimes receive a fourth bath.

In tanning with extracts, canaigre seems to have a special value in giving colour and strength. Even when used in small quantities, with other powerful extracts, there is a noticeable difference if canaigre is used instead of valonia, the leather having a brighter yellow colour.

Canaigre is better than any other material for retanning leather which has been imperfectly tanned.

Canaigre is very similar to gambier in its tanning properties, and if it is ever to be exported in large quantities to Europe it will have, for the same amount of tanning power, to be as cheap or cheaper than the last-named article. It is for this reason that gambier has a particular importance for anyone interested in the sale of canaigre.

The firm of Blau and Schindler, of Hamburg, has imported more canaigre than any other continental firm, but, according to its own statement, has only imported it in small quantities, as it is not cheap enough at present to compete with gambier. Practically, the entire crop of gambier is shipped from India to England, and thence exported to the other European countries and to the United States. Gambier proper sells in Vienna or Hamburg at 8 dols. per 100 kilos. (220·46 lb.), while cutch, or wood gambier, is cheaper.

##### PATENTS.

*Improvements in the Manufacture of Leather for Gloves and other Purposes.* L. Cöhn, Johannegeorgenstadt, Germany. Eng. Pat. 12,518, July 2, 1894.

THE flesh side of the kid or other leather destined for the manufacture of gloves is to be rendered as smooth and soft as the grain side, which is usually made the outside surface of the glove. The flesh side (inside of the glove) is also to be tinted either of the same or of another tint as that employed for the grain side. In this way the finished glove will be of a better appearance and will keep cleaner than is at present the case.—A. G. B.

*An Improved Method or Process of Fastening the Colours upon and within Coloured Leathers.* J. T. Hardy, A. Brownson, and F. J. Bradley, Nottingham. Eng. Pat. 18,349, July 4, 1894.

THE skins, when dyed, are struck out and dried in the usual manner, and the grain is then covered with chrome-alumed gelatin, prepared by dissolving 1½ lb. of gelatin in three gallons of water and adding to the hot liquid ½ lb. of chrome alum dissolved in one pint of water. A mixture of glue (48 parts) and chrome alum (2 parts) with water (50 parts) may be substituted for the gelatin mixture. When the concoction has set to a jelly it is brushed over the skins, which are then exposed to the light, whereby the gelatin becomes insoluble. The seasoning and finishing processes are then effected as usual.—A. G. B.

## XV.—MANURES, Etc.

*The Solubility of the Phosphoric Acid contained in Bone Meals.* L. Gebek. Zeits. angew. Chem. 1894, 193–197.

It is well known that the manurial value of the phosphoric acid in bone meals is comparatively small, and not at all in accordance with the results to be expected from analytical data. Certain observations, as, for instance, the fact that a considerable portion of the  $P_2O_5$  is "citrate-soluble," indicate the presence of dibasic calcium phosphate—a compound very generally accepted as being of value for

plant-food. Indeed, from the author's analyses—the results of which are given in a series of tables—it appears that the bulk of the phosphate in bone meals is citrate-soluble. In practice, however, he finds that the amount of  $P_2O_5$  extracted by ammonium citrate (Wagner's solution; this Journal, 1894, 219, and also 769) depends very greatly on the degree of subdivision of the material under examination. For example, a series of bone meals when finely powdered yielded on an average over 80 per cent., and in two cases over 90 per cent., of their  $P_2O_5$  to the solvent; whilst from coarse meals, just able to pass through a sieve with meshes 1 mm. wide, only about 50 per cent. were extracted. On the other hand, ignition of the meals was found to materially alter their constitution, the soluble  $P_2O_5$  being reduced to 30 per cent. The extraction of the gelatin by steaming has a similar effect, although less marked (soluble  $P_2O_5$ , 68 per cent.). It is obvious, therefore, that investigations concerning the nature of the mineral constituents of bone must not be made on bone ash. For such researches Gabriel's method of removing the gelatin (by heating to 200° C. with glycerol and subsequently washing) proved very satisfactory, since the percentage of citrate-soluble  $P_2O_5$  is not sensibly altered thereby.

From his results the author concludes that only a small proportion of the  $P_2O_5$  of bones can exist as tricalcic phosphate; and he suggests, as more probable, that the bulk exists as dibasic phosphate, the remainder being in the form of a tribasic phosphate in which part of the lime is replaced by an organic base. On ignition, the organic base would be destroyed, its place being taken by lime derived from calcium carbonate.—H. T. P.

*Method for the Volumetric Estimation of the Phosphoric Acid Soluble in Water, contained in Superphosphates.* W. Kalmano and K. Meissels. Chem. Zeit. Rep. 18, 1894, 180.

See under XXIII., page 905.

#### PATENT.

*Improvements in Means for Treating the Refuse of Inhabited Areas.* R. C. Sayer, Bristol. Eng. Pat. 1152, January 23, 1894.

See under XVIII. B., page 899.

### XVII.—BREWING, WINES, SPIRITS, Etc.

*The Ready-formed Sugars of Malt.* E. Ehrich. Chem. Zeit. Rep. 18, 1894, 70.

The sugars occurring in barley-malt are cane sugar, invert sugar, and maltose. Isomaltose is not present.

The average percentage of these sugars is—Cane sugar, 4.54; invert sugar, 0.71; maltose, 0.82; total, 6.07.

The figures are looked upon as more accurate than those of O'Sullivan and Heron.

Malt is digested with water after treatment with strong alcohol, and, besides the estimation of the total weight of sugars by means of fermentation, the cupric reducing power and rotatory power can be determined. There are, however, present in the aqueous extract bodies other than sugars, which have a decided reducing power and rotatory power, and hence these determinations must not be taken as due entirely to sugars.—T. A. G.

*The Kiln drying of Malt as judged by the Colour of the Malt Combs.* Franz Uerny. Oester. Brauer- und Hopfen-Zeit. 7, 1894, 1.

It was found that under exactly similar conditions of curing the combs of a regularly-grown malt are darker than those of an overgrown malt, whilst the worts from the latter are the darker. Malts were cured without reference to the thermometer, and it was repeatedly found that with equal colours of malt-combs the malts gave worts differing greatly in colour. This difference is to be attributed to the different lengths of acrospire. Samples were taken at different stages from the malting-floor, and all cured under the same conditions at 100–105° C. The results indicate that the colour of a malt is not always indicated by the colour of the combs, but that the chief criterion of colour should be the acrospire. It follows then that younger and less fully-grown malts (as judged by the acrospire) should be kilned to yield darker combs than malts grown for a longer period or in which the acrospire is more fully developed.—T. A. G.

*The Composition of Hops and their Relation to Wort.* L. Aubry. Zeits. ges. Brauw. 17, 1894, 1.

The estimation of the alcoholic and water extracts is found to be useless, since two very dissimilar hops gave almost identical extracts. These two hops were extracted with petroleum ether, and the resin, ethereal oil, bitter principle, and nitrogen estimated both before and after removal of the resin.

The results show that very little nitrogenous matter is removed by water, hence it is of little importance in the wort. Worts boiled with the two kinds of hops yielded the same amount of total nitrogen; also the albuminoids in the worts boiled with resin-free hops were the same as in those boiled with normal hops. Petroleum ether removes no bodies from hops which could cause a precipitation of albuminoids.

Worts were boiled with completely ripe hops and hops containing many unripe cones, and the worts as well as the resulting beers were analysed. No difference could be detected in the beers on analysis, nor did the degree of hop-ripeness affect the degree of fermentation. Unhopped worts fermented less vigorously than hopped worts. The quality of hops has little influence on the composition of the wort and no influence on the extent of fermentation.—T. A. G.

*The Albuminoids contained in Worts and Beers.* Wahl and Hantke. Amer. Brewers' Review, 7 [32].

The authors have made a series of experiments on the albuminoids in worts, and also in fermented worts and beers.

They divide the albuminoids into the three classes, protein, peptone, and amide, and separate each class as follows:—

The protein albuminoids are separated by means of copper hydrate, lead or ferric acetate, or simply by heating with a few drops of acid, the results given in each case agreeing very closely.

The peptone albuminoids are separated either by means of tannin or phospho-tungstic acid, the tannin giving slightly larger results.

The amide albuminoids are estimated after the elimination of the two previous groups.

The authors also find that after the fermentation of worts the quantity of yeast obtained at the finish is considerably in excess of that used at the commencement, and during its growth that the yeast has absorbed a considerable quantity of the nitrogen of the albuminoids present in the original wort, the albuminoids which chiefly disappear being the non-coagulable or amide albuminoids. They also find that a larger amount of amide nitrogen disappears the higher the temperature at which fermentation takes place.—O. F. R.

*The Effect of Albuminoids in the Manufacture of Beer.* Wahl and Nilson. Amer. Brewers' Review, 1894 [38], 42.

The authors have made a series of experiments to find out which of the known albuminoids affect the stability, permanency of head, and maturity of a beer, and also whether these albuminoids have any disadvantageous effects, and whether the method of mashing in any way alters the character of the albuminoids.

*Influence of the Mash Temperature.*—The temperature at which the greatest amount of albuminoids is extracted is 110° F., the greatest sugar extract at 131°, and the greatest possible extract at 149°, at temperatures from 149° up to 152° F. the conversion of the starch becomes complete.

*Influence of Duration of Mashing.*—At 149° the quantity of albuminoids in the wort is not altered, whether the malt is mashed  $1\frac{1}{2}$  or 3 hours, but at temperatures below 140° the amount of albuminoids is greater after 3 hours' mashing than after  $1\frac{1}{2}$  hours.

*Coagulation of the Albuminoids.*—A greater amount of coagulable albuminoids is formed at a temperature ranging between 95 and 104° than at a high temperature such as 140—176°, the wort breaking much better in the former case.

*Effect of Concentration of Wort.*—The amounts of albuminoids and sugars extracted, do not vary between the limits of 1 part malt and 8 parts water, and 1 part malt to  $2\frac{1}{2}$  water, but at greater concentration the amount of albuminoids extracted, increases.

*Influence of Concentration and Temperature on Bodies containing the Amido Group.*—The proportion of amides to non-amides is a constant for different concentrations and temperatures.

When malt is poured into boiling water, the insoluble albuminoids or amides, are converted into soluble albuminoids, which are mainly composed of peptones.

—O. F. R.

*"Maltol," the Substance obtained from Crystallised Malt which gives, apparently, the Salicylic Acid Reaction.* Braud. Wochenschr. f. Brauerei 11, 1894, 514.

It has been previously shown that high dried and crystallised malts contain a substance which gives with ferric chloride a violet coloration similar to that afforded by salicylic acid. In this communication the author has isolated this substance, which he calls "Maltol," by extracting worts and beers, made from crystallised malts, with ether; "Maltol" is also found in pale malts to a small extent.

"Maltol" is a crystalline substance, which dissolves easily in water and all the usual organic solvents except petroleum spirit. It is not a true acid, although it dissolves in alkali and is precipitated by acid, but has rather the properties of a phenol; it also reduces Fehling's solution. The author gives it the formula  $C_6H_6O_3$ , and supposes it to be formed from the carbohydrate  $C_6H_{12}O_6$  by loss of  $3H_2O$ .

—O. F. R.

*The Detection of Boric Acid, especially in the Ash of Wines.* P. Kulisch. Zeits. angew. Chem. 1891, 147.

See under XXIII., page 904.

*Note on the Valuation of Potashes.* O. Bernheimer. Chem. Zeit. 18, 1894, 742.

See under XXIII., page 905.

*The Detection of Coal-Tar Dyes in Wines.* L. Sostegni and F. Carpentieri. Le Staz. sperim. agrar. ital. 1894, 26, 151.

See under XXIII., page 906.

*The Determination of Starch in Pressed Yeast.* F. Filsinger. Chem. Zeit. 18, 1894, 742.

See under XXIII., page 908.

## PATENTS.

*Improvements in Apparatus for regulating the Temperature of Fermenting Liquids.* E. N. Collios, Great Yarmouth. Eng. Pat. 16,692, September 5, 1893.

AN arrangement of horizontal flattened tubes, communicating at each end with a water space, is supported by means of tubular trunnions on tubular pedestals within the vessel containing the fermenting liquid. The cold water passes up one pedestal into the adjacent water space, then alternately to and fro through the tubes to the opposite water space, and thence out through the pedestal. The mounting on trunnions enables the apparatus to be readily cleansed.

—J. G. W.

*Improvements in Apparatus for the Manufacture of Beer.* A. W. Billings, London. Eng. Pat. 17,912, September 23, 1893.

THE converting vessel described in Eng. Pat. 8952, 1892, is provided with a heavy iron or steel bottom in which are the usual discharge openings. Over these is fixed a moveable slotted or perforated straining plate. The inventor claims that the apparatus may be used as a converter, boiling kettle, hop back, or cooler. For use as a converter the straining plate is removed and the mash made in the usual way and discharged into the filter tub. The straining plate having been fixed in position the clear liquor is now pumped back again, the hops added, and steam passed into the jacket so as to raise the contents to boiling. The author has found it advantageous to keep the stirrers in motion during boiling. Steam is then shut off and cold water run through the jacket to cool the wort, which is then strained off from the hops, leaving them in a suitable position for reboiling with fresh wort.—J. G. W.

*Improvements in the Treatment of Hops.* J. T. Connell, Edinburgh. Eng. Pat. 19,043, October 10, 1893.

It has been found that after hops have been employed in brewing they are not "spent," as is supposed, but may be revived so as to be fit for use a second time in brewing beer. They may in fact be "recured" by heating them suitably in a hop or malt kiln in the same manner as freshly picked hops, and it is claimed that they can then be used instead of new hops in brewing if a greater proportion be employed.—J. G. W.

*Aerated Wines and Spirits.* J. W. Fenn, London, and E. Abraham, Ripon. Eng. Pat. 4086, February 26, 1894.

CARBONIC acid gas is forced under pressure into wines or spirits, and is claimed to effect an improvement in quality and flavour.—J. G. W.

*Improvements relating to the Manufacture of Sugars, Syrups, Alcohol, and Beer.* H. H. Lake, London. From A. L. Tedesco, Paris. Eng. Pat. 9054, May 7, 1894.

THESE improvements are claimed to obviate the slight caramelisation which takes place during the saccharification of starch or amylaceous substances under high steam pressure. In the inversion of starch by acids, air is forced into the saccharifying vessel through the body of the contents, the temperature of which is not allowed to rise above 120° C. For converting worts by the agency of malt diastase the necessary quantity of malt is added to the wort, which is at a temperature of 60° C., contained in a closed saccharifying vessel, air or compressed gases being then forced into it. The employment of nitric acid in small

quantity under high pressure is also a ready method of inverting amylaceous materials. The amylaceous wort, to which one-half per cent. of nitric acid has been added, is liquefied by steam at a temperature of 100° C. The steam is then shut off and air forced into the vessel. When saccharification is complete the liquid is freed from acid by concentration in a vacuum apparatus.—J. G. W.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY AND WATER PURIFICATION, DISINFECTANTS.

### (A.)—CHEMISTRY OF FOODS.

*Discrimination of Drinking Waters.* D. Vedrádi. Chem. Zeit. 18, 1894, 585–586.

THE activity lately shown in certain districts of Hungary in the construction of deep wells has enabled the author to observe the interesting fact that the waters drawn therefrom were almost without exception of a nature usually considered as impure, containing, as they did, large amounts of organic matter, nitrates, ammonia, and in many cases even nitrites. The possibility of such contamination being derived from the influx of impure surface-waters was entirely negated by the nature and extent of the strata (mostly clay) passed through. Recent boring operations at the town of Debreczin put the author in a position to throw some light on the question. Although the depth attained was 840 metres, water was not found. Chemical examination of samples of the dry clay, sand, &c., excavated at various depths revealed the fact that organic matter (0.009 to 0.21 per cent., estimated by permanganate) was present at all depths, the maximum occurring at 650 metres. Chlorine and nitrous acids were found generally only in more or less strong traces; whilst ammonia and nitric acid were present in large amounts down to a depth of 200 metres: below that in traces. It is obvious, therefore, that the impurities in the class of water under consideration are derived from the soil itself, and are not due to pollution by sewage, &c. The author is of opinion that it is inexpedient to judge the fitness of such waters for drinking purposes by the usual standards; and that too much stress should not be laid on the presence of—otherwise inadmissible—proportions of organic matter, &c.—H. T. P.

*Poisonous Fodder Plants.* John R. Jackson. Chem. and Druggist, 1894, 267–268.

*Indian Peas (Lathyrus sativus).*—Artier has discovered the presence in the seed, of a volatile liquid alkaloid probably produced by some proteid ferment. The toxic action of the seeds was found to be destroyed by heat. The chief symptom is paralysis of the extremities, which in many cases becomes permanent.

*Sophora Secundiflora.*—A narcotic poison is found in the seeds of this species. It is a small tree or tall shrub growing in Mexico or Texas, forming dense thickets on the borders of streams. The seeds are rather larger than a haricot-bean and of a bright red colour. The Indians in the neighbourhood of San Antonio use them as an intoxicant. Half a seed produces exhilaration followed by narcosis extending over 2–3 days, whilst a whole bean is fatal. The principal is an alkaloid—*Sophorine*.

*Gastrolobium.*—Several species are commonly known in Australia as “Poison Bushes,” especially *G. bilobum*; *G. calycinum*; *G. callistachys*; *G. ocylobioides*; *G. spinosum*; *G. trilobum*; and *G. obovatum*. Large numbers of cattle are annually lost in Western Australia through eating these plants. The first symptom is *dyspnea*; the animals then stagger, fall, and die. The blossoms are most saturated

with the poison, though the young shoots even are poisonous. Sheep and cattle are killed by it, whilst wild pigeons eat blossoms or leaves with impunity.

*Gastrolobium grandiflorum* is a species found in Queensland and Northern Australia. It is known as the “Desert Poison-bush.”

*Gompholobum uncinatum.*—A leguminous plant, nearly allied to the *Gastrolobiums*. It is a small shrub of New South Wales, and is very poisonous.

*Tiphosia purpurea* and *T. rosea*, leguminous plants of South Australia, New South Wales, and Northern Australia. Also poisonous.

*Swainsonia.*—Several species of this kind have an evil reputation in Australia. The active principle has not yet been isolated. On drying the plants it is decomposed.

*Swainsonia galegifolia*, the “Darling Pea,” or “Indigo plant,” of Australia, is one of the plants most dreaded by stock-owners.

*Swainsonia Grayana* causes even more serious effects. The principle remains yet to be isolated.

*Trachymen australis*, an umbelliferous plant of Australia, known as the “Wild Parsnip.” It is terribly powerful, and no remedial measures are known.

*Sarcostemma australe*, the “Caustic Vine” of Queensland, is a plant belonging to the Asclepiadaceæ. Very poisonous.

*Erenophila maculata.*—Known in Queensland as the Native Fuchsia. Belongs to the natural order Myoporinæ. To this plant, it is said, though at first poisonous, stock may become accustomed. It is said to be more powerful after rain, and most dangerous when in fruit.

*Euphorbia Drummondii*, the “Caustic Creeper,” or “Milk Plant,” of Queensland. If eaten by sheep in the early morning before the sun has dried it up, it is almost certain to prove fatal.

*Stypandra glauca.*—A liliaceous plant, common around Sydney, the Blue Mountains, and other parts of New South Wales. It is known as the Candy Poison. It acts upon the eyes and causes blindness, but is not very fatal. The effects are known in Western Australia as “blind disease.” —W. S.

*Further Note on the Determination of the Fat in Bread.* M. Webull. Zeits. angew. Chem. 1894, 199.

See under XXIII., page 908.

### PATENTS.

*Improvements in the Manufacture of Gelatin.* E. S. Spencer, London. Eng. Pat. 14,491, July 27, 1893.

GELATIN in the form of sheets or in other solid, dry state is granulated or crushed, and, after the fine powder has been separated, the coarse granules are rolled between smooth rollers in such a manner that each granule is flattened into a thin flake. The object of the process is to reduce the cost of the manufacture of gelatin in a readily-soluble form, and to render the gelatin better adapted for resisting climatic influences.—A. G. B.

*Improvements in the Treatment of Eggs.* J. Fordred, London. Eng. Pat. 15,762, August 19, 1893.

THE essential feature of this invention is the use of sugars (preferably glucose, maltose, dextrose, &c.) under certain conditions for the preparation of the contents of eggs, in such a state which will enable the compound to be kept for a considerable time of uniform consistency, without deterioration, and readily miscible with water and other liquids. The yolks and whites of 1,200 eggs are carefully removed from the shells and well mixed or beaten; 100 lb. of this are placed in a pan fitted with stirring apparatus. This pan is fitted into a water-bath, the temperature of which should be from 160° to 170° F., so as to maintain the contents of the pan at about 145° F.; 50 lb. of sugar are then gradually added and stirred. The heat and

stirring is now continued until the mixture has lost 30 per cent. of its weight in five hours, when a deep and rich orange colour is produced. It is then strained through a hair sieve, and when cold, should be translucent and fit to be packed. The yolks or whites can be treated separately in the same way. It is also proposed to mix 3 parts of the above compound with 2 parts of condensed milk of commerce.—J. J. K.

*Improvements in Extracting Oil from Oil-containing Plants and Fruits, and in the Production of Fodder from the Residual Matter.* W. Spindler and C. Stautz, Darmstadt. Eng. Pat. 16,987, September 9, 1893.

See under XII., page 893.

*An Improved Food.* C. Morfit, London. Eng. Pat. 17,636, September 19, 1893.

This invention relates to the improvement of skim milk, whereby its deficiency in fat is restored. 100 gallons of the milk with 50 to 200 lb. of white sugar are boiled in a vacuum pan to one-third or even one-fourth of its bulk. The mixture is transferred to another pan, and 1½ to 2 oz. of refined cotton-seed oil are added, and the whole stirred until thoroughly blended. This artificial milk is termed "lactola." The admixture of coffee, cocoa, tea, or extract of meat with "lactola" is also claimed.—J. J. K.

*Improvements in and in Apparatus for the Preparation or Treatment of Alimentary Substances.* E. Maris, Monancourt (Eure), France. Eng. Pat. 17,732, September 20, 1893.

THE object of this invention is the preparation, in a highly-assimilable condition, of alimentary substances suitable for consumption by the human race and animals. To do this the substances are submitted to a preparatory treatment by crushing and artificial digestion, or reproducing as nearly as possible the different phases of natural digestion. The ground substance is put into digesting troughs together with three or four times its weight of water slightly acidified with hydrochloric acid, and sea salt, after which the necessary ferment (as pepsin, trypsin, invertin, amylase, and mineral ferment from grains, cellulose, &c.), to the extent of 5 per cent., are added, and the contents kept at a temperature of 35° C. for 12 hours, after which they are transferred to a digester, in which they are allowed to remain for two hours under steam pressure. The temperature is first raised to 65° C., and finally to 130° C. to destroy ferments and germs. The composition of a fodder, made from wood in this way, and which resembles that of good meadow hay, is—

Water.....	13.00
Albuminoid matter.....	8.74
Fatty matter.....	2.62
Non-nitrogenous extracts.....	38.64
Ligneous matters.....	31.00
Mineral matters.....	6.00
	<hr/> 100.00

—J. J. K.

*Process for Manufacturing a Food which increases the Production of Milk with Milk Animals.* T. Kluten-tröter, Magdeburg, Germany. Eng. Pat. 8360, April 27, 1894.

THE claim is for the manufacture of a milk-increasing food, by mixing 80 parts of basic phosphate of lime with the following mixture of 2½ parts of albumin, 2½ parts of yolk, and 15 parts of powdered sugar. The mixture is then ground. A daily dose of 75 grms. of this food are said to raise the production of milk by two litres.—J. J. K.

*Improved Means for Production of Effervescent Beverages.* R. Wichmann, Hamburg. Eng. Pat. 12,503, June 27, 1894.

"A CONTINUALLY acting effervescent mixture, consisting of comfit grains of desired size according to the duration of the effervescence." One half of the mixture is a salt, like bicarbonate of soda, enclosed within a layer of sugar. The other half consists of an acid, like tartaric or citric acid, similarly enclosed.—J. J. K.

## (B.)—SANITARY CHEMISTRY AND WATER PURIFICATION.

*Hermite's Process for Purifying Drainage Waters by Electricity.* Elektrotechn. Zeits. 1894, 15, 84.

A COMMISSION appointed by the "Conseil Central d'Hygiène" of Havre to investigate the above process, report that sterilisation is not effected rapidly enough to serve as a guarantee of its efficiency for disinfecting drainage waters. The process appears to be capable of destroying the unpleasant odour of certain chemical products.—A. R. L.

## PATENTS.

*Improved Antiseptic and Preserving Agents.* P. Håkansson, Esköf, Sweden. Eng. Pat. 10,465, May 27, 1893.

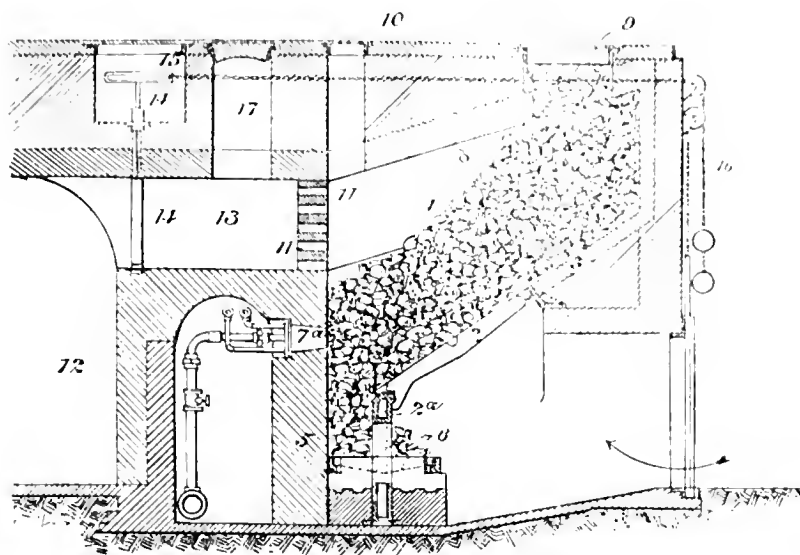
A MIXTURE of acetic ether and alcohol, with or without acetic acid, constitutes the new antiseptic and preserving agent called "salubrine." Suitable proportions are: acetic ether, 10—40 per cent.; alcohol, 20—50 per cent.; acetic acid, 2—3 per cent.; water, 68—7 per cent. When the above percentages are 25, 50, 2½, and 22½ respectively, the preparation is suitable for definite purposes, and is termed "salubrine A." A number of variations in strength for special purposes is quoted. Salubrine may be evaporated in a room to be deodorised; it may be used as a hair remedy, in which case aromatic oils are added; or it may be used as an insecticide to be applied to plants, in which case the acetic acid is omitted; when it is applied to the skin, glycerol, oil, or fat should be added.—A. G. B.

*Improvements in Furnaces for Burning Refuse Material, such as Town's Refuse.* J. B. Alliot and J. McC. C. Paton, both of Nottingham. Eng. Pat. 15,717, August 18, 1893.

THE figure shows a sectional elevation of this destructor furnace in which 1 is the main combustion chamber, 2 the firegrate, the lower end of which is carried on a hollow bearer 2a, through which water is circulated to keep it cool. The lower grate 6 is of sufficient length to prevent clinker, &c., which fall on to it from the upper grate, from falling over its front end. The clinker, &c. remains on this lower grate until more completely burnt and partially cooled, when it is raked off over the front end. 7, 7a, and 7b are tuyères through which air, in addition to that entering through the firebars, is forced; 7 and 7b, on either side of 7a, are not visible in fig. 3 is a drying-hearth on to which the refuse to be burnt is fed through the aperture 9. The stoking-hole 10 permits the introduction of an iron bar for keeping the grate and back wall 5 from getting clinkered up. The products of combustion are drawn through the openings 11, 11, which are at the hottest part of the fire, into the main flue 12, through an intermediate chamber 13, fitted with a damper 14. Three modified forms of the destructor are also described. The claims made in a furnace of this kind are for the combustion chamber, having a hearth so inclined that refuse and clinker thereon will move downwards automatically or with little help; for the fire grate, similarly inclined, and through which air is admitted to the combustion chamber; and for the back wall with inlet or inlets to admit air, to aid that admitted

through the inclined grate; also an opening or space behind the back wall and the adjacent end of the inclined

grate. Provisions for feeding refuse on to the upper grate, and for introducing air into the combustion chamber



FURNACE FOR BURNING REFUSE MATERIAL.

opposite to the feed end, as well as provision for the escape of products of combustion from the hottest part of the furnace, are further claimed. Finally, the intermediate chamber 13, as arranged, is claimed.—R. B. P.

**Improvements in and Retort Apparatus for Treating Town Refuse or Excrementitious or like Matter, and obtaining useful Products therefrom.** J. Hanson, Wakefield. Eng. Pat. 16,368, August 30, 1893.

According to this invention, a closed retort or vessel is provided, into which the refuse to be treated is introduced, and in which it is subjected to heat by which it is converted into an inodorous and innoxious carbonaceous residue (which may be stored, and is suitable for fuel) and an inflammable gas, which may either be stored in a holder and used for heating purposes as required, or at once used for firing the retorts.

The patentee proposes to use vertical retorts fitted at the top with hoppers for receipt of the refuse, and shoots at the bottom for the discharge of the carbonaceous residue. Each retort is furnished with means whereby the contents may be stirred during heating and adherence of carbonised matter to the sides thus prevented. This stirring apparatus may consist of a shaft running through the retort and furnished with arms, where the retort is charged and then closed during treatment of the charge; or of an Archimedean screw where (as is preferred) the refuse is supplied, treated, and expelled continuously. A steam jet may be introduced into the retort to produce a partial vacuum and thus help the operation or to aid in the production of inflammable gas, or a fan or other means of producing a reduction of pressure in the interior of the retort may be employed.—L. T. T.

**Improvements in the Purification of Sewage.** L. Archbutt and R. M. Deeley, Derby. Eng. Pat. 18,416, October 2, 1893.

THE effluent from a plant in which sewage is treated by the methods described in previous patents (this Journal, 1892, 595; 1893, 942) still contains albuminoids in solution, which are incapable of removal by chemical precipitation, but are destroyed by the agency of the nitrifying organism and other bacteria. The improved process is carried out as

follows:—The impure water is mixed in a tank with the chemicals necessary for precipitation, agitated with the precipitate from previous operations, and allowed to settle; the clear liquid is then transferred to another tank, treated with fuel gases in sufficient quantity to convert all free alkali into bicarbonate, and turned on to a filter bed. This treatment with fuel gases prevents the clogging of the filter bed and promotes the growth of micro-organisms. The filter bed consists of a layer of 8 inches of coarse gravel in which drain-pipes are laid, 5 inches of fine pea-gravel, and 5 feet of coarse mortar sand; it is of such an area that the whole contents of the carbonating tank shall have drained away before the next contents are ready for filtration; in this way the filter bed will be kept well aerated.—A. G. B.

**Improvements in Means for Treating the Refuse of Inhabited Areas.** R. C. Sayer, Bristol. Eng. Pat. 1452, January 23, 1894.

THE object of this invention is to treat refuse by heat so as to render it innocuous, and to utilise the residues. The refuse is treated in closed destructor furnaces, with or without the addition of fuel, whereby a gas is obtained. Part of the furnace-heat is used for generating steam, which drives an air compressor. Part of the compressed air from this is heated and injected through tuyères into the furnace, whilst another part, together with some of the gas from the furnace, is circulated through pipes to the houses in the district served by the plant. The gas and air mixture is supplied to and utilised in "cabinets," in which the solid and liquid human excrement is separated. The excrementitious matter is reduced by heat raised by burning the gas, and by the air, until the residue is drawn away by suitable means through pipes. The residue unites with the foul air from rooms, and with the products of combustion from house fires, after they have been purified with water, and is drawn back to the destructor along suitable conduits. It is further reduced in these by jets of gas and air. The dust residues obtained are used as manure, and the gaseous ones, after being washed with water, pass through a chimney into the atmosphere. The purifying water is used again after filtration through a combustible material. The latter, when saturated, is burnt in the destructor as fuel.—R. B. P.

*An Improved Method to Precipitate the solid Animal, Mineral, and Vegetable Matter contained in Sewage Water.* A. Mosley, London. Eng. Pat. 11,484, June 13, 1894.

When water which is colder than the sewage to be treated, is thrown upon the surface of such sewage water, the suspended matters settle to the bottom more rapidly than would otherwise be the case. To apply the principle, the sewage, to which chemicals have been added, would be warmed by hot-water pipes during its flow through the culvert leading to the settling tanks, and when it had arrived in these tanks, cold water would be sprinkled or poured on its surface.—A. G. B.

### (C.)—DISINFECTANTS.

#### PATENTS.

*Improvements in the Process of Obtaining Aqueous Solutions of Phenols and other Substances.* L. O. Helmers, Hamburg, Germany. Eng. Pat. 20,245, October 26, 1893.

By the action of sulphuric acid upon the sulphuretted or non-saturated constituents of mineral and similar oils, such as are described in Eng. Pat. 19,502, 1890, sulphonated carbon compounds of a nature similar to ichthyol, thiol, tumenol, and like substances are obtained; these or their alkali salts are soluble in water. Such sulphonated carbon compounds consist of two different substances, of which the one (sulphonate) is *per se* soluble in water, whilst the other (sulphone-like body) is *per se* insoluble in water (compare Eng. Pat. 6044, 1893). The separation of these substances is effected either by extracting the former with alcohol or the latter with benzene. When the sulphone-like bodies are mixed with the sulphonates they become soluble in water. From this the patentee inferred that these sulphonates, made by neutralising the reaction-product obtained by treating mineral or similar oils with concentrated sulphuric acid or its substitutes, would enhance the solubility of other substances which are themselves insoluble in water. This proved to be the case.

To render a solid soluble by means of these sulphonates, both the solid and the sulphonate must be dissolved in alcohol, the alcoholic solutions must be mixed, and the alcohol distilled off. Among substances thus rendered soluble are cresol, guaiacol, thymol, and other phenols; many hydrocarbons, such as benzene, terpenes, and mineral oils; ethereal oils; camphors; iodine; nitrobenzene; aniline. The quantity of pure sulphonate required to render such a substance soluble depends on the nature of the substance; thus an equal quantity will suffice to render the cresols soluble, whilst benzene requires a threefold and camphor a tenfold quantity. When the mixture obtained by neutralising the product of the action of concentrated sulphuric acid on an oil is used, a much larger quantity is required for the purpose of rendering substances soluble, because the solvent action is already partially satisfied by the sulphone-like substance which is present.

The products thus prepared are claimed as therapeutical and disinfecting agents.—A. G. B.

*An Improved Disinfecting Apparatus.* H. K. Andersson and D. Sjöström, Stockholm. Eng. Pat. 10,328, May 28, 1894.

THE apparatus consists of a cylindrical receptacle of sheet metal with a much smaller central cylinder. The outer cylinder is filled with water, and a red-hot and closely-fitting plunger is introduced into the inner cylinder. When this plunger becomes cooled it is removed, re-heated, and reintroduced, and this is repeated till the water boils. The steam then issues from an opening to which is affixed a hose and jet, so that it may be directed to any point. The volatile disinfectant is dissolved or suspended in the water. In the second form the disinfectant is introduced into the

inner cylinder and is volatilised by the plunger, which in this case only fits loosely and is pointed at the bottom. The vapour so generated issues from a jet in the side of the inner cylinder, which terminates in the centre of the steam outlet from the outer cylinder. The steam thus produces suction on the inner cylinder, and it is claimed that a thorough admixture of the disinfectant with the steam is effected.—L. T. T.

*Improvements in Disinfecting and in Apparatus therefor.* S. Pitt, Sutton, Surrey. From La Société Anonyme de l'Institut Raoul Pictet, Fribourg. Eng. Pat. 11,097, June 7, 1894.

THE patentee introduces the articles to be disinfected into a large vessel capable of resisting atmospheric pressure. This vessel is then exhausted by means of an air-pump, and sulphurous acid, chlorine, or the vapour of any desired antiseptic introduced. These vapours or gases are left to react for a certain time on the articles to be disinfected, and are then again exhausted and led into a gas-holder for use in a subsequent operation, or, if vapours, they may be condensed. Air is now admitted into the disinfecting vessel and the articles removed. The patentee claims that disinfection is more thorough from the better permeation of the disinfectant, whilst the articles disinfected are less injured than is now usual.—L. T. T.

*A New or Improved Fumigant for Use in Horticulture! Buildings.* T. Smithson, Bristol. Eng. Pat. 12,628, June 29, 1894.

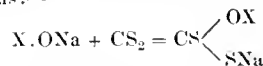
FIFTY parts tobacco waste, 3 parts cayenne pepper, 6 parts hellebore, 3 parts asafetida, 1 part lampblack, 5 parts sago flour, and 9 parts saltpetre are all finely ground and then thoroughly and intimately mixed and made up with a little water into a thick paste. This paste is either rolled and cut into shapes or moulded into convenient biscuits, and then dried in an oven. The patentee claims that this gives a fumigant which, whilst destructive to parasitic insects which infest vegetation, such as green-fly, thripp, red spider, and the like, may be used without injury to the most delicate plants.—L. T. T.

## XIX.—PAPER, PASTEBOARD, Etc.

*Commercial Cellulose Derivatives.* C. Beadle. Chem. Trade J. 1894, 128—129.

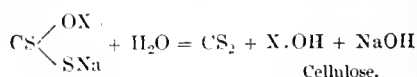
ONE of the most interesting class of cellulose derivatives is that of the thiosulpho-carbonates discovered by Messrs. Cross, Bevan, and Beadle. C. Beadle gave a communication to the Chemical Section of the Franklin Institute on this subject. (See also this Journal, 1893, 498—500.)

When alkali cellulose is treated with carbon-bisulphide vapour, at the end of three hours the mass will have changed to a deep golden yellow. This, when treated with water, swells up to a gelatinous mass and finally dissolves, yielding a very viscous solution resembling treacle. The reaction is expressed thus:—



Alkali cellulose. Cellulose xanthate.

where X represents the reacting unit of cellulose. The cellulose xanthate is soluble in water. This solution as it evaporates decomposes with a reversal of the above reaction as follows:—





Those solutions are most permanent which contain the smallest quantity of water. Sulphuric and most mineral acids at once precipitate the cellulose with evolution of  $\text{H}_2\text{S}$ . Acetic and many weak organic acids evolve  $\text{H}_2\text{S}$  without precipitation of cellulose, the solution becoming nearly white and colourless, but much more liable to rapid coagulation. Bisulphite of soda or sulphurous acid decolorises the solution without the evolution of sulphuretted hydrogen, if carefully added. Zinc sulphate or chloride precipitates the zinc salt in horny flakes, which can be washed and easily re-dissolved in weak soda. Alcohol or brine precipitates the cellulose as thiocarbonate, which can be washed free from by-products and dissolved in water. The solution is very viscous, and viscosity attains a maximum when treatment with  $\text{CS}_2$  immediately follows mercerising. Thus, when cotton is used in a 7 per cent. solution a viscosity equal to that of glycerol is attained, measured by rate of flow.

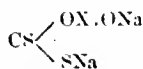
Increased temperature induces coagulation of the solution; thus, a solution that would last 40 days at 35° F. would coagulate in 25 days at 47° F., or in six days at 65° F. When a solution is spread on the surface of glass and exposed to a temperature of 250° F., coagulation only takes a few minutes. The mass, when washed free from by-products, comes off the surface as a transparent film.

Mass has a great effect on coagulation; a small sample of 30 grms. coagulates much quicker than a 400-lb. lot of the same solution, when both are kept at the same temperature. Coagulation is induced by exposure to air, probably due to the presence of carbonic acid, which aids dissociation of the cellulose thiocarbonate by forming sodium carbonate and liberating  $\text{CS}_2$ .

A solution once coagulated furnishes a substance which, although retaining the water required for its solution, is no longer soluble. If a coagulum contains much under 10 per cent. of cellulose it will contract on itself, retaining the form in which it was cast, and separating from the water and soluble by-products till it contains about 10 per cent. of cellulose and 90 per cent. of water. But if the coagulum contain a high percentage of cellulose it will expand and take up water.

This coagulum is not dehydrated by the action of alcohol. When the washed coagulum is taken out of the water and placed in air the mass contracts on itself until the whole of the water is eliminated. The resulting material is a hard, horny substance with a specific gravity of 1.5.

When a crude solution is treated with benzoyl chloride the cellulose is eliminated as a cellulose benzoate. Hence it may be right to ascribe the following formula to the compound:—



wherein X represents a cellulose residue of variable molecular weight, reacting as an alkali cellulose.

**Commercial Production.**—In preparing large quantities of this cellulose compound, a moist, disintegrated cellulose is taken, and incorporated with strong caustic soda solution by agitation under considerable pressure. The cellulose, when acted upon by the soda, swells up and absorbs all the solution, becoming flaky. This is passed through a sieve in order that it may be of uniform density and occupy as much bulk as possible. This process takes about 30 minutes, and 150 lb. of mercerised product is obtained at each mixing. One cubic foot of the material ready for treatment with the  $\text{CS}_2$  weighs about 15 lb. This is brought in contact with  $\text{CS}_2$  vapour when the mass has been carefully raised to 80° F. and kept in agitation. The reaction develops enough heat to raise the temperature to 105° F., when at the end of one hour the reaction is complete. The material still retains its former structure, but has changed in colour to a golden yellow. It is discharged into, and carefully mixed with, its own volume of water. In seven hours the mass has become a homogeneous stiff dough. To obtain a clear solution from this, it is passed, together with its own weight of water, through a centrifugal pump, and to free it from mechanical impurities it is passed under pressure through a filtering medium. The amount of caustic soda required is expressed

in its simplest terms by the ratio  $\text{C}_{12}\text{H}_{10}\text{O}_6 : 2\text{NaOH}$ . The proportion of  $\text{CS}_2$  used is expressed by the ratio  $\text{CS}_2 : 2\text{NaOH}$ .

The most favourable conditions for the interaction are:  $\text{C}_{12}\text{H}_{10}\text{O}_6 : 2\text{NaOH} : 2\text{CS}_2 : 35\text{H}_2\text{O}$ .

**The Production of Continuous Films.**—When dehydrating agents, such as brine or alcohol, act on the cellulose thiocarbonate solution, the effect produced is dependent upon the constitution of the solution. Thus, if a fresh solution be used and brine added, the flocculent precipitate obtained is entirely redissolved in water. If the solution has been made some days and approaching the point of coagulation, the precipitate so formed is during precipitation converted into insoluble cellulose. If some of this solution be spread as a varnish on a glass plate, and immersed in brine, the cellulose can in a few seconds be stripped off the surface in the form of a film. A machine has been devised which produces and reels a film in a continuous web. The thick solution, which has been kept till on the point of coagulation, is spread on the surface of a revolving cylinder, half immersed in a bath of brine. The solution is spread by means of an adjustable doctor, attached to side wheels which regulate the width of the film required. The film as it leaves the brine is stripped from the cylinder and passed through a series of vats for washing, decoloring, dyeing, and finishing. It can be dried by passage over steam cylinders and reeled. The film when in the web, before passing on to the drying cylinders, can be grained or stamped by rolls carrying designs in imitation of any surface. Cellulose films prepared by this method have an affinity for many aniline colours not shared by any other form of cellulose.

It becomes dyed so readily with some aniline colours without a mordant that the operation can be performed in the web as it passes over the machine. When dried and finished it becomes resistant and loses considerably its chemical activity.

**Commercial Production of Thick Films.**—(1.) From crude coagulum. In order to prepare thicker films the solid coagulum is cut up into pieces of any desired thickness. These are washed to free them from by-products, and treated for some time under pressure, and calendered. Calendering has a marvellous effect on the films, if they are not thoroughly dehydrated. The material of the film flows under great pressure, increasing the area and decreasing the thickness of the film, and the annealing it gets very much improves its texture and strength. It has recently been found possible to forego the process of washing out the by-products, and thus films sufficiently pure for some purposes have been obtained by the following process:—

(2.) From decolorised coagulum. To the crude thiocarbonate solution is added a weak solution of sulphurous acid, which has the effect of decolorising the by-products by converting them for the most part into sodium thiosulphate. The coagulum formed from this is a semi-opaque neutral jelly. This is cut up into pieces of the size required, and each piece is submitted to a process of annealing under pressure, which results in the dehydration and separation of the cellulose from the by-products. It has lately been found possible to pulverise the solid decolorised coagulum and cause it to go together into compact sheets under pressure; also to force several sheets together into one solid compact sheet. With all other mixtures failure had followed efforts to obtain a pulverised substance that would go together under pressure, when the above mixture was found to succeed. It is very difficult to explain this peculiar behaviour of a cellulose coagulum, and why the various mixtures should behave so differently. As a marked instance of what pressure will do in effecting a molecular change it has been found possible to stamp boxes direct from the coagulum, embossing and dehydrating them in one operation.

Films may also be produced by taking a continuous veneer from a revolving cylinder of the dehydrated substance.

**Inertness of the Dehydrated Product.**—This is well exemplified in its application to woven fabrics. Cotton and linen goods behave very differently in regard to their affinity for, and power of fixing, these cellulose derivatives. When the grey linen is passed through a weak solution of cellulose thiocarbonate it is easily fixed by



the film. It appears to penetrate the cell-wall and build itself up in the fibre. The cellulose so fixed is not removed by the various chemical treatments the cloth afterwards receives. The character of the solution used has a great effect on the finish of the cloth. It is possible to obtain a very marked difference in the feel by the addition of only 2 per cent., or to add to the cloth as much as 25 per cent., without making any marked difference. In all cases the cellulose derivative shows itself less acted upon by chemical treatment than the fibre of the cloth.

**Properties and Constitution.**—Where it reacts it does so in a different way from cotton and linen cellulose. Its carbon percentage is somewhat lower and its weight is somewhat greater than the cotton cellulose from which it is prepared. The original molecule appears to have undergone hydration in the ratio  $2C_{12}H_{10}O_{10}, H_2O$ . The hygroscopic moisture varies from 10 per cent. to 15 per cent., according to the method used for its production. It is turned blue by iodine solution.

## PATENTS.

**Improvements in the Manufacture or Treatment of Paper.** A. Yockney, Wilcove, Cornwall. Eng. Pat. 16,947, September 8, 1893.

THE inventor claims the use of boric acid or a compound of boron, such as borax, as a means for preventing paper from deteriorating in the hottest and dampest climates. The borax or similar compound may be added to the paper while in the pulp stuff condition, or after it is webbed and made into a sheet by soaking it in a solution of the acid or salt. A sizing is recommended consisting of 1 oz. of gelatin soaked in a quart of water, then boiled, allowed to stand for 24 hours, and reboiled. To this is added 1 oz. of boric acid solution, consisting of 20 grains of acid in 1 oz. of water. This size is said to be very suitable for high class papers, rendering them of a less perishable nature.

—S. P. E.

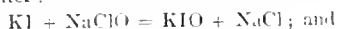
**Improvements in and relating to the Manufacture or Treatment of Paper.** E. J. Lushy, London. Eng. Pat. 18,538, October 3, 1893.

THIS invention consists in the treatment of paper, before or after webbing, with certain essential oils possessing medicinal and other beneficial properties. Creosote, oils of *pinus pumilio*, eucalyptus, and of any coniferous or resinous trees are recommended.—S. P. E.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

**Moist Iodoform.** G. Vulpius. Pharm. Central-II. N.F. 1894, 15, 259.

THE author has found in a sample of iodoform—otherwise normal—13 per cent. of water, and ascribes the fact to the practical difficulty experienced in drying this substance, especially when prepared according to one of the newer methods, viz., by allowing a dilute solution of sodium hypochlorite to trickle into a solution consisting of 50 parts of potassium iodide, 2 of caustic soda, and 2 of acetone in 2,000 of water:—



It will be well, therefore, in the examination of samples of iodoform, and in view of this possible method of preparation, to estimate the amount of water present. The author fixes the permissible maximum at 1 per cent.—H. T. P.

**The Occurrence of Copper in Extracts.** G. Hell. Pharm. Post. 1894, 27, 182.

THE author has examined numerous drugs and found in all of them copper in a soluble form. The requirement laid down in the pharmacopœia that extracts should be free from copper is, therefore, not tenable. As a matter of fact, all extracts contain copper as a normal constituent. The author suggests, therefore, that a maximum admissible limit be fixed upon, and some simple method be adopted for determining whether this limit has been exceeded.—H. T. P.

**Preparation of Alcohol from Apples.** Vivien and Dupont. Bull. Assoc. Chim. 11, 1894, 526 and 528.

IN view of the enormous harvest of apples this year, the authors have experimented as to the manufacture of alcohol from apples. 100 litres of apple juice weighing 106.4 kilos., contained 89.75 per cent. of water, 0.30 per cent. of ash, 2.04 per cent. of pectin bodies, and 2 per cent. of cane sugar, 2.97 per cent. dextrose, 8.50 per cent. levulose, and 0.84 per cent. of other sugars; total sugars, 14.31 per cent.

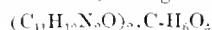
On adding phosphoric acid, potash, and ammonia (or sodium nitrate) the fermentation proceeds as quickly and completely as with turnip juice, and by this means 5 per cent. of alcohol is obtained from the apples. This alcohol was considered to be of better quality than the ordinary alcohol from cider. The grounds remaining compose 18 per cent. of the apples taken, and contain 2.5 per cent. of sugar.

—T. A. G.

**On  $\beta$ -Resalylin (Phenyldimethylpyrazolone  $\beta$ -resorecylate).**

A. Petit and A. Fèvre. Chem. Zeit. 18, 1894, 1094.

THE authors have isolated a compound by mixing concentrated aqueous solutions of one molecular proportion of  $\beta$ -resorecyllic acid with two molecular proportions of antipyrine. The new substance, having the formula—



crystallises from alcohol or acetic ether in colourless needles having a sweetish acid taste. It melts at 115° C., dissolves in 150 parts of cold and 20 parts of boiling water, and is easily soluble in alcohol, acetone, chloroform, and acetic ether, but insoluble in ether and petroleum ether. Its aqueous solution is acid to litmus, and when decomposed with soda lye, it yields sodium resorecylate and analgesine. Heated with phthalic anhydride to 220° C.,  $CO_2$  is given off and fluorescein is formed; whereas salipyrine, when similarly treated, gives a yellowish-red colouring matter. The new compound differs from resopyrine (resorcinol-antipyrine) in melting point and in the fact that it is precipitated by acids. Since, according to the analysis, two molecules of antipyrine combine with one molecule of  $\beta$ -resorecyllic acid, the reaction appears to be a function of the hydroxyl and not of the carboxyl groups, and the authors propose to investigate the behaviour of the ethers of phenol carboxylic acids in this direction. Gallic acid, which contains three hydroxyl groups, ought to condense with three molecules of antipyrine.—T. A. L.

**Cannabindon.** R. Kobert. Chem. Zeit. 18, 1894, 741—742.

A NEW substance obtained from Indian hemp (*Cannabis Indica*) by a process detailed at length, and consisting essentially in the successive treatment of the drug with various solvents, the insoluble portion (found in every case to be physiologically inactive) being at each stage rejected. The final product was a dark cherry-red syrupy liquid to which the name "Cannabindon" was assigned. Cannabindon is soluble in alcohol, ether, chloroform, benzene, fatty oils, &c., and has the composition expressed by the formula  $C_{11}H_{12}O$ . When heated with fuming sulphuric acid it dissolves, forming a product soluble in water with dark brown colour and an odour, both of which resemble those of a solution of lethyol. The aqueous

solution of *cannabindon sulphonic acid* when neutralised with soda yields precipitates with a large number of various metallic salts. *Cannabindon* reduces ammoniacal silver nitrate, alcoholic gold chloride, and Fehling's solutions; but is not itself appreciably reduced by treatment with sodium. Physiologically, *cannabindon*, in doses of 0.02 to 0.08 gm., exerts on human beings and cats a toxic action, which manifests itself chiefly in the shape of hallucinations, sometimes of a very disagreeable nature. By repeated use, however, the system becomes habituated to the drug. The substance does not appear to be particularly poisonous, for doses up to 1 gm. administered to cats did not produce fatal results.—H. T. P.

*The Influence of Alcohol, Glycerol, and Fatty Oils on the Bactericidal Action of Phenol and of Corrosive Sublimite.* M. Lenzl. Rev. d'hygiène et de police sanitaire, per Rep. Pharm. 1894, 111.

THE author confirms Koch and Wolffhügel's observation that phenol, when dissolved in alcohol or oil, has no antiseptic influence. The following conclusions are drawn:—Absolute alcohol destroys the bactericidal action of phenol and mercuric chloride on the spores of mildew. In the case of 0.1 per cent. corrosive sublimate solutions, an antiseptic action only becomes evident when the amount of water exceeds 2 per cent.; whilst 70 per cent. must be present in the case of phenol, and even then the destructive action is very slow, requiring 24–28 hours for its completion. A 2 per cent. solution of sublimate in glycerol requires, in order to become active, the addition of at least 4 per cent. of water, whilst 10 per cent. phenol in the same solvent must be diluted with 80 per cent. water. Phenol dissolved in oil entirely loses its disinfecting qualities.

—H. T. P.

*Note on Ipecacuanha.* J. Moeller. Pharm. Post. 1894, 27, 165.

*Carthagena Ipecacuanha*, also known as *Saranilla ipecacuanha*, has been obtainable for the past three years on the London and Hamburg markets. It is derived from *Cephaelis acuminata*, and is in every respect equal to the official Rio variety, from which it differs mainly in that it occurs in much larger pieces (6–8 mm.).

*False Carthagena Ipecacuanha.*—In opposition to Hartwich, the author is of opinion that this variety is identical with the true *Carthagena ipecacuanha*, and that the absence of alkaloid from the roots is attributable to the fact that they are not collected at the period of maturity. *False East Indian Ipecacuanha* occurs in grey and yellowish-brown sinuous pieces, 4–9 mm. in diameter. It possesses a feeble odour resembling that of the tonka bean. Its cross-section is white, floury, with a central, brownish, woody core, not sharply separated from the bark. The drug contains no emetin. Its derivation is, as yet, unknown, but judging from its structure it probably belongs to the *Aroideae*.—H. T. P.

*Quinaseptol or Diaphthol.* Guinard. Rép. Pharm. 50, 1894, 65.

QUINASEPTOL or Diaphthol is hydroxyquinoline-*m*-sulphonic acid. It consists of a powder which has a melting point of about 295° C., liberates free hydroxyquinoline on decomposition, and gives a green coloration with ferric chloride and a rose-red colour with uranium acetate. It possesses an antiseptic action, but its use is not permissible in surgical operations owing to its blackening action on the steel of surgical instruments. Sodium diaphthol is bright yellow and more strongly antiseptic than diaphthol itself, destroying many organisms in 30–50 minutes. On the other hand, diaphthol is not very poisonous, since 3 grms. were required to prove fatal to a dog of 1 kilo. in weight. The author believes that diaphthol should prove a valuable antiseptic for internal or external use.—T. A. G.

*A New Colour Reaction of Iridol.* E. Nickel. Chem. Zeit. 18, 1894, 531.

See under XXIII., page 906.

*The Examination of Liquid Extracts.* O. Linde. Pharm. Central-Bl. N.F. 15, 1894, 39.

See under XXIII., page 909.

*Poisonous Fodder Plants.* J. R. Jackson. Chem. and Druggist, 1894, 267.

See under XVIII. A., page 897.

## PATENTS.

*Production of Substances suitable for Manufacture of Perfumery and Aromatics.* J. C. W. F. Tiemann, Berlin. Eng. Pat. 17,539, September 18, 1893.

THE patentee has already described (this Journal, 1894, 272) the preparation of perfumes from pseudo-ionone, a substance formed by shaking a mixture of citral and acetone with barium hydrate. He now states that acetone may be replaced by its homologues, and the natural citral by artificial citrals obtained by the oxidation of the alcohols of the formula  $C_{10}H_{19}O$  and present in many etheral oils, such as geraniol, rhodinol, &c.; or by citronellone (citronellal), an aldehyde containing two more atoms of hydrogen, with equally useful technical results.—L. T. T.

*Production of Artificial Musk from Hydrogenated Aromatic Hydrocarbons.* A. Baur, Mulhouse. Eng. Pat. 17,703, September 20, 1893.

TRINITRABUTYLXYLENE, the artificial musk referred to in Eng. Pat. 4963 of 1889 (this Journal, 1890, 545), is obtained as follows:—108 grms. of hydrometaxylene are mixed with 500 grms. of carbon bisulphide and 92.5 grms. of butyl chloride (or equivalent weight of bromide or iodide), and about 20 grms. of aluminium chloride are gradually added. The butylated derivative formed is distilled with steam, and then fractionated, and the fraction 190–210° C. nitrated by introducing it into a mixture of one part fuming nitric acid and two parts fuming sulphuric acid containing 10–15 per cent. of sulphuric anhydride.—L. T. T.

*A Process for the Production of Artificial Musk.* A. Baur, Mulhouse. Eng. Pat. 17,781, September 21, 1893.

THE patentee has already shown (preceding abstract, and this Journal, 1889, 1004; and 1890, 545 and 964) that musk substitutes may be prepared by the nitration of homologues of benzene containing the methyl, ethyl, butyl, propyl, or amyl side groups. He now finds that many of the more complicated aromatic compounds on nitration yield the similar products, being split up during nitration. Thus dibutyliditolylmethane and tributyltritolylmethane yield trinitrobutyltoluene, whilst dibutylidixylmethane and tributyltrixylmethane give trinitrobutylxylene. Both of these compounds are crystalline substances smelling strongly of musk.—L. T. T.

*Improvements in Cinchona Preparations.* F. W. Fletcher, Enfield. Eng. Pat. 12,796, July 2, 1894.

THIS invention consists in the extraction of cinchona bark by hydrobromic acid, by which means an extract is obtained containing the whole of the alkaloids of the bark, together with the kinic, kinovic, and cincho tannic acids and the other natural and more or less undefined constituents of the bark, all of which possess valuable tonic, aromatic, and astringent qualities.

The extract is most advantageously prepared as follows:—The cinchona bark employed should be of the finest quality, and contain not less than 8–10 per cent. of total alkaloids. 15 lb. of this bark is ground to the degree of fineness technically known as No. 40. 27 fluid ounces of hydrobromic acid of sp. gr. 1.275 are diluted with distilled water to 6 gallons. The ground bark is thoroughly damped with half a gallon of this acid and left for six hours for the acid to permeate and the bark to swell. It is then packed in a displacement apparatus, and percolated with the remainder of the acid, and finally with distilled water till the latter extracts no more alkaloid. 30 fluid ounces of glycerin are added to the percolate, and the latter then evaporated to 240 fluid ounces. This concentrated extract is preserved in closely-stoppered vessels, 1 fluid ounce thereof containing all the soluble constituents of 1 ounce by weight of cinchona bark in intimate association and combination with bromine.—L. T. T.

### XXIII.—ANALYTICAL CHEMISTRY.

#### INORGANIC CHEMISTRY.—QUALITATIVE.

*The Detection of Boric Acid, especially in the Ash of Wine.* P. Kulisch. Zeits. angew. Chem. 1894, 147–148.

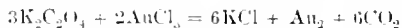
RUBER'S test for boric acid consists in dissolving the ash of 25–50 cc. of wine in 10 cc. of distilled water, adding 2 cc. of hydrochloric acid of sp. gr. 1.19, and testing with turmeric paper in the usual way. The present investigation shows that the delicacy of the reaction is affected chiefly by using unduly diluted solutions. Hence it is preferred that the ash should be treated with sufficient hydrochloric acid to render it distinctly acid, say by addition of 1–2 cc. hydrochloric acid of 1.12 sp. gr. The turmeric paper should be prepared from a strong solution of the dye. In dealing with small quantities, the colour may be best observed at the moment the paper is on the point of drying. The sensitiveness of the reaction is interfered with, more or less—even in an acid solution,—by the presence of salts.

—H. A.

#### INORGANIC CHEMISTRY.— QUANTITATIVE.

*The Volumetric Estimation of Gold.* G. B. Franceschi. Boll. Chim. farmac. 1894, 33, 35.

WHEN a gold chloride solution is boiled for some minutes with potassium oxalate in excess, the gold is all reduced and separates according to—



Evidently then, 8.3 parts of potassium oxalate precipitate 6.533 parts of gold.

The following are the reagents required:—

(1.) Solution containing 8.3 grms. potassium oxalate per litre.

(2.) Solution of potassium permanganate which is exactly reduced by an equal volume of potassium oxalate.

Oxalate in excess is added to the gold solution, and the mixture boiled for a few minutes. Cool, filter off the gold, and wash. The filtrate, after mixing with the washings, is rendered acid with sulphuric acid, and the excess of oxalate solution titrated back with permanganate. Then if—

$n$  = cc. of oxalate solution

$n_1$  = cc. of permanganate solution;

the gold reduced is represented by  $(n - n_1) 0.0065$ .

—T. A. G.

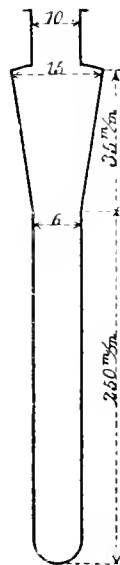
*The Estimation of Carbonic Acid in the Presence of Soluble Sulphides.* A. Wolkowicz. Zeits. angew. Chem. 1894, 165.

THE weighed quantity of the substance is treated in excess with a cupric chloride solution of 20 per cent. strength. The sulphur of the sulphides present in the substance thus becomes eventually transferred to the copper, forming the sulphide, insoluble in hydrochloric acid. Hydrochloric acid is next used to liberate the carbonic acid in the usual way. An ordinary Fresenius' apparatus is used, and the carbonic acid is absorbed by soda-lime.—F. M.

*Volumetric Estimation of Sulphuric Acid in Crude Commercial Hydrochloric Acid.* L. Rürup. Chem. Zeit. 18, 1894, 225.

COMMERCIAL hydrochloric acid always contains more or less sulphuric acid. When the latter amounts to about 1.5 per cent. of  $SO_3$ , it makes the hydrochloric acid unsuitable for use in preparing iron or steel articles for being galvanised. It causes black specks; that is to say, places where the zinc does not adhere to the iron. Hydrochloric acid containing more than, at the most, 1.0 per cent. of  $SO_3$  should not therefore be used. The author employs the following method of making a rapid estimation of the  $SO_3$  in hydrochloric acid. The results he obtains agree to 0.05 per cent. with gravimetric determinations.

He uses a glass tube similar to the tubes used in estimating phosphorus in iron and steel by the method of measuring the bulk of precipitate of phosphomolybdate



formed, and which is shown in the annexed figure. The tube is graduated by adding 0.4, 0.6, &c., up to 3 per cent. of sulphuric acid to portions of 10 cc. of pure hydrochloric acid. The acid is heated to boiling, rinsed into the tube with a little water, nearly neutralised with concentrated ammonia, and then the sulphuric acid is precipitated by a hot solution of barium chloride, the tube stoppered, and shaken for about two minutes. The precipitate of barium sulphate is then allowed to settle. Any particles attached to the side of the tube are detached by a glass rod with a piece of india-rubber tubing on the end; but such particles affect the result very slightly. A mark is made on the tube with a diamond at the level of the top of the precipitate, and the percentage of  $SO_3$  is also written on the glass at this mark with a diamond. By dividing the spaces between the marks, the percentage can be read to 0.05 per cent.

In carrying out a test, the author takes 10 cc. of hydrochloric acid out of every 10 carboys, by means of a glass tube, pours it into a small beaker, and nearly neutralises with

ammonia; then heats to boiling, and pours the liquid into the testing tube. About 5 cc. of boiling saturated solution of barium chloride are added, the tube closed with an india-rubber stopper, and shaken for two minutes, and after allowing the precipitate to settle, which takes about five minutes, the percentage is read off at the mark at which the level of the precipitate stands.—H. S. P.

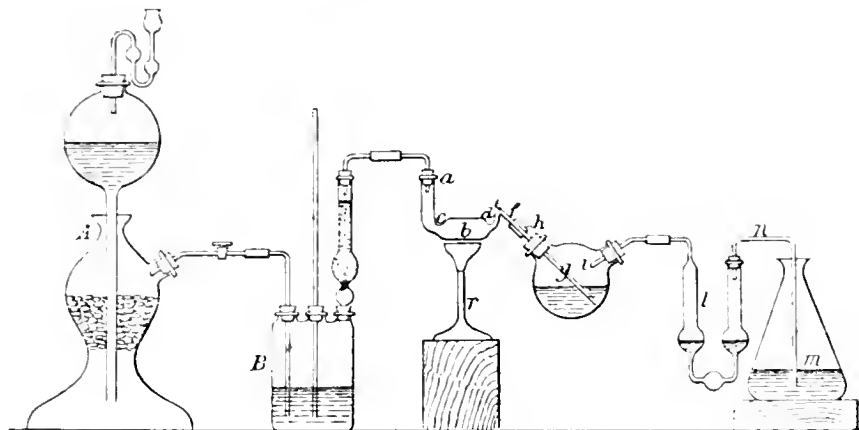
*Note on the Valuation of Potashes.* O. Bernheimer, Chem. Zeit. 18, 1894, 742—743.

It is usual to proceed as follows:—From the total potash (determined as platino-chloride) the amounts existing as sulphate, chloride, and, in accurate analyses, as phosphate and silicate, are subtracted, the remainder being calculated into potassium carbonate. Then, the total alkalinity of sample (by titration with acid), less the potassium car-

bonate, gives the amount of sodium carbonate. The author shows that this method of procedure may yield results erroneous to the extent of several per cent. in the case of potashes prepared from distillery residues, &c., which occasionally contain nitrogen acids (0.75 per cent.  $N_2$  or more) combined with potash. It is necessary to make an allowance for the nitrogen thus combined (estimated by Kjeldahl's method), bearing in mind that  $N_2$  is equivalent to  $K_2CO_3$ .—H. T. P.

*The Estimation of Sulphur in Sulphides, with Simultaneous Determination of Arsenic.* P. Jannasch, Zeit. anorgan. Chem. 6, 1894, 303.

In the annexed figure, which almost explains itself, the tube *b* measures 9.8—10 cm. in length from *a* to *c*; 6.2 cm. from *c* to *d*; and 3.5 cm. from *d* to *e*. The bulb *i* contains



a 3 per cent. solution of hydrogen peroxide mixed with 5 cc. of strong nitric acid; *l* and *m* contain the same mixture diluted with an equal bulk of water. The flask *m* is covered with a perforated watch-glass, and is capable of holding from 900 to 1,000 cc. of liquid. For the analysis, from 0.6 to 0.75 gm. of the very finely powdered substance are introduced into *b*; it is here heated by the burner *r* in a stream of oxygen which has been purified by passing first through strong sulphuric acid and then over soda-lime, in an apparatus substituted for *A* and *B* in the figure. With the  $SO_2$  some of the arsenic is sublimed, and the condensed oxide is driven by a second gas-flame into the small tube *d e*. When all the  $SO_2$  has passed over, the tube is cooled, and then immersed in a dish containing water at  $100^\circ C$ .; then *A* and *B* having been connected, a gentle stream of pure hydrogen chloride gas is led over the warm substance for  $1\frac{1}{2}$ —2 hours. The apparatus is then disconnected, the solutions from *i*, *l*, and *m* are mixed with a little strong nitric acid, to prevent loss of arsenic, and evaporated down to a bulk of 20—25 cc. This is next diluted to 200 cc., and a known quantity of barium chloride is added to the boiling solution (8—9 cc. of a 10 per cent. solution to 0.5 gm. of arsenical pyrites), and when cool it is filtered. After rendering alkaline with ammonia, magnesium chloride (4—5 cc. of a 25 per cent. solution for 0.6 gm. of substance) is added, and the arsenic determined in the usual way; citric acid should also be employed if alumina be present. If sulphur should ever be retained in the tube *b* as calcium or barium sulphate, it may be rendered volatile by passing over it, when heated, air charged with ammonium carbonate, whereby ammonium sulphate is formed and afterwards caught in the condensing tubes.—W. G. M.

*A Volumetric Method for Estimating Sulphuric Acid.* Windisch. Wochenschr. f. Brauerei, 11, 1894, 607.

This method consists in precipitating the  $SO_3$  in a solution of the substance to be analysed, with an excess of a standard

solution of barium chloride, precipitating the excess of barium chloride with an excess of standard potassium chromate and estimating the excess of potassium chromate by means of standard arsenious acid and iodine, the end reaction being determined by the use of starch paste.

The author prefers the use of  $\frac{N}{50}$  to the usual  $\frac{N}{10}$  standard solutions.

The method appears to give excellent results, and may be used in estimating the  $SO_3$  in water. 100 cc. water are evaporated with a few drops of HCl and treated with 50 cc.  $\frac{N}{50}$  barium chloride and 50 cc.  $\frac{N}{50}$  potassium chromate solutions; the volume is then made up to 300 cc. and 100 cc. filtered off and shaken with 50 cc.  $\frac{N}{50}$  arsenious acid and a few drops of sulphuric acid; starch paste is added and a  $\frac{N}{50}$  iodine solution run in till a permanent blue coloration is obtained.

—O. F. R.

*Method for the Volumetric Estimation of the Phosphoric Acid soluble in Water, contained in Superphosphates.* W. Kalmann and K. Meissels. Chem. Zeit. Rep. 18, 1894, 180.

THE method depends upon the different behaviour of the phosphates towards methyl orange and phenolphthalein as indicators. 20 grms. of the superphosphate are dissolved and made up to a litre. Methyl orange is added to 100 cc. of the filtered solution, which is then accurately neutralised with semi-normal alkali; then, after adding phenolphthalein, the same liquid is titrated with a further quantity of the alkali; the number of cc. of the alkali used is represented by *a*. A second quantity of 100 cc. is now mixed with an excess of the semi-normal alkali in a 250-cc. flask and filled up to the mark. To 100 cc. of the filtrate from this, phenolphthalein is added, and the liquid is neutralised with semi-normal acid; after the addition of methyl orange it is

further titrated with the acid. The number of cc. used (*b*) multiplied by 2.5, represents the amount required for 100 cc. of the original superphosphate solution. The arithmetical mean of *a* and *b* multiplied by 0.0355 gives the weight of  $P_2O_5$  in 100 cc. of the original solution or in 2 grms. of the substance.—W. G. M.

*The Solubility of the Phosphoric Acid contained in Bone Meals.* L. Gebek. Zeits. angew. Chem. 1894, 193.

See under XV., page 894.

## ORGANIC CHEMISTRY.—QUALITATIVE.

*The Detection of Coal-Tar Dyes in Wines.* L. Sostegni and F. Carpentieri. Le Staz. sperim. agrar. ital. 1894, 26, 151.

An improvement on Possetto's modification of Arata's method. 200 cc. of wine are boiled to expel the alcohol, and 2—4 cc. of 10 per cent. hydrochloric acid added. A few strands of white wool (perfectly free from fat) are then immersed in the liquid and the boiling continued for five minutes, after which the wool is washed successively with water, very dilute acid, and again water, and subsequently digested at a boiling temperature with 50 cc. of water and 2 cc. of ammonia. The ammoniacal liquid is acidified with hydrochloric acid, boiled with a fresh portion of wool, and the latter finally washed and dried. The following results were obtained from pure wine to which 2 mgrms. per litre of various dyes had been added:—

Dye employed.	Colour of Wool.
Vinolin .....	Rose-red to violet.
Bordeaux red .....	" "
Ponceau red .....	Rose-red.
Magenta .....	Dirty white.
Safranin .....	Faint pink.
Tropaeolin ol. ....	Straw yellow.
Tropaeolin oo. ....	Faint orange.
Corallin .....	Dirty white.

For the purposes of identification, the colouring matter may be afterwards extracted from the wool by dilute ammonia, the solution evaporated to dryness, and the residue submitted to the action of various reagents, such as strong acids, alkalis, potassium bichromate and sulphuric acid, &c. In applying the latter test it must be noticed that wool absorbs appreciable quantities of hydrochloric acid, which appears in the above residue as ammonium chloride, and might in some cases modify the colour reaction. 92 specimens of pure Italian wines were examined by the above process. The colour of the wool was generally pure white; frequently, however, dirty white or faintly yellow or orange; but never pink or red. Magenta and corallin are not fixed by the fibre under the above conditions; for these, therefore, the method yields negative results.—H. T. P.

*A New Colour Reaction of Iridol.* E. Nickel. Chem. Zeit. 18, 531.

IRIDOL,  $CH_3 \cdot C_6H_4(OH)(OCH_3)_2$ , has only recently been prepared by Tiemann. Iridol, like vanillin, gives a characteristic reddish-violet coloration with Millon's reagent. Now the author has shown that vanillin gives a violet colour reaction with mercuric chloride in presence of nitrites, and he finds that the same is true of iridol. The reagent employed consisted of a solution of mercuric chloride (2 parts) and

sodium nitrite (1 part) in water (40 parts). Solutions containing only one-fifth of the above amount of nitrite act most delicately with vanillin solution, but iridol solutions are not so sensitive. The aqueous alcoholic solution of iridol or vanillin is treated with about an equal volume of the reagent, and the mixture heated to boiling, when after some minutes there is developed a beautiful violet coloration having a shade of blue. There is no essential difference between the colorations produced by these two compounds.—A. R. L.

*The Behaviour of Triphenylmethane Colouring Matters to Nascent Bromine.* W. Vaubel. Chem. Zeit. 18, 1093.

See under IV., page 876.

*Properties of Sunflower Oil.* D. Holde. Mitt. aus. dem königl. tech. Versuchsanst. zu Berlin, 1894, 36.

See under XII., page 892.

*On  $\beta$ -Resalgin (Phenyldimethylpyrazolone- $\beta$ -resorcylate).* A. Petit and A. Fèvre. Chem. Zeit. 18, 1894, 1094.

See under XX., page 902.

*Detection of Disguising and Perfuming Media in Oils and Fats.* D. Holde. Mittheilungen aus den königl. tech. Versuchsanst. zu Berlin, 1894, 31—35.

NITRONAPHTHALENE,  $C_{10}H_7NO_2$ , is used to destroy the fluorescence of mineral oils, and nitrobenzene,  $C_6H_5NO_2$ , is employed to disguise any unpleasant or characteristic fatty smell. The latter may be readily recognised by its smell of bitter-almond oil. It is also worthy of note that strongly fluorescent American mineral oil loses its fluorescence at once on being shaken with nitrobenzene. Nitronaphthalene possesses the advantage over nitrobenzene of not being recognisable by its smell; it has a feebly aromatic, almost uncharacteristic smell, it crystallises in thin yellow needles, and when used to remove fluorescence in mineral oils it must be dissolved in the oil by warming. Oils so treated soon darken, which, as oils are commercially judged by colour, is a drawback to its use for hiding the adulteration of fatty oils with mineral oils.

The author recommends the two following reactions as a means of detecting the presence of nitrobenzene and nitronaphthalene in oils and fats:—

1. The aromatic nitro compounds of the general formula  $R \cdot NO_2$ , to which nitrobenzene and nitronaphthalene belong, are converted by the action of suitable reducing agents into red-coloured azo compounds. For this purpose the author uses concentrated alcoholic potash. According to Richter (*Chemie der Kohlenstoffverbindungen*, 1888, 627) nitronaphthalene forms an exception and is not reduced; and, according to Schultz (*Ber.* 17, 478), neither sodium amalgam nor zinc-dust and alcoholic potash, which reduce all the other nitro compounds, will reduce nitronaphthalene. The author finds, however, that when nitronaphthalene is boiled for 1—2 minutes with concentrated alcoholic potash a red colour is produced, which would point to the probable formation of azonaphthalene, although it is possible that the colour may be due to other products of reduction. With dilute alcoholic potash no red colour was obtained, and therefore the degree of concentration of the alcoholic potash is an important factor. Nitrobenzene gives a red colour even with dilute alcoholic potash. The alcoholic potash to be used in the test is made by dissolving 10 grms. of potassium hydrate in as small a quantity of water as possible and then diluting with 100 cc. of absolute alcohol. If a pure clear mineral oil be boiled from 1—2

minutes in a test-tube with from 2—3 cc. of concentrated alcoholic potash, a yellow or brownish-yellow solution is yielded, and most of the fatty oils behave similarly; only in the case of a few substances, *e.g.* train-oil, is a yellow-red or blood-red colour produced after long continued boiling. When oils or fats containing nitrobenzene or nitronaphthalene are heated in this way after boiling from 3—1½ minutes a blood-red or violet-red colour is produced, and it will be observed that on warming the upper part of the test-tube where there are adhering drops of the mixture of potash solution and oil just above the main bulk of the liquid, these drops at once become coloured reddish-violet. This last-named appearance the author has never obtained with any oil free from nitro compounds.

As all colour reactions with fatty oils are liable to be much modified by the nature of the materials present, this test is only recommended as a preliminary one, which, in the event of a negative result, may save much time. The author relies on the following method for definitely ascertaining the presence of nitronaphthalene and nitrobenzene.

2. By the action of strong reducing agents (*e.g.* nascent hydrogen) upon the nitro compounds they are converted into amido compounds—thus nitrobenzene,  $C_6H_5NO_2$ , yields aniline,  $C_6H_5NH_2$ ; and  $\alpha$ -nitronaphthalene,  $C_{10}H_7NO_2$ , yields  $\alpha$ -naphthylamine,  $C_{10}H_7NH_2$ .

The author has already shown (Mittheilungen, 1892, 186; this Journal, 1893, 527—528) how this reaction may be used for detecting nitrobenzene in fats, the aniline obtained being recognised by the characteristic colour it gives with bleaching powder. In a similar way naphthylamine may be obtained and recognised by its smell and by its colour reaction with ferric chloride—an azure-blue colour passing into red-violet.

A few cubic centimetres of the oil to be examined are placed in an Erlenmeyer's flask and boiled briskly for about 10 minutes "with zinc and hydrochloric acid (one part of concentrated and one part of dilute acid)." The naphthylamine formed goes into solution as hydrochloride, together with zinc chloride in the aqueous liquid, which is then separated by means of a separating funnel from the layer of oil, and filtered. The clear aqueous solution is then placed in another separating funnel and treated with as much caustic potash or soda solution as will redissolve the precipitated zinc hydrate. The solution is then cooled and shaken with from 10—20 cc. of ether. The ether extracts the  $\alpha$ -naphthylamine from the solution, and the presence of the base is indicated by the violet colour and fluorescence of the ethereal solution. The ethereal solution is then separated and evaporated to dryness, and leaves a residue of violet-coloured strongly-smelling  $\alpha$ -naphthylamine. On treating this with a few drops of hydrochloric acid the hydrochloride is formed and remains partially insoluble and partially soluble, but on evaporating the excess of acid a clear aqueous solution is obtained in which ferric chloride produces an azure blue precipitate. When filtered, this precipitate becomes purple-red, and the filtrate is blue-violet. Aniline gives with ferric chloride at first a green precipitate, which afterwards turns deep blue and does not become purple-red even on long standing, and the filtrate from the precipitate is yellow coloured. When  $\alpha$ -naphthylamine and aniline are present together, the precipitate is at first azure blue and afterwards a dirty red-brown, and finally purple-red, and the filtrate is violet. Aniline alone also dissolves readily on addition of a few drops of hydrochloric acid without previously evaporating the excess of acid.

By the above-described method the author has detected nitronaphthalene in Scotch mineral oil rendered non-fluorescent, in refined, almost odourless, and non-fluorescent rosin oil, and in grease for treating leather.

After this article was in manuscript there appeared in the Chemical News, December 22, 1893, 297, a short description by N. Leonard of a similar process.

Other means of reducing  $\alpha$ -nitronaphthalene may of course be used, *e.g.*, stannous chloride and hydrochloric acid, and other reactions for proving the presence of the  $\alpha$ -naphthylamine, *e.g.*, its colour reaction with azobenzene-sulphonic acid.—H. S. P.

## ORGANIC CHEMISTRY.—QUANTITATIVE.

*The Influence of "Weighting" on the Specific Gravity of Silk.* H. Silbermann. Chem. Zeit. 18, 1894, 744—745.

In determining the specific gravity of silk with the hydrostatic balance, the use of chemically pure benzene for the immersion fluid, as recommended by Vignon, is advised. This recommendation is given after due comparisons with the stereometer of Say, the volumometer of Regnault, and the mercury densimeter of Bianchi. Benzene is specially preferred, since it can be brought to ebullition easily under a pressure represented by 450 mm. of mercury at 10° C., and thus all gases and vapours in the pores of the silk can be expelled. Also the density of the benzene at temperatures between 0 and 30 is accurately known. (Heilstein, 2, 16.) The vessel containing the sample soaked in benzene is placed under the bell jar of the air-pump, and a partial vacuum is obtained corresponding to a pressure of 50 mm. of mercury. The vessel is then transferred to the hydrostatic balance. Thus the specific gravities of some textile fibres were estimated, as follows:—Wool, 1.28—1.33; cotton, 1.5—1.55; raw silk, 1.3—1.37; boiled-out silk, 1.25. (This Journal, 1892, 1002.) The specific gravity of silk varies in course of chemical treatment by dyeing, bleaching, and weighting, and in course of mechanical treatment by stretching, souping, &c.

In order that useful conclusions as to the approximate amount of weighting present in any given silk may be deduced from its specific gravity, the nature of such weighting and the process of dyeing used must be, at least, qualitatively known. For this purpose the author proceeds as follows:—Readily soluble materials, such as cane-sugar, glucose, glycerol, magnesium salts, &c., are estimated directly by boiling the silk with water, and testing the extract with Fehling's solution, &c. About 2—3 grms. of the silk are reduced to ash, and the latter tested by the usual methods of mineral analysis for tin (present in the fibre as basic chloride and stannic acid), chromium, iron, &c. Fatty matters, wax, and paraffin are detected by extraction with ether or benzene. The silk is soaked in warm, dilute hydrochloric acid (1:2). If by this treatment the fibre be almost decolorised, only a slight yellow tint remaining, whilst the solution assumes a deep brownish colour *not* changed to violet by lime-water, it is safe to conclude that the silk had been dyed by alternate passage through baths of iron and tannin. The yellow colour of the fibre is due to a residuum of tannin, and the precise shade (from greenish to brownish-yellow) enables one after some practice to form an idea as to the nature of the tanning material used (sumach, dividivi, catechu, &c.). Decolorisation of the fibre, the acid extract being pink, changed to violet by lime-water, indicates a logwood black (so-called "English black"). If the fibre retains a deep greenish tint and the solution be yellow unaffected by lime-water, the black is dyed on a Prussian blue ground. If the latter, as is often the case, has been produced during the final stage of dyeing, this is shown by its solubility in the acid. A green fibre and pink solution altered to violet by lime-water, point to a logwood black on a Prussian blue ground. In the hydrochloric extract metals, such as lead, tin, iron, chromium, and aluminium, may be estimated. Blacks produced by artificial dyes on an iron tannin or iron-blue catechu ground, are recognised by the coloration imparted to acid and sodium hydrate solutions. As regards blacks produced solely by the agency of aniline dyes, &c., treatment with a hydrochloric solution of tin-salt does not affect aniline and alizarin black; naphthol-black is altered to a reddish-brown colour, whilst wool-black becomes yellowish-brown. Aniline and alizarin blacks may be distinguished by means of sulphurous acid, which attacks only the former, turning it greenish. Tannin substances in general may be extracted by alkaline solutions and subsequently precipitated and distinguished by ferric acetate. To remove the whole of weighting material and dye, it is recommended that the silk be boiled with acid potassium oxalate, washed with dilute hydrochloric acid, and finally treated with soda solution. When iron and tin are both present in the fibre, it is well to previously extract the latter by means of an alkaline sulphide. In conclusion, the author gives the following

experimental results, which exhibit the relation existing between the density of silk and the percentage and nature of the weighting material. The figures for weighting and loss (during softening, boiling, &c.) are in every case referred to 100 parts of raw silk.

*Coloured Silk.*—A. Softened silk (Sample):—Unweighted; loss, 6 per cent.; sp. gr. 1.34. Weighted with tin chloride, 45 per cent.; sp. gr. 1.61; with tin chloride and tannin, 16 per cent.; sp. gr. 1.53; with tannin alone, 46 per cent.; sp. gr. 1.48.

B. Boiled silk (Unit):—Unweighted; loss, 20 per cent.; sp. gr. 1.33. Weighted with tin chloride, 21 per cent.; sp. gr. 1.52; with tin chloride and tannin, 22 per cent.; sp. gr. 1.54; with tannin, 26 per cent.; sp. gr. 1.48.

*Black Silk.*—A. Softened silk:—Weighted with Prussian blue and catechu, 126 per cent.; sp. gr. 1.74; with tin chloride, Prussian blue, catechu, and extract of nut-galls, 120 per cent.; sp. gr. 1.88.

B. Boiled silk:—English black on an iron ground, 28 per cent.; sp. gr. 1.58; mineral black, *nil*; sp. gr. 1.37; black on iron ground, catechu and pyrolignite, 20 per cent.; sp. gr. 1.10.—H. T. P.

*Estimation of the Titre of Tallow.* F. Wolfbauer. Chem. Zeit. Rep. 18, 1894, 28–29.

For this estimation 120 grms. of the fat to be tested are melted in a beaker with 45 cc. of 48 per cent. potash (sp. gr. 1.509) and heated at 100° C. until complete saponification is obtained, which is recognised by the complete solubility of the products in 50 per cent. alcohol. The soap is then decomposed by boiling with 165 cc. of sulphuric acid of sp. gr. 1.143 until the fatty acid has separated as a clear solution from the acid potassium sulphate solution; the latter is removed and the fatty acid digested in a covered vessel containing sulphuric acid (5 cc. per 100 cc. water) for 15 minutes.

The fatty acid is then washed by boiling with about 100 cc. of water, and dried for two hours at 100°.

The estimation of the solidification point is conducted in the usual manner.—T. A. G.

*Valuation of Crude Glycerol.* O. Heller. Ber. der pharm. Ges. 4, 1894, 17.

The distillation method of the author is described. A double-necked distilling flask of about 500 cc. capacity is required. Through one neck passes a thermometer and through the other, two glass tubes, one of which reaches to the surface of the glycerol and the other is cut off just below the neck. The latter tube is connected with four Erlenmeyer flasks provided with condensing coils. The last flask communicates with an air-pump and the glycerol is heated in an air-bath to 100°. As soon as this temperature is reached a current of air is drawn through, at first slowly and then more rapidly. The temperature is kept at 180° to 190° C., and the glycerol vapour, carried over by the air current, condenses in the flasks. These are exactly tared, so that it is only necessary to re-weigh them. The author recommends this method for other bodies not readily volatile, like aniline, heavy hydrocarbons, fatty acids, &c.—T. A. G.

*The Determination of Starch in Pressed Yeast.* F. Filsinger. Chem. Zeit. 18, 1894, 742.

According to the author the methods usually adopted for the above purpose are utterly unreliable. He recommends a process originated by E. Fickert, based on the fact that when starch containing yeast is suspended in water, the starch tends to settle down first. In practice 20–30 grms. of yeast are stirred up with 250 cc. of water, and sufficient iodine solution added to stain the starch and render it plainly visible (and also specifically heavier). After settling, the supernatant liquid is siphoned off, and carries with it a great deal of the yeast, the starch being left behind. The process of decantation is repeated until yeast cells are no longer discernible, and the starch finally

collected on a tared filter-paper, is dried at 105° C., and weighed. During drying, practically the whole of the iodine absorbed by the starch is expelled. According to C. Luders, the method gives almost absolutely accurate results in presence of 10 per cent. of starch and upwards. With smaller percentages the figures come out somewhat low.

—H. T. P.

*Further Note on the Determination of the Fat in Bread.*

M. Weibull. Zeits. angew. Chem. 1894, 199–202.

In a former communication (this Journal, 1894, 294) the author shows that the ordinary method for the estimation of the fat in bread by ether extraction always yields low results, owing to the fact that the fatty particles are so surrounded by starchy and dextrinous matters as to be quite inaccessible to the solvent. An improved method was therefore devised, according to which the bread is boiled with very dilute sulphuric acid to convert the starch into dextrose, then neutralised with powdered marble, the liquid and sediment, &c., being finally absorbed by and dried on a strip of filter-paper, and extracted by ether, as in Adam's process for milk analysis. The chemists of the German Board of Health in the main confirm the author's strictures on the old process, but failed to obtain higher figures by the new method; and it was suggested that the fat was again occluded by the dextrose during the drying of the inversion products. The author has therefore reinvestigated the subject, and in a table gives the results of a series of fat determinations in various flours and the bread baked there-with. The conclusions arrived at are:—

1. The fat in rye-bread, not directly estimable, may be completely extracted by the inversion method.

2. The ethereal extract from coarse or fine rye-flour is sensibly the same as that from the bread baked therewith, allowance being made for any difference in the percentage of moisture.

3. Coarse flours cannot be freed from fat by ordinary extraction; either the inversion process must be employed or the material must be reduced to fine powder prior to analysis.

4. In the case of brans also low results would probably be obtained by the ordinary method for fat determination.

—H. T. P.

*The Albuminoids contained in Worts and Beers.* Wahl and Hantke. Amer. Brewers' Review, 7 [32].

See under XVII., page 895.

*Methods of Estimating Free Tartaric Acid in Wines.*

A. Carpenté. Selmi, 1894, 4, 13.

The method recommended is this:—

20 cc. of wine are evaporated to 10 cc., and the residue shaken up with 50 cc. of ether-alcohol and allowed to stand for 12–18 hours. The clear liquid is then poured off and the precipitate washed with 15 cc. of ether-alcohol, and filtered. The washings are mixed with the first filtrate and decomposed with 0.01 gm. of tartaric acid dissolved in 1–2 cc. of water. Shake and let stand 2–3 hours in a cold place. If a crust of tartar crystallises out, the presence of acid potassium salts (malate, succinate, acetate) is established, and the presence of free tartaric acid negatived. If no crust forms the wine may contain free tartaric acid. In this case the ether-alcohol should be distilled off and the residue be dried in the flask, when any tartrates of magnesium, &c., will be rendered insoluble. On digestion with 50 cc. of ether-alcohol the free tartaric acid is dissolved. After some hours filtration follows, then addition of 4–8 drops of potassium acetate and the tartar formed is precipitated. The tartar is titrated with alkali and calculated to tartaric acid, when the amount thus found, less the tartaric acid added, equals that originally present in the wine.

—T. A. G.



*The Examination of Liquid Extracts.* O. Linde. Pharm. Central-H. N.F. 15, 1894, 39.

For the estimation of glycerol contained in liquid extract, Linde employed Proskauer's method devised for the examination of sweet wines. This method, however, was not applicable, because the resin contained in the glycerol forms line soaps with the calcium hydrate, which, especially in presence of glycerol, are soluble in water and ether-alcohol. The method also indicates too high results. The following method was successful:—10 grms. are evaporated to half bulk, taken up with 50 cc. of water, and lead acetate added gradually as long as a precipitate forms. After filtration, the filtrate is mixed with a few drops of dilute sulphuric acid and concentrated solution of tungstic phosphate added as long as a precipitate forms. After settling, filter, wash, neutralise the filtrate with dilute soda, evaporate to a syrup, and take up with 30 cc. of ether-alcohol. Filter again, wash with ether alcohol, and evaporate the filtrate in a long-necked flask until a constant weight is obtained.

Sometimes the resulting glycerol is slightly coloured, in which case it can be decolorised with animal charcoal, although the coloration is usually sufficiently slight to be negligible.

To detect glycerol obtained from extract:—

1. Render slightly alkaline with sodium carbonate, mix with powdered borax, and ignite on a platinum wire, when a green colour is obtained.

2. Red litmus paper is rendered blue by borax and dried, and on dipping it into the solution of glycerol and sodium carbonate the red colour is restored, immediately with strong and gradually with weak solutions.—T. A. G.

## ANALYTICAL AND SCIENTIFIC NOTES.

### *The Use of Sodium Peroxide for Analytical Purposes.*

T. Poleck. Ber. 27, 1051.

Sodium peroxide behaves similarly to the better known barium and hydrogen peroxides, but much more energetically. Silver, mercury, and gold salts are reduced with evolution of oxygen. Platinum chloride is only reduced when the compound  $\text{PtCl}_2\text{H}_2$  is decomposed by a silver salt. Then both the silver chloride and the platinum chloride are reduced to the metallic state. The same thing holds good with the platinum double salts of the alkalis and of the alkaline earths, reduction to metallic platinum only taking place after addition of a silver salt.

Treated with sodium peroxide, ferrous and ferric salts give ferric hydrate. Manganese and cobalt salts give manganese dioxide and cobalt oxide. Permanganic acid is reduced to manganese dioxide. Chromium oxide is oxidised to chromic acid. Uranium salts give sodium peruranate  $\text{U}_2\text{O}_5\text{Na}_4 + 8\text{H}_2\text{O}$ . Bismuth hydrate and its salts are oxidised to bismuth pentoxide.

Potassium ferriocyanide is reduced by sodium peroxide to ferrocyanide, and this reaction may be used for gravimetrically determining the purity of the peroxide. By this method the sodium peroxide used in the author's experiments was found to contain 83.62 per cent. of the pure product.

It may be used for the separation and estimation of iron and chromium, and of manganese and chromium. The iron and the manganese are precipitated as ferric hydrate and as manganese dioxide, whilst the chromium goes into solution as chromic acid, and may be reduced by alcohol and weighed as chromic oxide. In a mixture of known quantities of manganese sulphate and chrome alum, 99.96 per cent. of the former and 99.67 per cent. of the latter were found, and in a mixture of ferrous ammonium sulphate and chrome alum, 99.81 per cent. of the former and 99.93 per cent. of the latter were found by this method.

It may be used also for the separation and estimation of antimony, tin, and arsenic. The metals are precipitated by sulphuretted hydrogen, dissolved in ammonium sulphide, evaporated to expel excess of ammonium sulphide, or reprecipitated; then water and sodium peroxide are added until the yellow colour of the sulphur compounds has disappeared,

which takes only a few minutes. The metals and sulphur are thus completely oxidised and the oxygen compounds of the metals are separated by known methods. In two experiments with mixtures of tartar emetic, tin, and arsenic acid, the contents of which were known, the following percentages of the constituents were found.

	I.	II.
Tartar emetic .....	Per Cent. 99.67	Per Cent. 99.96
Tin .....	99.04	99.91
Arsenic acid .....	99.84	99.75

This method of oxidising the metallic sulphides is preferable to the ordinary nitric acid method in toxicological examinations, because it allows of "Marsh's" apparatus being immediately applied.

The fusing of minerals and ores with sodium peroxide for purposes of analysis, has been described by several investigators. The most complete oxidation takes place by using a mixture of equal parts of sodium carbonate and sodium peroxide, and this would be an excellent analytical method did not the choice of vessel for fusion present difficulties. Silver, platinum, and nickel are strongly acted upon. A platinum crucible lost 0.05—0.1 gm. in weight after each operation.

By the action of sodium peroxide on lead oxide in presence of water, not ortho- but meta-plumbate of soda is formed,  $\text{PbO}_3\text{Na}_2 + 4\text{H}_2\text{O}$ . The author was altogether unsuccessful in obtaining the orthoplumbate,  $\text{Pb}(\text{O}^+\text{Na})_2$ .

By heating iodine with sodium peroxide, the sodium periodate,  $\text{Na}_2\text{H}_3\text{IO}_6$ , is formed.

The oxidation of carbon compounds in alcoholic solution by sodium peroxide promises good results. Since alcohol is not attacked by it.—H. M.

### *The Action of Formaldehyde on Vegetable Tissues.*

J. Hofkett. Ber. d. Pharm. Ges. 1894, 4, 81.

FORMALDEHYDE, now commercially obtainable under the name "Formalin," is an excellent agent for hardening fresh vegetable preparations for the purposes of botanical research. A 4 per cent. solution of the reagent hardens vegetable objects more rapidly and effectually than absolute alcohol, and possesses besides two great advantages: it does not extract or otherwise affect the natural colouring matters of flowers, leaves, &c.; and, moreover, the tissues immersed in it do not appreciably shrink.—H. T. P.

## Trade Report.

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

(From the Board of Trade Journal.)

#### RUSSIA.

##### Customs Divisions.

Dissolvable glass, with an admixture of glue, under section 71, point 5, letter a: duty, 2 roubles, gold, per pound.

Caterpillar glue (Ernisch) for preserving trees from noxious insects, prepared from tarry substances with an admixture of mineral salts, to be cleared under section 71, point 5, letter b: duty, 40 copecks, gold, per pound.

Salts of antimony (double antimonial nitrothioric acid salt), used in some instances in lieu of tartar emetic, to be cleared under section 112: duty, 1 rouble and 50 copecks, gold, per pound, gross.



Paranitraniline, not constituting in itself an aniline colour (pigment), to be cleared under section 112; duty, 1 rouble and 50 copecks, gold, per pound, gross.

Carbolic acid in a purified state or only partly purified, to be cleared under section 112; duty, 1 rouble and 50 copecks, gold, per pound.

Phosphite (a triturated mixture of tale and hydrous copperas), used for preserving vines and other plants from noxious insects and disease, to be cleared under section 125, point 2, letter *b*; duty, 35 copecks, gold, per pound, gross, if the admixture of hydrous copperas does not exceed 10 per cent. in quantity.

Zinc dust, under section 147, point 1; duty, 40 copecks, gold, per pound.

Tartar emetic, notwithstanding that it may have been slightly purified, under section 112; duty, 1 rouble and 50 copecks, gold, per pound.

#### UNITED STATES.

##### Customs Decisions.

Direct black, not a simple extract or decoction of a dye-wood, but a constructed dye and a chemical compound, is dutiable at 25 per cent. *ad valorem* under paragraph 76 N.T. as a chemical compound.

Oxide of cobalt, blues, satin-white, chromes, ochres, umbers, vermilion reds, white leads, verdigris, and other colours in oil, and in small tubes, commercially known as artists' colours, in tubes, are dutiable at 25 per cent., under paragraph 61.

Prune pure, a coal-tar colour or dye, produced by combining methyl with galloxyaniline, is dutiable at 35 per cent. *ad valorem* under paragraph 18 N.T.

Lanae, a preparation produced from wool grease, substantially free from alkalis, is dutiable at the rate of 25 per cent. *ad valorem* under paragraph 76 as a rendered oil.

"Curriers' fat blacking" is dutiable at 25 per cent. *ad valorem* under paragraph 11 N.T.

Business cards made of collodion or celluloid are dutiable at 60 cents per pound and 20 per cent. *ad valorem* under paragraph 21 N.T.

Crine, composed of a mixture of fine clay and a fat oil, and used by artists, sculptors, and others for modelling, not wax, is dutiable at 20 per cent. *ad valorem* under section 4 of the Tariff Act.

Essential oil of nutmegs is dutiable at 25 per cent. *ad valorem* under paragraph 76 N.T.

Sulphate of ammonia is dutiable at  $\frac{1}{2}$  cent per pound under paragraph 10 of the existing tariff.

Thin strip of magnesium about one-sixteenth of an inch in width, known as "magnesium ribbon," is dutiable at 45 per cent. under paragraph 215 N.T.

#### GENERAL TRADE NOTES.

##### OLIVE OIL INDUSTRY IN SPAIN.

The *Moniteur Officiel du Commerce* for the 26th July states, that according to the average of the last five years, the annual production of olive oils in Spain has been 2,976,584 quintals. The home consumption is distributed as follows:—Oils for purposes of food, 1,108,151 quintals; for factories engaged in the preserving industries, 53,713; for purposes of lighting, 467,200; for soap factories and other industries, 675,000; for oiling machinery, looms, ships, &c., 450,000; or a total of 2,754,064 quintals.

The annual exports amount to 222,320 quintals, distributed as follows amongst the countries of destination:—Spanish possessions, 63,486 quintals; France, 46,019 quintals; Portugal, 29,766 quintals; England, 17,126 quintals; Denmark, 16,893 quintals; Germany, 11,316 quintals; Asia and Africa, 15,529 quintals; America, 12,624 quintals; and other destinations, 9,561 quintals. Total, 222,320 quintals.

##### DISCOVERY OF SALINES IN MEXICO.

The French Minister at Mexico in a recent report states that immense salines have recently been discovered in the district of Astapa, State of Tabasco. A powerful American company is about to be formed for the purpose of working them.

##### RUSSIAN ALCOHOLIC PRODUCTION.

The Russian *Gazette du Commerce et de l'Industrie* states that from the 1st January to the 1st June 1894, the production of the Russian alcohol distilleries amounted to 29,282,951 vedros of pure alcohol against 26,604,345 vedros in 1893. The vedro may be taken as equivalent to 2.7 imperial gallons.—*Ibid.*

##### ON A NEW DEPOSIT OF PHOSPHATES IN THE SOUTH OF FRANCE.

In 1882, while engaged in the study of the geological structure of the valley of the Aude, on the north-eastern flank of the Pyrenees, the author undertook the investigation of the deposits in a series of caverns contained in the limestone cliffs of the River Cesse. These limestones, which are of Eocene date, form elevated barren tablelands known as "causses," rising to about 1,000 ft. above the sea-level, and, where cut through by the streams, are seen to lie at a low angle upon hard siliceous limestones of Devonian age. The surface of junction is irregular, and the upper strata near the contact are eroded into long ramifying galleries whose roofs and sides are in the nummulitic rock, while the floors, apart from the filling material, are formed by the Devonian rock. The most important of these caves, known as the Grotte de Minerve, situated near the village of the same name, about 17 miles from Narbonne and 22 miles from Carcassonne, is a system of two principal galleries from 25 to 30 ft. wide, and 30 to 33 ft. high, which cross obliquely and extend for about a total length of 3,600 ft., following the dip of the nummulitic strata. The floor is covered with cave earth and bone breccia, containing worked flints, pottery, and other prehistoric antiquities, the bones of the cave bear and other quadrupeds to a depth of 10 to 15 ft., below which concretions of phosphoric, and other forms of phosphates, both of lime and alumina, are found in quantities; the deposits, proved by numerous trial pits, varying from 15 to 50 ft. in depth, being estimated to contain from 120,000 to 300,000 tons of phosphatic minerals, which differ in many respects from the ordinary phosphorites of stratified formations. Among these, one of the most interesting is brushite, a hydraulic dibasic calcium phosphate ( $2\text{CaO}, \text{P}_2\text{O}_5, 5\text{H}_2\text{O}$ ) which had only been known previously as a secondary product incrusting the rock guano of Aves Island and Sombbrero in the West Indies. It contains phosphoric acid 43 per cent., lime 34 per cent., and water 23 per cent., and, as a rule, occurs in crusts upon and filling fissures in the limestone rocks, and in the purest form is a bright powdery substance made up of thin crystalline plates exactly similar to those described by the discoverers of the original mineral. The bulk of the deposit is, however, made up of mixtures of tribasic calcic phosphate and neutral phosphate of alumina, which vary within rather wide limits, but frequently are in nearly equal proportions, about 24 to 25 per cent. of each, containing about  $27\frac{1}{2}$  per cent. of phosphoric acid, which is mostly all soluble in weak acids. A third, and previously undescribed substance, to which the name of minervite has been given, is a hydrated aluminic phosphate ( $\text{Al}_2\text{O}_3, \text{P}_2\text{O}_5, 7\text{H}_2\text{O}$ ) which was found as a white plastic mass, filling a vein from 2 to  $2\frac{1}{2}$  ft. thick.—*Annales des Mines, through Engineering and Mining Journal.*

##### ALKALI LANDS IN NORTH CHINA.

A correspondent of the North China *Herald* gives the following information concerning the alkali lands in the north part of that country:—

Chinese may often be heard to say, in the north especially, that certain ground is useless on account of alkalinity. The following report shows that this land has a value of its own which will probably be appreciated hereafter.

During the dry time of the year the surface of the land in many parts of the north of China is covered with a white incrustation of salts, called *chien* by the natives, which easily dissolves in water, and therefore disappears during the rainy season. A sample collected in the neighbourhood of Peking, a few li to the S.W. of the city, contained:—

	Per Cent.
Chloride of sodium (common salt) .....	23.8
Carbonate of sodium (soda) .....	12.4
Sulphate of sodium (Glauber's salts) .....	63.8
	100.0

I obtained large quantities of these salts, by having the ground swept with a broom, and lixiviating the sweepings with warm water. Over a third of the material thus collected proved to be soluble matter. It may be separated from the solid residue either by filtering or by siphoning the supernatant liquor and evaporating the saline solution, which shows a strong alkaline reaction on account of the carbonate of sodium contained in it. When sufficiently concentrated, the liquid, on cooling, deposits a large mass of crystalline matter, which for the most consists of sodium sulphate, a part of it, together with the sodium carbonate and chloride and sodium, remaining in the mother-liquor. The latter, on being evaporated by dryness, yields a brownish-

looking substance, the colour being due to organic matter—which, on being treated with vinegar, shows a brisk effervescence. It is, to all intents and purposes, the same substance as the one called *Tzu-chien* by the Chinese, which is an impure carbonate of soda extensively used in dye works. Not only does it serve for the cleansing of textile fabrics, but is also used as a mordant, for instance, in colouring cotton cloth with a solution of indigo, &c.

Judging from the above, there appears to be an abundance of sodium sulphate in some parts of China, which may become valuable some time or other when, with the introduction of foreign industries, such as the manufacture of glass and soap, a greater demand is created for this chemical substance than exists at present. As there is plenty of limestone and coal in many places, all the materials wanted are possessed in abundance. This, in addition to the cheapness of labour, makes it not improbable that, at some time or other, China will become one of the chief producing countries of soda.

In the table below I give an analysis of each of the four kinds of soda distinguished in trade. The samples were obtained from one of the great depôts outside the city. For comparison, the English commercial "degrees" have been added. Taking the quotations of a London trade report, I find that, on the whole, prices in Peking, at present exchange, do not rule much above those at home.

Colour.	Water.	Carbonate of Soda.	Chloride of Sodium.	Sulphate of Sodium.	English Commercial Degrees.	Price per 100 Catties, or 1 Picul.
<b>K'ou-chien (from Kalgan):—</b>						
1. P'ien chien, yellowish white .....	Per Cent. 15.61	Per Cent. 53.00	Per Cent. 1.33	Traces	31.41	49.4 Tiao; Shanghai Tls., 3.8
<b>Hsi-k'ou-chien (from Shansi):—</b>						
2. Pai chien, white .....	52.60	45.71	1.07	Traces	27.09	46.8 Tiao; Shanghai Tls., 3.6
3. Tzu chien, first quality, dirty yellow ....	28.65	11.61	6.29	23.46	21.66	39.0 Tiao; Shanghai Tls., 3.0
4. Do., second quality, brown .....	17.30	53.13	18.14	31.27	19.19	35.1 Tiao; Shanghai Tls., 2.7

At present this substance is extensively used by the Chinese for cleansing purposes, taking the place of soap in foreign countries. It also serves as a baking powder in the preparation of Chinese bread. As far as I have been able to ascertain, the article which is sold at Peking is nearly all derived from places outside the Great Wall. There are two different kinds—"k'ouchien," which is brought here from Kalgan, and "ksik'ouchien," which comes from the northern part of the province of Shansi, the prefecture Tat'ungfu. The former, I am told, is obtained from salt lakes, pools, or pits dug for the purpose of collecting the brine with which the soil is impregnated. It is a very superior article, almost free from impurities and largely used in the preparation of *mant'ou* and other kinds of bread. The material which furnishes the *ksik'ouchien* is found as a white efflorescence on the soil, similar to that observed in the neighbourhood of Peking. According to the account given by the Chinese, it forms layers of considerable thickness in some places, and is a good deal richer in the carbonate than what is to be got hereabout. There are several sorts of it, one of them called "*pachien*" being of an excellent quality. It is almost free from chlorides, and shows hardly a trace of sulphate, which is all the more remarkable, as it is said to be prepared by a process of crystallisation from some of the inferior sorts of *tzu-chien* already spoken of.—*Engineering and Mining Journal*.

#### CHROMIC IRON IN QUEBEC, CANADA.

There are indications that the raising of chromic iron ore may become one of the mining industries of the province of Quebec. It has long been known that chromite occurs in pockets in the Cambrian Serpentine that stretch through the province from the Vermont boundary to Gaspé, and in the past a few tons of the ore have been taken out at various times and from various points. But disappointment in the mode of occurrence of the ore, and in the price that could be obtained for the same, led to a speedy abandonment of the attempt to mine it.

So far as can be learned, the total amount of chromite mined in Canada during the past 10 years is less than 100 tons. One of the chief obstacles to chrome ore mining in Canada, common to the great majority of places where this ore is found, is the mode of occurrence. It is rarely found in beds or veins, but in detached pockets which yield from a few pounds to hundreds of tons, the larger pockets being comparatively rare.

In consequence of the mode of this mining, investors hesitate to embark in a species of mining more than usually uncertain, and having entered upon it do not feel warranted in putting up expensive machinery. Another and more serious obstacle is the present attitude of the users of chrome ore, the principal of whom are the manufacturers of bichromate of potash. These are unwilling at the present time to purchase ore carrying less than 50 per cent. of chromic sesquioxide, although it is believed that under certain conditions ore of 48 per cent. is accepted. Most, if not all, the Canadian deposits that have been tapped are below this grade. But during the present season deposits of chromite of a grade sufficiently high to meet the demands of manufacturers have been discovered and are being worked within two miles of Black Lake Station on the Quebec Central Railway, and in close proximity to the well-known Black Lake asbestos mines.

A large body of prospectors is out searching for chrome deposits, and finds are reported, but as yet only three lots are being worked. The pioneer lot in this district, and that on which most mining has been done, is the property of Mr. M. H. Lambly, of Inverness, Quebec, and associates. In this the chromite occurs in a series of pockets, extending in an east and west direction, and the series in an easterly direction into the adjoining lot. In these two lots some of the pockets are lying against a dyke of fine-grained granulite, but whether there is any relationship between the igneous rock and chromite it is impossible to say. Other pockets seen elsewhere in the vicinity are not associated with the granulite, but those in which the granulite lies

against the chromite have yielded a large amount of ore of very fine quality. From one pocket on the Lambly property nearly 500 tons have been taken out, and it is still producing. The writer selected two hand specimens of this Lambly ore as free as possible from foreign matter, and found them to yield on analysis 54 per cent. and 56.02 per cent. of chromic sesquioxide.

It is evident, of course, that a shipment of ore would not average as high as these selected hand specimens, and that careful dressing would be necessary to insure that shipments should not fall below the grade the market demands. Mr. Lambly states that a shipment of two cars tested 51 per cent. and realised 26 dols. per ton of 2,240 lb. delivered in Baltimore, which is equal to 20.50 dols. per ton f. o. b. at the mines.—*Ibid.*

#### ANTIMONY AND BISMUTH IN BOLIVIA.

The Consul-General of France at La Paz in Bolivia has recently made a special report on the mines of bismuth and antimony in that country. The only deposit of bismuth ore actually known is that of Quechisla (also known as Chorolque), though some exploration for others has been made, but without success. The returns show that the production of this mine is about 500 Spanish quintals, or 23,000 kilos., per month. This production, however, is regulated in concert with the European producers. In addition to bismuth the Quechisla mine yields some tin and a little silver.

Antimony is found in many places in the department of Potosi, generally in connection with gold and silver ores. In the province of Chayanta there are many veins of the sulphuret of antimony, which have become more accessible than formerly since the building of the Antofagasta Railroad. Owing to the present low price of the metal, however, it does not pay to work the poorer deposits. The Amayapampa Company, a recently formed Bolivian corporation, is now producing and exporting 100 metric tons a month of 65 per cent. ore. The mine is 18 miles from the railroad, over a difficult mountain trail. With better transportation the output could be largely increased. It is said, however, that all the veins so far found diminish in richness with depth. The industry is just beginning in Bolivia, and the Government has freed the producers from all direct tax and also from export duty.—*Ibid.*

#### PROPOSED GERMAN ANALYTICAL ASSOCIATION.

The German Government intend to take the initiative in the formation of a German association of analytical chemists, which will be charged with the drawing up of a code for the examination of alimentary substances and with the execution of a law relating to the adulteration of such substances. At a congress of chemists and microscopists recently held at Eisenach, and attended by Dr. Köhler, the director of the National Department of Public Health, the preliminaries of the association were discussed and agreed upon.—*Chemist and Druggist.*

#### THE CHEMICAL INDUSTRY OF BERLIN.

The following is an abstract of the report on the German chemical trade during 1893, prepared by the Berlin Chamber of Commerce.

During the year under review the German chemical trade has suffered severely from the effects of the tariff war with Russia. Although this warfare has been brought to an end, Germany will still labour under the disadvantage of having to recover trade that has been temporarily diverted to other quarters. Another drawback has been the unsettled state of the money market in the United States, as well as the tendency to withhold orders till the promulgation of the new United States Tariff Bill.

Up to the present time little advantage seems to have accrued to German manufacturers from the Chicago Exhibition, and the general consensus of opinion seems to be that the results so far have not repaid the costs involved in representation.

Almost the only products which have sustained any appreciable advance in price are the ammonia compounds. Two causes are suggested for this improvement—a decrease

in the English production of gas-water, and an increased consumption of sulphate of ammonia for agricultural purposes. The price of *ammonium sulphate* at the beginning of the year stood at 21 m. to 22 m. per 100 kilos. This gradually advanced to 30 m. per 100 kilos., and had only declined to 28 m. by the end of the year.

*Aqueous ammonia* 0.910 sp. gr. advanced from 22 m. to 35 m. per 100 kilos. *Ammonium carbonate*, unfortunately, did not share in the improvement to the same extent, and quotations did not rise above 62 m. per 100 kilos.

Technically pure *White Sal ammoniac* suffered a decrease in price out of proportion to its value, and quotations sank to 40 m. per 100 kilos.

The depressed condition of the *Potash* market has continued unchanged, 40 m. per 100 kilos. being quoted for 96 to 98 per cent.

*Oralic Acid* and *Oralates* have been unable to advance beyond last year's prices, 60 m. per 100 kilos. being the average figure for the first-named.

*Potassium Cyanide* continued in good demand, but towards the end of the year the price declined somewhat, bringing the general average to about 140 m. per 100 kilos.

For *Yellow Prussiate of Potash* the demand was so brisk that makers were emboldened to raise the price to 180—190 m. per 100 kilos.

The figure realised for *Silicate of Soda* has declined still further, although the consumption of the article has remained the same. Tariff troubles have largely conduced to this result, and the trade with the United States has practically died out.

*Soda Crystals*, which began the year at 6 m. per 100 kilos., were raised 50 pf. in consequence of an understanding with foreign producers, but as *Soda Ash* declined somewhat in the autumn, the price of crystals again fell to the first-named figure.

*Nitric Acid* underwent little change, 23 m. per 100 kilos. being the quotation for the refined 36° article.

*Sulphuric Acid*, especially of the lower qualities, underwent a considerable improvement, mainly because the fall in silver had caused many of the metallurgical works to suspend operations, and in consequence the supply of the acid was curtailed. This position of affairs naturally caused a corresponding scarcity of *Hydrochloric Acid*.

*Acetic Acid* remained at the unremunerative values of the preceding year.

*Wood Spirit* enjoyed a brisk sale at a slight improvement upon last year's prices.

In the *Tar* trade the various products remained in good demand, but sales were only effected at the cost of a continued decline in prices. There appears to be little hope for the success of the English movement to arrest this decline, as the yield from coke ovens is a continually increasing one.

The lowest *Benzol* price of the preceding year, 45 m. per 100 kilos., was soon reached, nor did the price halt here, but sank further to 40 m.

*Nylol* was in poor demand, and witnessed a decline in values, but on the other hand a brisk trade in *Toluol* sprang up, with a corresponding advance in price.

No particular change occurred in the *Anthracene* market, although large stocks were held in England, and a general disposition to withhold orders was exhibited by German consumers.

The demand for *Naphthalene* and *Phenol* continued good, but the decline in values was not arrested.

*Cresol* was well inquired for, and has realised its early promise of employment for disinfecting purposes.

*Roofing Varnish* and *Pitch* have found ready sales, but again at lower prices.

The prospects of the *Aniline Dye* industry in the beginning of the year were very favourable, but the silver troubles entirely changed the aspect of affairs. Two other causes are mentioned as contributing to the same result, the tariff war with Russia (which seems to be held responsible for a good many German misfortunes), and the unsatisfactory state of the Swiss patent laws, which permit of a wholesale piracy of German patented inventions.

The trade in non-poisonous *Chemical and Mineral Colours* has been characterised by extreme dullness, and the prices of previous years have not been maintained. This is attributed largely to the tariff troubles which were indicated in last year's report.

*Varnishes and Lacquers* have suffered from slackness in the building industry, and *Oil of Turpentine* having declined about 6 m. per 100 kilos., is again at the lowest point that has been touched. On the other hand, *Lead Compounds* have suffered little in value.

Makers of pharmaceutical chemicals who, in anticipation of a return of the cholera epidemic, had laid in considerable stocks, were heavy losers by the transaction, as no considerable demand was noticed in this direction. In consequence of the tariff difficulties, German pharmaceutical products are unable to find their way into Russia direct in anything like quantity, but as Russian buyers appreciate the German manufactures, these products have been handicapped by having to find their way to Russian consumers by more roundabout means.

An arrangement between the Stassfurt syndicate and the American producers has resulted in a greater steadiness in the *Bromine* market, and the price has risen 50 pf. per 100 kilos.

The demand for *Chloral Hydrate* has declined somewhat, in consequence of the increasing reputation of other similar products, but makers have covered this by a mutual arrangement with regard to prices.

The trade in *Citric Acid* has not come up to anticipation. Early in the year prices stood at 6d. to 7d. for 34 per cent., to 35 per cent., and 7d. to 8d. for 38 per cent., to 40 per cent. Quotations rose in February to 7d. to 8d. and 8d. to 9d., respectively, but after that declined slowly to 5½d. and 6d. to 7d., respectively.

*Glycerine* commenced the year in favourable demand, but the American crisis practically destroyed the trade with that country, and difficulties were experienced in inducing purchasers to take even their contract quantities.

The *Iodine* market has been in a disturbed state, owing to rumours of defection from the Iodine Convention. The effect of this has been to cause buyers, taught by previous experience, to withhold their orders.

Two new *Disinfectants* have made their appearance. "Formalin" is said to be an excellent disinfectant for houses and objects of all kinds, while for "Tri-cresol," that is a mixture of meta-, ortho-, and para-cresol, an equal efficiency is claimed for use in the treatment of wounds.—*Chemical Trade Journal*.

# IMPORTS AND EXPORTS OF DYEING MATERIALS THROUGH THE GERMAN CUSTOMS FOR 1892 AND 1893.

Dye.	Imports, 100 kg. nett.		Exports, 100 kg. nett.		Imports from Great Britain, 100 kg. nett.	Exports to Great Britain, 100 kg. nett.
	1892.	1893.	1892.	1893.	1893.	
Indigo.....	17,435	12,725	6,196	5,917	5,429	57
Indigo carmine.....	557	592	720	583	..	..
Logwood.....	416,625	437,581	102,045	83,898	..	..
Fustic wood.....	30,824	55,912	16,902	10,494	..	..
Red dyewood.....	3,952	25,902	12,194	8,310	..	..
Dyewood extract.....	43,270	53,923	15,204	16,945	2,313	..
Madder.....	2,147	2,449	1,898	1,545	..	..
Cochineal.....	674	757	333	331	..	..
Safflower.....	24	16	1	1	..	..
Quercitron.....	10,467	9,776	2,504	796	..	..
Catechu.....	67,523	57,190	15,534	13,521	8,147	..
Archil extract.....	2,479	5,194	2,380	1,315	..	..
Alizarin.....	421	394	70,779	89,358	70	27,289
Aniline and other coal-tar colours.....	6,859	7,302	107,251	115,539	694	23,632
Lac colours.....	109	77	7,279	5,858	..	1,711
Vermilion cinnabar.....	296	179	2,304	2,487	..	996
Mauve.....	4,425	3,842	54,378	58,574	3,077	12,908
Berlin blue.....	2,738	2,863	5,252	6,313	..	1,228
Ultramarine.....	431	461	43,553	41,423	..	15,304
Printers' black.....	312	282	9,031	8,938	..	..
Lamp black.....	1,834	2,133	7,696	9,788	..	..
Natural pigments.....	60,712	76,565	109,973	96,891	13,669	5,505
Paint and water colours.....	606	756	13,157	13,544	186	1,462

## BOARD OF TRADE RETURNS.

## SUMMARY OF IMPORTS.

Articles.	Month ending 31st August.	
	1893.	1894.
	£	£
Metals.....	1,656,211	1,559,209
Chemicals and dyestuffs.....	449,622	369,972
Oils.....	650,377	652,372
Raw materials for non-textile industries.	4,781,648	4,512,360
Total value of all imports ....	37,092,772	31,638,521

## SUMMARY OF EXPORTS.

Articles.	Month ending 31st August.	
	1893.	1894.
	£	£
Metals (other than machinery) ....	2,691,541	2,278,711
Chemicals and medicines .....	715,113	659,722
Miscellaneous articles.....	2,322,854	2,280,435
Total value of all exports.....	19,530,178	18,581,210

## IMPORTS OF METALS FOR MONTH ENDING 31ST AUGUST.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Copper:—			£	£
Ore..... Tons	6,122	6,012	23,865	35,615
Regulus .....	8,681	7,192	197,075	160,120
Unwrought ....	3,433	4,240	156,831	169,399
Iron:—				
Ore.....	421,370	387,338	204,916	269,279
Bolt, bar, &c....	7,784	5,820	73,347	57,857
Steel, unwrought..	874	324	9,476	3,428
Lead, pig and sheet	17,610	11,835	174,384	107,956
Pyrites .....	54,644	54,578	92,737	93,737
Quicksilver.....	Lb. 7,500	55,938	620	1,519
Silver ore.....	Value £ ..	..	237,439	151,459
Tin.....	Cwt. 45,395	73,105	191,078	253,883
Zinc .....	Tons 4,123	4,525	71,773	79,637
Other articles ...	Value £ ..	..	132,070	181,155
Total value of metals	..	..	1,656,211	1,559,209

## IMPORTS OF OILS FOR MONTH ENDING 31ST AUGUST.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Cocoa-nut.....	Cwt. 8,715	36,844	16,066	41,105
Olive .....	Tuns 1,343	2,282	51,295	71,395
Palm .....	Cwt. 122,621	110,319	149,174	116,949
Petroleum .....	Gall. 10,909,767	13,360,521	190,631	191,429
Seed .....	Tons 2,145	2,137	53,086	45,259
Train, &c.....	Tuns 1,937	3,272	39,787	54,592
Turpentine .....	Cwt. 76,167	59,931	78,411	55,357
Other articles ..	Value £ ..	..	77,687	73,376
Total value of oils...	..	..	650,377	652,372

## IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDING 31ST AUGUST.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Bark, Peruvian ..	Cwt. 3,712	6,036	6,965	10,817
Bristles.....	Lb. 224,852	327,139	30,683	42,043
Caoutchouc.....	Cwt. 24,805	16,371	265,307	156,061
Gum:—				
Arabic.....	" 8,124	4,224	16,154	8,954
Lac, &c.....	" 4,844	5,796	22,855	30,862
Gutta-percha ....	" 2,858	3,749	16,095	33,021
Hides, raw:—				
Dry.....	" 31,193	33,086	75,019	80,978
Wet .....	" 58,892	74,363	120,007	153,922
Ivory .....	" 734	543	34,577	23,693
Manure:—				
Guano .....	Tons 176	2,155	1,224	11,768
Bones.....	" 1,365	5,169	5,868	23,968
Nitrate of soda...	" 10,498	3,208	92,894	28,917
Phosphate of lime	" 49,156	46,732	65,658	92,510
Paraffin.....	Cwt. 72,501	34,116	70,562	37,941
Lineu rags.....	Tons 1,145	1,853	11,297	16,535
Esparto.....	" 15,139	13,025	67,481	53,403
Pulp of wood ....	" 16,731	24,464	98,289	118,205
Rosin.....	Cwt. 292,897	145,469	62,404	38,654
Tallow and stearin	" 156,681	187,776	217,578	239,327
Tar .....	Barrels 42,890	34,423	23,196	20,835
Wood:—				
Hewn .....	Loads 316,679	270,285	639,839	515,301
Sawn .....	" 857,234	858,428	1,826,959	1,864,233
Staves .....	" 17,007	16,912	52,305	61,234
Mahogany .....	Tons 3,007	3,332	26,312	25,787
Other articles....	Value £ ..	..	932,120	823,391
Total value .....	..	..	4,781,648	4,512,360

Besides the above, drugs to the value of 63,031*l.* were imported, as against 72,504*l.* in August 1893.

IMPORTS OF CHEMICALS AND DYE-STUFFS FOR MONTH  
ENDING 31ST AUGUST.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Alkali..... Cwt.	9312	7919	8,103	6322
Bark (tanners', &c.) ..	36,349	17,752	13,916	19,881
Brimstone..... "	21,756	79,368	7,015	15,178
Chemicals..... Value £	..	..	119,258	93,877
Cochineal ..... Cwt.	410	274	2,467	1,628
Cutch and gambier Tons	1,086	1,393	22,200	43,317
Dyes:—				
Aniline..... Value £	..	..	18,676	28,820
Alizarin ..... "	..	..	19,172	17,921
Other ..... "	..	..	2,122	2,111
Indigo ..... Cwt.	707	1,698	12,361	22,958
Nitrate of potash . "	26,982	25,435	22,724	22,935
Valonia ..... Tons	3,215	2,317	41,436	27,317
Other articles... Value £	..	..	129,839	97,674
Total value of chemicals	..	..	440,922	399,972

EXPORTS OF METALS (OTHER THAN MACHINERY) FOR  
MONTH ENDING 31ST AUGUST.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Brass..... Cwt.	10,461	7,742	40,151	29,146
Copper:—				
Unwrought..... "	35,879	41,537	85,011	91,601
Wrought..... "	25,874	26,600	73,912	69,635
Mixed metal .... "	21,977	23,844	51,881	50,292
Hardware ..... Value £	..	..	166,983	153,178
Implements..... "	..	..	95,809	92,464
Iron and steel..... Tons	281,832	210,977	1,579,846	1,572,183
Lead ..... "	4,353	3,161	48,012	38,566
Plated wares... Value £	..	..	26,211	23,022
Telegraph wires .. "	..	..	176,859	67,550
Tin ..... Cwt.	15,373	9,966	67,930	35,581
Zinc ..... "	9,779	12,142	8,564	8,374
Other articles .. Value £	..	..	70,372	51,019
Total value .....	..	..	2,691,541	2,278,711

EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING  
31ST AUGUST.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Alkali..... Cwt.	117,031	389,574	117,332	110,978
Bleaching materials ..	87,050	91,548	35,781	35,663
Chemical manures. Tons	18,705	49,162	237,926	218,698
Medicines..... Value £	..	..	76,765	81,738
Other articles ... "	..	..	237,369	210,705
Total value .....	..	..	715,113	659,722

EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH  
ENDING 31ST AUGUST.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Gunpowder..... Lb.	649,700	911,400	17,998	24,973
Military stores.. Value £	..	..	89,875	113,933
Candles..... Lb.	1,445,300	1,495,200	26,949	25,675
Caoutchouc .... Value £	..	..	107,979	96,274
Cement..... Tons	36,918	38,526	63,707	65,920
Products of coal Value £	..	..	75,955	77,330
Earthenware ... "	..	..	157,134	144,569
Stoneware ..... "	..	..	12,602	9,982
Glass:—				
Plate..... Sq. Ft.	171,278	119,965	8,785	6,647
Flint..... Cwt.	6,834	7,039	16,038	15,801
Bottles..... "	56,361	42,845	28,031	20,561
Other kinds.... "	15,717	16,984	11,846	11,798
Leather:—				
Unwrought .... "	11,734	12,066	109,399	105,891
Wrought ..... Value £	..	..	33,569	33,621
Seed oil..... Tons	4,934	3,862	108,843	81,355
Floorcloth ..... Sq. Yds.	1,583,709	1,629,500	65,117	66,821
Painters' materials Val. £	..	..	121,162	110,969
Paper ..... Cwt.	68,628	69,105	113,081	116,119
Rags..... Tons	3,318	4,412	19,738	22,872
Soap..... Cwt.	56,309	91,579	58,725	49,335
Total value .....	..	..	2,332,854	2,280,435

## Monthly Patent List.

\* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to oppositor within two months of the said dates.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### APPLICATIONS.

- 15,366. I. Levinstein. *See Class VII.*
- 15,413. E. Fudiekar. Improvements in and relating to apparatus for heating liquids, air, and other fluids. Complete Specification. August 13.
- 15,453. B. C. Tilghman, jun. An improved method of and apparatus for detecting and measuring the presence and amount of inflammable gases or vapours mixed with air. Complete Specification. August 14.
- 15,463. A. Bassett. *See Class XVII.*
- 15,736. W. J. C. Marie, Baron de Liebhaver. New apparatus to produce vapours of chlorhydric acid and other substances. August 18.
- 15,773. J. L. Osborn. — From C. Neuhaus, Austria. Improvements in apparatus for burning liquid fuel. August 18.
- 15,881. W. Oliphant. Improvements in filtering apparatus. Complete Specification. August 21.
- 15,885. J. C. S. McLay. Improvements in non-heat-conducting materials. August 21.
- 16,043. W. Spencer. Improvements in kilns for burning or calcining lime-stone or like substances. August 23.
- 16,235. A. Crerar. — From W. R. Boon and E. A. Hancock, West Indies. An improved process of and apparatus for evaporating or concentrating saccharine and other liquids. August 25.
- 16,246. H. B. Clark. A new and improved liquid refrigerator or cooling apparatus for breweries, dairies, and the like. August 25.
- 16,278. J. Swinburne. Improvements in and connected with electrical measuring instruments, organs, electrolysis, and working aluminium. August 27.
- 16,658. J. T. Bottomley. Apparatus for evaporation, distillation, or concentration of liquids. September 1.
- 16,692. F. Besin. An improved apparatus for the continuous heating of water. September 1.
- 16,728. F. W. Crossley and J. Atkinson. Improvements in apparatus for transferring heat or refrigerating. September 3.
- 16,938. E. Herscher. An improved continuous apparatus for the sterilisation of water or other liquid by ebullition. Complete Specification. September 5.
- 16,973. J. C. Sewell. New or improved apparatus for manufacturing heat-non-conducting material. September 6.
- 17,063. J. E. Smith. Improvements in apparatus for agitating or mixing materials, whether solid, liquid, gaseous, or mixtures thereof, applicable more especially for stuff chests for paper mills and chemical agitating machines. September 7.
- 17,160. T. Morton. Improvements in apparatus for heating, switching, or incorporating liquids, semi-liquids, or the like substances. September 10.
- 17,175. J. B. Hepburn and C. R. Honiball. Improvements in machinery for producing distilled water from sea or other impure water, and for concentrating liquids. September 10.

17,212. T. Glover and J. Brock. Improvements in or connected with concentrating or evaporating vessels. September 10.

17,219. R. Altsebul. Improvements in or connected with feeding liquid fuel, such as petroleum and other oily and inflammable substances, to steam-boiler or other furnaces, and burning same therein. September 10.

17,274. T. Craney. Improvements in apparatus for drying salt and other substances. Complete Specification. September 11.

17,289. H. E. Newton. — From E. Hart, United States. *See Class VII.*

17,314. J. S. Detwiler. Improvements in apparatus for treating powdered materials. Complete Specification. September 11.

17,315. J. S. Detwiler. Improvements in apparatus for blending powdered materials. Complete Specification. September 11.

17,374. G. Fouchard. Improvements in filters. September 12.

17,593. T. T. Best, S. A. Hollingsworth, and J. Brock. Improvements in apparatus for raising or pumping corrosive or other liquids. September 15.

#### COMPLETE SPECIFICATIONS ACCEPTED.\*

1893.

16,869. J. Gamgee. Improvements in vapour condensers or evaporators. September 5.

17,736. J. W. Pickles. The challenge sulphide rectifier. September 12.

19,630. G. E. Davis. Improvements in the construction of ammonia apparatus. August 22.

19,894. R. E. Dickinson. A new or improved bung for metallic casks or drums for containing mineral and other oils or the like. August 29.

20,303. T. F. Ennis and B. B. Waller. *See Class II.*

20,331. C. Bartelt and F. Bartelt. Improvements in apparatus for impregnating liquids with gases. September 12.

20,359. J. Musgrave and G. Dixon. Improvements in means for super-heating steam. September 19.

20,797. L. H. Bristowe. Improvements in filtering apparatus. September 5.

21,876. G. Dean. Improved Hoffman's kiln. September 19.

1894.

5187. C. Long and W. H. Barkitt. An improvement in acid raisers of steam jet pumps for raising acids, alkalis, and other liquids. September 5.

7318. T. Wheeler. An improvement in metallic thermometers and barometers. September 12.

7893. J. T. Sands. Improvements in apparatus for bleaching smoke. September 5.

9210. E. Cramer. Improvements in kilns for burning cement and other minerals. August 29.

14,184. F. Kleemann. Improvements in the process of and apparatus for beating and concentrating liquids. September 12.

15,153. B. C. Tilghman, jun. An improved method of and apparatus for detecting and measuring the presence and amount of inflammable gases or vapours mixed with air. Complete Specification. September 19.

### II.—FUEL, GAS, AND LIGHT.

#### APPLICATIONS.

15,469. A. McDougall. Improvements in or relating to the treatment of coal dust, small coal, and coke to improve them or render them fit for use as fuel or for the manufacture of gas. August 14.

15,486. E. Edwards.—From The Internationale Gesellschaft für Torfverwertung, Germany. Improvements in apparatus and process for manufacturing coke and charcoal and for separating the products obtained in such manufacture. August 14.

15,878. A. W. Burgess. Improved firelighter. August 21.

16,087. G. S. Cory and C. Cory. Improvements in the manufacture of artificial fuel. August 23.

16,149. H. Drehschmidt. A process for obtaining cyanogen or its compounds from the gases resulting from the dry distillation of substances of organic origin. August 24.

16,202. J. H. Crosby and M. McInerney. Improvements in firelighters, and in apparatus therefor. August 25.

16,342. G. B. Ellis.—From T. L. Willson, United States. Improvements in the production of metallic carbides and in illuminating gas. August 27.

16,395. W. F. Browne. Improvements in the manufacture of gas and in apparatus therefor. Complete Specification. August 28.

16,491. T. Barker. Improvements applicable to gas lights and other lights. August 30.

16,700. G. S. Cory and C. Cory. An improvement in the manufacture of so-called patent fuel. September 1.

16,701. G. S. Cory and C. Cory. Improvements in the manufacture of so-called patent fuel. September 1.

16,978. A. E. Tavernier and W. L. Malcolmson. A new and improved manufacturing of briquette fuel. September 6.

17,083. I. Carr. Improvements in or connected with the manufacture of gas. September 8.

17,219. R. Altschul. See Class I.

17,484. E. Kirbis. Method of manufacturing artificial fuel. Complete Specification. September 14.

17,491. G. Rose. Improvements in apparatus for the making of hydrogen gas to be used with an admixture of air for lead burning, brazing, and other purposes. September 14.

17,555. H. R. Dinsmore. Improvements in or connected with apparatus for making gas from coal. September 15.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

19,185. S. Psaroudaki and A. Blondel. Improvements in the diffusion and distribution of light. August 29.

20,303. T. F. Ennis and B. B. Waller. An improved gas-stoking tool. August 22.

21,485. J. Gray. Improvements in or connected with retorts for distilling shale or oil-yielding minerals. September 19.

22,185. C. D. Abel.—From M. M. Rotten and Co. Improved manufacture of fire-lighters. September 5.

1894.

11,657. J. T. Westcott. Improvements in apparatus for the manufacture of water-gas. September 5.

15,323. J. Schrader. An improved process for purifying petroleum. September 19.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

#### APPLICATION.

15,945. J. Y. Johnson.—From J. E. Pellagrini, Spain. Improvements in the manufacture or production of ethylene (bi-hydrocarbon  $C_2H_4$ ). August 21.

### IV.—COLOURING MATTERS AND DYES.

#### APPLICATIONS.

15,564. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. The manufacture and production of new azo dyes. August 15.

15,621. H. E. Newton. From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of dye-stuffs and materials therefor. August 16.

15,859. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. The manufacture of new dyes of the rhodamine series and intermediate products and leuco-compounds related thereto. August 20.

16,083. O. Imray.—From The Farbwerke vorm. Meister, Lucius, and Brüning, Germany. Manufacture of substituted ethenylidiphenylamines and ethenylmonophenylamines. August 23.

16,530. C. D. Abel.—From The Actien-Gesellschaft für Anilin-Fabrikation, Germany. Improved manufacture of colouring matters dyeing blue with mordants. August 30.

16,559. G. W. Johnson.—From Kalle and Co., Germany. Improvements in the manufacture or production of ortho- and para-oxy-ortho-toluic acids and meta-cresol. August 30.

17,048. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of a yellow basic colouring-matter. September 7.

17,049. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of yellow basic dye-stuffs. September 7.

17,133. E. Straub. Improvements in the manufacture of hydrazo compounds from aromatic nitro hydrocarbons. September 8.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

15,698. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of azo colouring matters from diamidodibenzimidazols. August 22.

18,924. J. J. Johnson.—From The Badische Anilin und Soda Fabrik. Improvements relating to the manufacture of dihydroxynaphthalene sulpho acids, and of corresponding azo-dyes. August 22.

19,623. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of colouring matters and new materials therefor. August 22.

19,624. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of a trihydroxy-naphthalene mono-sulpho acid. September 19.

19,880. R. Vidal. Improvements relating to the manufacture of colouring matters. September 22.

19,962. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik. Improvements in the manufacture and production of mordant dyeing colouring matters. August 29.

20,579. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of new colouring matters. September 5.

20,580. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture of a new anidonaphthol disulpho acid and colouring matters therefrom. September 5.

21,753. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik. Improvements in the manufacture and production of new azo dyes. September 19.

22,204. H. H. Lake.—From K. Oehler. Improvements relating to the manufacture of colouring matters. August 29.



1894.

15,064. J. C. L. Durand, D. E. Huguenin, and A. J. J. d'Andiran. Manufacture of new condensation products and colouring matters for dyeing and printing. September 12.

## V.—TEXTILES, COTTON, WOOL, SILK, Etc.

### APPLICATIONS.

15,718. H. H. Leigh.—From C. B. Schneider, Holland. See Class XIX.

15,727. C. Malley, J. Brett, and W. H. Hughes. A new and improved process for cleaning and bleaching jute, jute tow, and waste, flax and flax waste, and such like fibres, and preparing the same for spinning and felting. August 18.

15,815. J. V. Eves. Improvements in apparatus for use in the wet spinning of flax. August 20.

16,137. C. H. Arnold. Improvements in the manufacture of waterproof fabrics. Complete Specification. August 24.

16,541. J. M. Webster. Improvements in machinery or apparatus for the manufacture of wood wool. August 30.

16,648. J. Foulkes, J. E. Park, and G. N. Peake. Improvements in machines for scutching the leaves and stems of fibrous plants. September 1.

16,785. G. Jacober and A. Rabourdin. Improvements in the manufacture of filamentous products from cellulose. September 3.

17,070. E. Hornung and R. Liebl. Improvements in waterproof fabrics, garments, wrappers, and coverings, and in the process and composition for manufacturing the same. Complete Specification. September 7.

17,162. D. Whiteley. Improvements in apparatus for registering or recording the length of yarn produced in spinning, doubling, twisting, winding, and like textile machinery. September 10.

17,205. A. Dickins. Improvements in the treatment of wool and other textile materials with liquids, and in apparatus therefor. September 10.

17,370. G. Riche and A. Rivret. A process and apparatus for separating burrs and other foreign matters from wool. September 12.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

15,945. A. F. B. Gomess. A new and improved process for the treatment of textile vegetable fibres (more especially those of the urtica family). August 29.

1894.

7907. W. Pickstone and A. Greenhalgh. Improvements in and relating to the manufacture of crinkled textile fabrics. August 29.

13,672. E. Richard-Lagerie. Improvements in apparatus for extracting suint from wool and other substances. August 22.

14,299. W. Pickstone and A. Greenhalgh. An improved thick, soft, absorbent cloth. August 29.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

### APPLICATIONS.

15,365. W. B. Hart and C. A. McKerrow. Improvements in and connected with dyeing and printing vegetable fibres and goods made therefrom. August 13.

15,433. H. Wylie and J. Morton. Improvements in dyeing certain colours on cotton yarns and fabrics. August 14.

16,128. J. A. O'Loughlin. Improvements in producing patterns on indigo-dyed cotton fabrics. August 24.

16,252. J. W. Sanderson. Improvements in or relating to the process of indigo dyeing and to apparatus employed therefor. August 25.

16,661. C. D. Leng. Improved method of producing tinted or coloured covers for periodicals and the like. September 1.

16,987. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Process for the production of red tints by combining on the fibre paranitraniline with mixtures of betanaphthol and its sulphonie acids. September 6.

17,172. J. Pritchard and F. A. Blair. Improvements in apparatus for dyeing yarns and slubbings or rovings, or otherwise treating the same with liquids. September 10.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

20,916. J. C. Walker and J. E. Stephenson. Improvements in apparatus for scouring or washing wool and other fibrous substances. September 12.

21,087. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Process for producing insoluble azo colours on fibre. September 12.

21,843. W. P. Thompson.—From T. Salzmänn. An improved process and apparatus for treating dyed cotton yarns and the like —August 29.

1894.

14,223. H. H. Lake.—From M. E. Waldstein, A. H. Peter and E. Spott. Improvements relating to the dyeing of fibre. August 29.

## VII.—ACIDS, ALKALIS, AND SALTS.

### APPLICATIONS.

15,366. I. Levinstein. Improvements in apparatus employed in the concentration of sulphuric acid and other liquids. August 13.

15,473. F. L. Slocum and C. Y. Wheeler. Improvements in and relating to a process of making green oxide of chromium. Complete Specification. August 14.

15,702. S. B. Archdeacon. A process or method of treating siliceous material chiefly intended for the production of salts of sodium and potassium, and including the recovery of the waste products. August 17.

15,886. S. A. Saddler and R. H. Wilson. Improvements in process and apparatus for the manufacture of chlorine. August 21.

15,941. J. E. Chaster. Improvements in the manufacture of cyanides. August 21.

15,942. J. E. Chaster. Improvements in the manufacture of cyanides and apparatus therefor. August 21.

16,014. R. Loewenherz. Method of producing solid persulphate of sodium. August 22.

16,067. J. Grossmann. Improvements in the preparation and manufacture of hydrosulphurous acid and hydrosulphites. Complete Specification. August 23.

16,068. J. Grossmann. Improvements in the preparation and manufacture of hydrosulphurous acid and hydrosulphites. Complete Specification. August 23.

16,119. H. Drehschmidt. See Class II.

16,227. P. R. J. Krause. A new or improved process for the production of gas-formed hydrochloric acid by gas-formed nitric acid and chlorine. August 25.

16,258. H. H. Lake.—From J. H. C. Belinke and The Chemische Fabrik in Billwärd, vorm. Hell and Stäumer, A. G., Germany. Improvements relating to the manufacture of formates and of cyanides from the same. August 25.

16,320. E. Buckse. Process and apparatus for obtaining magnesium manganate and hydrochloric acid from the double chloride of manganese and magnesium, or for utilising certain residual liquors produced in the manufacture of chlorine. August 27.

16,342. G. B. Ellis.—From T. L. Willson, United States. See Class II.

16,529. G. Kraemer. Manufacture of potassium cyanide and yellow prussiate of potash from carbazol and its alkali compounds. August 30.

16,536. R. England. Improvements in or connected with the transport of sulphuric, hydrochloric, nitric, and other corrosive acids. August 30.

16,705. T. L. Willson. Improved metallic carbides and means for producing the same. September 1.

17,112. N. McCulloch. A new or improved method for oxidising the proto-salts of iron to the per-salts. September 8.

17,274. T. Craney. See Class I.

17,275. T. Craney. Improvements in salt evaporators. Complete Specification. September 11.

17,289. H. E. Newton.—From E. Hart, United States. Improved apparatus for the condensation of nitric acid and other vapours. Complete Specification. September 11.

17,556. J. Aseough. Improvements in the manufacture of sodium bichlorate or borax. Complete Specification. September 15.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

17,639 E. Dyson. Improvements in apparatus for concentrating sulphuric and other acids. September 19.

18,139. J. Reich. Improvements relating to the manufacture of alkaline carbonates and silicates. September 12.

18,786. J. B. Murray and M. B. Baird. An improved process for recovering alkali from a waste product. September 12.

20,104. C. T. J. Vantin. An improved process and means used therein for the production of caustic alkali. September 5.

20,604. D. A. Peniakoff. See Class X.

21,356. J. M. Milnes and A. Milnes. Improvements connected with apparatus for the manufacture of bleaching powder (chloride of lime) or similar commodities. September 19.

1894.

14,228. E. W. Enequist. Improvements in the process of manufacturing carbonic acid and in the production of by-products, such as sodium sulphate and magnesium sulphate. August 29.

15,473. F. L. Slocum and C. Y. Wheeler. Improvements in and relating to a process of making green oxide of chromium. Complete Specification. August 14.

#### VIII.—GLASS, POTTERY, AND EARTHENWARE.

##### APPLICATIONS.

15,607. R. C. Baker. Improvements in or relating to the manufacture of glass. August 16.

15,792. W. L. Pilkington. Improvements in the production of corrugated sheet glass, and in apparatus employed therein. August 20.

16,073. L. Böhm. Improvements relating to the decoration of glass. August 23.

17,096. P. Shelley. Improved material for sticking edges of pottery together during firing. September 8.

17,318. R. Stanley and G. Beck. Improvements in or relating to machinery employed in the manufacture of earthenware and like sanitary pipes. September 11.

17,366. Craven, Dunnill, and Co., Limd., and T. Pitt. Improvements in apparatus for the manufacture of tiles. Complete Specification. September 12.

17,579. T. Harvey. An improvement in the manufacture of flower pots and other decorative ornaments. September 15.

##### COMPLETE SPECIFICATIONS ACCEPTED.

1894.

9546. H. J. Haddan.—From The Columbian Pottery and Brick Kiln Co. Improvements in kilns. September 12.

9860. F. W. Golby.—From M. Hoffmann. A process for producing pressed glass plates for lining walls, ceilings, and for similar purposes. September 12.

14,938. W. Grane, sen. Improvements relating to the ornamentation of porcelain, glass, and the like. September 12.

#### IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

##### APPLICATIONS.

15,460. J. Sheppard and F. Dashwood. A combined fireproof floor and ceiling. August 14.

15,755. J. Elliott. A stone composition for repairing any natural stone and refacing cement floors, steps, &c., also for making imitation stone and bricks of any colour. August 18.

15,818. F. Young. Improvements in the manufacture of fireclay, refractory, and non conducting bricks and blocks, building bricks, or the like. August 26.

15,834. H. Wood. Improvements in the manufacture of bricks. August 20.

16,152. E. T. Hughes.—From J. Seipel, United States. Improvements in fireproof floor and ceiling constructions. August 24.

16,781. R. Haddan.—From J. Beauléquin, France. Improvements in the manufacture of material similar to mosaic. September 3.

16,875. W. West and O. V. Rhodes. A machine for forming rolls of stiff or plastic clay by means of a revolving and internally grooved or rifled cutter, which may be attached or affixed to a pug-mill or other clay-mixing machine. September 5.

17,059. J. H. Amies and J. U. Robertson. Improvements in pavements. Complete Specification. September 7.

17,208. F. H. Lenders.—From C. Bertin, Belgium. An improved composition forming a substitute for stone, marble, plaster, and the like. September 10.

17,221. J. Rueben. Improvements in or relating to fire-proof ceilings. Complete Specification. September 10.

17,486. J. Matthews. A composition for preserving wood, also applicable to other uses. Filed September 14. Date applied for 28th February 1894, being date of application in United States.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

19,939. A. Mack. Improvements relating to composite boards or slabs for building purposes. September 5.

22,227. M. Williams. Improvements in the manufacture of cement mortar. September 12.

1894.

3496. L. Tebbutt. New method of covering bricks, walls, and similar surfaces with a glazed or enamelled surface. September 19.

5626. T. L. Banks. Improvements in the construction of fireproof floors. August 29.

12,233. A. J. B. Ward. Non-slippery pavements, cellar covers, stair-treads, and the like. August 22.

14,599. E. Groebe. Improvements in the preservation of wood. September 5.

20,343. J. C. Montgomerie. Improvements in and in connection with the extraction of gold and silver from ores or compounds containing the same. September 5.

20,601. D. A. Periakoff. Improvements in the manufacture of alumina and aluminous compounds and metallic aluminium or alloys of aluminium, and obtaining chlorine, hydrochloric acid, carbonate of soda, and other by-products resulting from this manufacture. August 22.

20,684. E. Commelin. An improved furnace for roasting and smelting ores and reducing them to the metallic state. September 5.

22,397. B. Mierisch. Improvements in the extraction of gold and silver from ores or matte. September 19.

1894.

3224. J. M. While. Improvements in the manufacture of steel or iron plates and apparatus therefor. September 12.

11,447. J. J. W. Carr. Improvements in furnaces for melting, heating, or annealing metals, metallic alloys, and the like. September 12.

12,582. O. Klatte. New or improved process for producing rolled bars. August 29.

12,879. E. W. McKenna. Improved process for renewing old steel rails. September 5.

14,502. J. W. Dixon and W. Skinner. An improved metallic alloy. September 5.

### X.—METALLURGY, MINING, Etc.

#### APPLICATIONS.

15,761. R. Biewend. Process for treating sulphureous zinc ores in blast furnaces by the addition of ferri-ferrous substances. Complete Specification. August 18.

15,847. H. L. Sulman. Improvements in or relating to the precipitation of precious metals contained in solutions. August 20.

15,848. A. V. Newton.—From A. Nobel, France. Improved means for increasing the tenacity of metals. August 20.

15,891. R. A. Smith. A new metallic alloy. August 21.

16,041. W. K. Greenway. Improvements in the coating or galvanising of metals. August 23.

16,071. H. R. Angel. Improvements in the extraction of lead, silver, and gold contained in sulphide of zinc. August 23.

16,173. F. L. Lesage and A. F. Breuilland. Improvements in the method of and apparatus for obtaining metallic zinc from zinc ores. August 24.

16,557. W. P. Thompson.—From Stum Bros., Germany. Improvements in the manufacture of metal ingots. August 30.

16,785. W. Roberts. Improvements in utilising the bosh cinder produced in the puddling of iron, and in puddling and mill furnaces to be employed in utilising the said bosh cinder. September 3.

17,340. G. Love. Improved treatment of molten iron or steel. September 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

17,623. R. I. Roman. An improved solder for use with aluminium or aluminium alloys. August 22.

19,543. R. I. Roman. An improved aluminium alloy. September 5.

20,068. H. H. Lake.—From G. F. Simonds. Improvements in metallurgical furnaces. August 29.

### XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

#### APPLICATIONS.

15,712. D. B. Morison. Improvements in what are known as dry voltaic cells or batteries. August 17.

15,920. G. H. Reid. Improvements in and connected with primary batteries. August 21.

16,002. H. A. House, H. A. House, jun., and R. R. Symon. Improvements in apparatus for refining metal by electrolysis. Complete Specification. August 22.

16,100. H. Auer and F. Hunter. Improvements in apparatus for use in the electrolysis of alkaline chlorides and other alkaline salts. August 23.

16,105. R. Heathfield and W. S. Rawson. Improvements connected with the electrical deposition of zinc and other metals. August 23.

16,145. C. Hessel. Improvements in electrolytic processes and apparatus. August 24.

16,162. M. Engel. Improvements in accumulators or secondary batteries. August 24.

16,425. D. Young.—From The Hess Storage Battery Co., United States. Improvements in electric accumulators or storage batteries. Complete Specification. August 28.

16,686. C. Vogt. Improvements in the construction of galvanic cells. September 1.

16,696. H. Le Roy Bridgman. Improvements in or relating to electro-depositing apparatus. September 1.

16,697. H. Le Roy Bridgman. Improvements in or relating to electro-depositing apparatus and to products obtained thereby. September 1.

16,774. D. G. Fitz-Gerald. Improvements in the construction and working of voltaic batteries. September 3.

16,775. D. G. Fitz-Gerald. Improvements in voltaic batteries and electrolytic cells or tanks. September 3.

17,353. S. Miller and C. J. Grist. Improvements in charging primary batteries. September 12.

17,369. L. Epstein. An improvement in secondary voltaic batteries. September 12.

17,510. H. Le Roy Bridgman. Improvements in or relating to electro-depositing apparatus. September 14.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

16,307. J. Swinburne. Improvements in and connected with electric meters, for alternating currents. September 5.

17,466. W. Wright and J. B. Hamond. Improvements in the treatment of zinc produced by electrolysis. September 19.

19,688. J. C. Richardson. Improvements in safety appliances to be used in connection with electrical decomposing apparatus. August 29.

19,791. F. Hurter, H. Auer, and E. K. Muspratt. Improvements in or connected with electrolytic cells. August 29.

24,017. E. Stouls. Improvements in or relating to processes and apparatus for obtaining and depositing metals by electrolysis, and in the treatment of the metals after leaving the electrolytic bath. September 5.

1894.

9862. W. Morison. Improvements in portable primary batteries. September 12.

15,756. C. N. Waite. Improvements in or connected with electrolytic cells, and diaphragms therefor. September 5.

13,795. W. P. Thompson.—From C. M. Barber. Improvements in the process of electro-plating, and apparatus therefor. August 22.

14,642. V. I. Feeny.—From F. Dubero and P. Mohrdieck. Electrical battery. September 19.

15,276. M. Kiliani, W. Rathenau, C. Suter, and the Electrochemische Werke Gesellschaft mit Beschränkter Haftung. Improvements in or connected with diaphragms or electrolytic purposes. September 19.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

## APPLICATIONS.

15,780. G. Steeken. Improvements in the manufacture of soap. August 18.

16,040. S. Neumann. Improvements in or connected with the manufacture of fluorescent soaps. Complete Specification. August 23.

16,416. S. Rosenblum and F. L. Bartelt. Improvements in the treatment of vegetable, animal, and fish oils, fats, or grease. August 28.

16,417. S. Rosenblum and F. L. Bartelt. Improvements in the bleaching and refining of animal, vegetable, and fish oils, fats, and grease. August 28.

17,396. M. Evans. Improvement in bar soap. September 13.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

17,309. A. J. Inston. Improvements in recovering grease from waste suds which have been used in washing wool, hair, or other fibre. September 12.

19,475. J. P. F. Field. Improvements in soaps and washing compounds. August 22.

19,732. M. Baerlein. Improvements in the manufacture of soap compositions. August 29.

19,733. M. Baerlein and C. Dreyfus. A paste or mixture for cleansing various materials and articles. August 22.

1894.

1963. A. R. Scott. Improvements in the manufacture of cleansing soap powder for washing filcons and other materials. September 19.

13,672. E. Richard-Lagorie. Improvements in apparatus for extracting suint from wool and other substances. August 22.

14,821. W. O. Robbins. A new or improved apparatus for treating substances containing fats and oils. September 12.

## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, ETC.

## APPLICATIONS.

15,676. J. Tennant and A. Tennant. An improved composition paint. Complete Specification. August 17.

15,836. B. Angel and F. W. Lack. Improvements in paints and printing inks. August 20.

15,914. A. V. Newton.—From A. Nobel, France. An improved manufacture of artificial india-rubber, gutta-percha, and leather, suitable for conversion into varnish. August 21.

15,982. D. Williams. Improvements in or appertaining to a paint. August 22.

16,381. J. McLintock. Improved quick-drying anti-fouling paint. August 28.

16,664. R. Hutchinson. Improvements in treating bulky tree gum. September 1.

17,113. N. McCulloch. A new oil improved process for making red oxide of iron pigments from hydrated peroxide of iron. September 8.

17,145. W. B. Priest. Improvements in the manufacture of lead pigments or lead compounds for use as pigmentary materials. September 8.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

15,686. C. U. King and R. V. Jellicoe. An improved material or compound adapted for use as a varnish, as a substitute for india-rubber, gutta-percha, and like substances, and for other purposes, and the process for its production. August 22.

16,638. C. H. M. Lyte. An improved process for the production of iron oxide for use as a pigment. August 29.

16,800. T. W. Elliott. A new or improved composition for coating iron and steel. September 12.

17,159. W. V. Wilson and W. B. Priest. The manufacture or production of paints. September 12.

17,711. H. Mullerson. "Terpentine," a chemical preparation for cleaning and reviving all kinds of clothing. August 22.

19,163. L. Le Brocqy. A new substitute for india-rubber and gutta-percha. August 22.

19,284. M. Gerber. Improvements in devulcanising india-rubber and other like gums. August 22.

20,253. W. M. Walters and F. R. Stone. Improved preservative coating for iron and steel. September 5.

1894.

8112. C. W. Kriens. Improvements in the manufacture of animal charcoal. September 12.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

## APPLICATIONS.

16,611. S. H. Epstein. Improvements in the treatment of fancy leather. August 31.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

14,491. E. S. Spencer. Improvements in the manufacture of gelatine. August 1.

15,937. H. H. Lake.—From G. Sachsenroder. Improvements relating to the manufacture of a substitute for leather. August 29.

16,783. J. D. de Joseph. Improvements in or connected with tanning hides and skins. September 12.

1894.

14,697. H. Hartmann. A new or improved process for the production from skins or hides of a substitute for whalebone, horn, or the like. September 12.

## XV.—AGRICULTURE AND MANURES, Etc.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

17,235. G. F. Redfern.—From J. Hensel and F. Spiethoff. Improvement in the manufacture of mineral manure. September 12.

19,255. J. Morten. The universal weed, seed, worm, grub, and insect destroyer and fertiliser. September 19.

22,559. J. Carter. Improvements in making manure from sewage precipitate, sediment, residue after filtration, or from liquid sewage. August 22.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

## APPLICATIONS.

15,772. L. Fulda. Improvements in or connected with the manufacture of confectionery. August 18.

15,777. J. Y. Johnson.—From J. E. Pellegrini, Spain. Improvements in the manufacture of production of invert sugar or syrup. August 18.

15,976. P. S. Alexander. An improved process and machine for manufacturing confectionery. August 22.

16,235. A. Crerar.—From W. R. Boon and E. A. Hancock, West Indies. An improved process of and apparatus for evaporating or concentrating saccharine and other liquids. August 25.

16,759. C. M. Lafontaine. New treatment of raw sugars by sulphurous anhydrides and their derivatives by oxygenation for decolourating, purifying, whitening, and refining the crystallised mass by means of a special turbine, their direct agglomeration in parallelopipeds, slates, or equal cubes by a new machine for agglomerating sugar. September 3.

## COMPLETE SPECIFICATION ACCEPTED.

1893.

14,086. J. Y. Johnson.—From J. E. Pellegrini. Improvements in the manufacture of sugar and in the apparatus employed therein. September 19.

## XVII.—BREWING, WINES, SPIRITS, Etc.

## APPLICATIONS.

15,463. A. Bassett. Improved method of and apparatus for measuring or checking the production of alcoholic liquids, or the flow of liquids. August 14.

15,778. J. Y. Johnson.—From J. E. Pellegrini, Spain. Improvements in the manufacture or production of alcohol. August 18.

15,833. A. W. Billings. Improvements in the manufacture of beer. August 20.

16,704. G. C. Downing.—From L. Christ, France. A new and improved process and apparatus for extracting the saccharine juice from sugar-cane and bagasse. September 1.

17,143. T. D. Lichtenstein. The manufacture and production of a new material especially intended for use in the manufacture or treatment of beers, spirits, vinegar, aerated waters, and other beverages. September 8.

17,144. T. D. Lichtenstein. Improvements in the manufacture or treatment of beers, spirits, vinegar, aerated waters and other beverages. September 8.

17,277. J. Takamine. Improvements in diastatic enzyme taka-koji, diastase taka-koji ferment, and alcoholic ferment mash for developing active ferment cells. Complete Specification. September 11.

17,309. C. W. Ramstedt. A Pasteurising apparatus with rotating, heating and cooling elements. Complete Specification. Filed September 11. Date applied for 12 February 1894, being date of application in Sweden.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

19,549. F. N. Mackay. Improvements in and relating to brewing plant. August 22.

20,960. E. W. Greening and E. O. Greening. An improved method of utilising and means for treating yeast. September 19.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

## APPLICATIONS.

## A.—Chemistry of Foods.

15,558. D. A. Sutherland. An improved process and means used therein to effect the preservation of perishable substances. August 15.

15,906. I. Gilson. A new alimentary product. August 21.

16,548. R. Haddan.—From Kathreiner's Malzkaffee-fabriken mit beschränkter Haftung, Germany. An improved process for producing substitutes for coffee. August 30.

16,998. T. O. Kent. An improved coffee extract. September 6.

17,251. J. A. Wilson. Improvements in methods of treating meat in the raw condition in order to arrest or retard decay. September 11.

17,463. D. Cook. Process for treating meat, game, fish, and the like. September 14.

17,548. J. Gray. Improvements in and relating to the treating and aerating of milk. September 15.

## B.—Sanitary Chemistry.

15,620. R. A. Chesebrough. Improvements in the method of cremating garbage and other refuse. Complete Specification. August 16.

16,724. E. W. Ives. Improvements in screening and mixing chambers for precipitating tanks and the like for treating sewage and other foul or waste waters. September 3.

17,254. R. Pickup. Utilization of waste products for purpose of purification of foul waters. September 11.

17,372. W. Lawrence. Improvements in treating, purifying, and sterilizing water or contaminated liquids, and in apparatus therefor. September 12.

### C.—Disinfectants.

15,805. P. Nussbaum. A new process of making combustible capsules for medical and disinfecting purposes. August 20.

16,161. Sir W. G. M. Call, Bart. Improvements in antiseptic compounds and in the application of the same for the preservation of perishable goods. August 24.

16,414. M. S. Sharp. Improved means for disinfecting or deodorising. August 28.

16,422. R. England. Improvements in or connected with the manufacture of disinfectants. August 28.

16,448. J. Taylor. An improved manufacture of insecticide material. August 29.

### COMPLETE SPECIFICATIONS ACCEPTED.

#### A.—Chemistry of Foods.

1893.

20,027. W. P. Thompson.—From C. Artus. Improvements in the treatment and preparation of butter, butter substitutes, margarine, and alimentary fats or compounds. August 29.

20,477. T. Block. Improvements in the composition and manufacture of chocolate. September 5.

24,970. M. Watson. Improvements in cereal leguminous or other vegetable foods. August 29.

#### B.—Sanitary Chemistry.

1893.

21,024. T. Twynam. Improvements in the utilisation of sewage sludge. September 12.

21,820. T. W. Baker. Cremating and destroying obnoxious gases from dust destructors, &c. September 19.

1894.

13,777. W. J. Pughley. Improved means of purifying the effluent waters of tin-plate works and the like. August 22.

#### C.—Disinfectants.

1893.

19,622. H. E. Newton.—From the Farbenfabriken vormals F. Bayer and Co. Improvements in the manufacture of antiseptics. August 22.

### XIX.—PAPER, PASTEBOARD, Etc.

#### APPLICATIONS.

15,718. H. H. Leigh.—From C. B. Schneider, Holland. An improved process to render paper, cellulose, textile, or any other material fireproof and non-inflammable. August 17.

16,612. P. C. Peebles. An improvement in the manufacture of paper. August 31.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

20,036. C. D. Ekman. Improvements in obtaining useful products from the liquors resulting from the manufacture of cellulose or fibres by the sulphite process. September 12.

20,043. E. G. Law. Improvements in and relating to continuous paper for writing and copying purposes. September 19.

20,144. M. Levy, A. Rustin, and L. Boyer. Improvements relating to the manufacture of artificial whalebone, and to apparatus therefor. September 19.

20,353. J. F. H. V. Hoop. Improved decorative paper. September 5.

### XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

#### APPLICATION.

15,168. A. Sommer. Solution of sweet carbamides in oils, fats, waxes, resins, and process of making the same. Complete Specification. August 14.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

22,114. J. C. W. F. Tiemann. A process for converting compounds of the citral series (geranium series) into isomers having a higher specific weight and a lower boiling point than the original bodies. August 22.

12,174. The Peptine Maltine Co., Lim., and G. Bartlett. Peptonised extract of malted and other cereals. September 12.

14,138. L. R. Seammell. An improved process for the extraction and estimation of encalyptol. September 12.

1894.

15,468. A. Sommer. Solution of sweet carbamides in oils, fats, waxes, resins, and process of making the same. August 11.

### XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.

#### APPLICATIONS.

16,693. E. de Couinck. Improvements in the manufacture of dry coloured plaques for photography. September 1.

17,362. C. D. Durnford. Improvements in effecting photographic exposures. September 12.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

16,354. E. Albert. Improvements in the production of photographic negatives. August 29.

19,810. A. A. Barratt and A. J. F. Hill. A new or improved process for separating photographic gelatin films from celluloid supports or bases. September 12.

## XXII.—EXPLOSIVES, MATCHES, Etc.

## APPLICATIONS.

15,887. G. J. Baechert. Explosive compounds. Complete Specification. August 21.

15,977. R. W. Anderson. Igniting matches. August 22.

17,527. W. Hope. Improvements in sporting and other ammunition. September 14.

17,577. G. Trench. Improvements in blasting cartridges. September 15.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

20,327. A. Schindler. Improvements in the manufacture of gunpowder. August 29.

23,773. H. H. Lake.—From C. Pieper. Improvements in the manufacture of explosives. August 29.

1894.

9798. J. Hauff. Employment of trinitroresorcine as smokeless driving powder. September 5.

13,714. L. Jaroljmek. Improvements in or relating to the exploding of blast charges in mines. August 22.

14,068. R. Odeyn. A new or improved combustible composition. August 29.

## XXIV.—PATENT UNCLASSIFIABLE.

## COMPLETE SPECIFICATION ACCEPTED.

1893.

15,453. F. Chaplet. Manufacture or production of substances of extreme hardness. August 22.

# THE JOURNAL

OF THE

# Society of Chemical Industry:

A MONTHLY RECORD

FOR ALL INTERESTED IN CHEMICAL MANUFACTURES.

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Notice is hereby given that, in accordance with the alterations in Rule 27 sanctioned at the Annual General Meeting in Edinburgh, all new members elected after January 1st next, and also those elected after the date of this notice who do not pay subscription for 1894, will be required to pay an entrance fee of one guinea. In accordance with the same rule the Life Composition Fee will be 20/-, instead of 15/- as heretofore, after the 1st January next.



## BANKERS' ORDERS.

To the convenience of Members, the Treasurer has arranged for the Bankers of the Society that they shall collect subscriptions from Bankers in town and country; and Members, who have not already done so, are invited to fill up and sign the Banker's Order enclosed with the December number of the Journal, which should then be sent to the Honorary Treasurer, Mr. E. Rider Cook, East London Soap Works, Bow, E.

Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 41 of the bye-laws, the Society has the right of priority of publication for three months of all such papers. Infringement of this bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. EYRE and SPORRISWOOD, the Society's printers and publishers, to whom all communications respecting them should be addressed.

Notice is hereby given that the next List of Members will contain the professions or business occupations of the Members of the Society. Any Member who desires to confirm or alter his description as given before election should communicate with the General Secretary.

Foreign and Colonial Members are reminded that the subscription of 25s. for 1895, payable on January 1st next, should be sent in good time to the Treasurer, in order to ensure continuity in the receipt of the Society's Journal. Any changes of address to appear in the new List of Members now in course of preparation, should reach the General Secretary not later than January 15th, 1895.

## LIST OF MEMBERS ELECTED 23rd OCTOBER 1894.

Abbott, Cecil W., 6, Chichester Terrace, Brighton, chemical student.

Abbott, Fred. L., 6, Chichester Terrace, Brighton, chemical student.

Augell, Arthur, Shirley, Southampton, analytical chemist.

Aseough, Jesse, The Grange, Laurel Road, Handsworth, Birmingham, borax manufacturer.

Bamber, H. K. G., Portland Cement Works, Dovecourt, Harwich, Essex, chemist and manager.

Biekes, Theodor, 309, Manhattan Avenue, Brooklyn, N.Y., U.S.A., chemist.

Cousins, W. J., 11 and 12, Southampton Buildings, Canncery Lane, W.C., consulting chemist and managing director.

Crossley, Atkinson, The Scarlet Ferric Oxide Co., Talywain, near Pontypool, chemist.

Crow, Henry W., Abbey Creek Wharf, West Ham, E., tar distiller.

Dawson, Jas., 27, St. Vincent Place, Glasgow, drysalter.

Glen, Chas., Glengowan Printworks, Caldercruix, via Airdrie, N.B., calico printer.

Harrison, J. Arnold, 4, Ryverdale Road, Stoke Newington, N., chemical manufacturer.

Hatfield, Jno. A., 89, Bridge Street, Wednesbury, Staffordshire, analytical chemist.

Hodge, Andrew, Dalmonach Printworks, Alexandria, N.B., chemist.

Jameson, A. H., Cleveland Linseed Oil Co., South Chicago, Ill., U.S.A., chemist.

Leslie, H. Mackenzie, c/o Mysore G.M. Co. Ltd., Oorgaum, Mysore, South India, chemical engineer.

Lloyd, Harold T., 1, Hermon Hill, Snaresbrook, N.E., analytical chemist.

Lungwitz, Theo., c/o Pfizer and Co., 81, Maiden Lane, New York City, U.S.A., superintending chemist.

Mabey, Fred. O., 193, Amhurst Road, Hackney, N.E., wine merchant.

Mackay, Geo. A. D., 1, Parliament Square, Edinburgh, sanitary inspector.

Mills, Chas., 21, St. Mary Abbots Terrace, Kensington, W., chemist (colour works).

Muir-Smith, W., Carolina Port, Dundee, manager (Dundee Oil Works).

Nason, Carleton W., 71, Beckman Street, New York City, U.S.A., mechanical engineer.

Packer, Thos. J., Bayonne, N.J., U.S.A., chemical works manager.

Paul, Dr. Benjamin H., 20, Victoria Street, Westminster, S.W., consulting and analytical chemist.

Pilling, John E., 28, Agnew Villas, Whitegate Lane, Blackpool, chemist.

Price, Raymond B., c/o Boston Woven Hose and Rubber Co., Cambridgeport, Mass., U.S.A., chemical engineer.

Proude, Jas., 13, Oak Terrace, Halifax, Yorks, soap works chemist and manager.

Ridding, Howard C., 20, Algernon Road, Birmingham, (A.R.S.M.), analytical chemist.

Robertson, Alex., Argyle Chemical Works, Oban, N.B., manufacturing chemist.

Rowe, G. Vanner, 11, The Green, Stratford, E., chemist.

Russell, Oliver F., 54, St. Germain's Road, Forest Hill, S.E., analytical chemist.

Salter, Chas. T. C., 84, St. Paul's Road, Camden Square, N.W., scientific journalist.

Sandys, Hon. Michael, Pyrford Place, Ripley, Surrey, stockbroker.

Settle, Wm., 57, Bradford Street, Haulgh, Bolton, mining engineer.

Spence, J. Napier, Heathfield, Harrow-on-the-Hill, teacher of chemistry.

Towner, Rev. Geo., Carpenters' Company's Institute, Stratford, E., head master of Organised Science School.

Walker, Dr. Jas., University College, Dundee, professor of chemistry.

Wanklyn, Jas. Alfred, New Malden, Surrey, professor of chemistry.

Wells, Pierson L., 86, Joralemon Street, Brooklyn, N.Y., U.S.A., patent lawyer, M.E.

White, Arthur J., Ariston Gold Mine, Klerksdorp, South Africa, assayer.

Willidigg, A. E., Ford Cottage, Coventry, varnish maker.

Williamson, J. Alex., c/o Millom and Askam Hematite Iron Co., Askam-in-Furness, Lancashire, analytical chemist.

## CHANGES OF ADDRESS.

Baister, Howard C., l/o Liverpool; Elmhurst, Blundell-sands, near Liverpool.

Barrett, A., l/o Alabama; Bronheulog, Glandydon, Mostyn, North Wales.

Brookes, E. A., l/o Claremont Grove; 31, Bamford Street, Didsbury, Manchester.

Buchanan, Joshua, l/o Krugersdorp; United Langlaagte G. M. Co., Box 1018, Johannesburg, S.A.R.

Caldecott, W. A., l/o Eureka City; Barberton, S.A.R.

Conrad, E. C., l/o Gravesend; 1, Sunnyhill Terrace, Lowestoft Road, Gorleston.

Croft, A., l/o West Bridgeford; 55, Beeston Road, Old Lenton, Nottingham.

Dawson, W. Haywood; Journals to 135, Herbert Road, Woolwich.

Dixon, W. Hepworth, l/o Bow; Wingfield, Waustead, E.

Doolittle, O. S., l/o Franklin Street; 130, North 5th Street, Reading, Pa., U.S.A.

Duncan, Jas., l/o Dalbeattie; 9, Mincing Lane, London, E.C.

Edwards, H. W., l/o Canada; c/o Copper Queen Consolidated Mining Co., Bisbee, Arizona, U.S.A.

Forrester, Jas., l/o Finchley; 73, Ridge Road, Stroud Green, N.

Freeman, And., l/o 111; 98, Needham Road, Edgchill, Liverpool.

Fuerst, Dr. A. F., l/o Belsize Park; c/o Wood Street Smelting Works, 30, Wood Street, London, E.C.

Fulton, R., jun., l/o Saltecoats; Ardeer Factory, Stevenston, Ayrshire, N.B.

Gamble, W., l/o St. Helens; Briars Hey, Rainhill, near Liverpool.

Green, A. G., l/o Clapton; 13, King's Drive, Heaton Moor, near Stockport.

Hendrick, Jas., l/o Young Laboratory; Agricultural Department, 60, John Street, Glasgow.

Holmes, F. G., l/o New Cross; 1, Grange Avenue, Chapeltown Road, Leeds.

Johnson, E. C., l/o Alexandra Park; 422, Stretford Road, Old Trafford, Manchester.

Joy, Douglas G., l/o Brongh; Sealcoats Oil Mill, Hull.

King, W. Grant, l/o Ithaca; The Selvay Process Co., Syracuse, N.Y., U.S.A.

Knecht, Dr. E., l/o Moss Lane East; Station Road, Crumpsall, Manchester.

Liddle, G. A., l/o Carr Bank; 313, Derby Place, Walmersley Road, Bury, Lancashire.

Louis, H., l/o London; c/o Iberian Ore Co., Ltd., El Pedroso, Seville, Spain.

Lunn, C., l/o Liverpool; Woodsome Lees, Kirkburton, near Huddersfield.

Mann, H. H., l/o Paris; 7, Minster Yard, York.

Martyn, Wm.; Journals to Afton House, Hebburn-on-Tyne.

Mellon, W. W., l/o Howden; 16, Sandymount Road, Dublin.

Moffat, Alex., l/o Linlithgow; 104, Bothwell Street, Glasgow.

Nicol, Dr. W. W. J., l/o Birmingham; 15, Blacket Place, Edinburgh.

Parker, Thos., l/o Newbridge; Manor House, Tottenhall, Wolverhampton.

Phillips, A., l/o Barnes; Heriot Watt College, Edinburgh.

Pope, Frank, l/o Pearl Street; 31, Lawrence Street, Cambridgeport, Mass., U.S.A.

Radmacher, H. A.; Journals to Goddard House, East Walpole, Mass., U.S.A.

Rindoul, Wm., l/o Greenwich; Royal Compository, Waltham Abbey, Essex.

Rowell, W. A., l/o Hedworth; retain all communications until further notice.

Soyers, Jos.; Journals to Mayville, Stevenston, Ayrshire, N.B.

Schad, J., l/o Cooper Street; York Buildings, 33, Mosley Street, Manchester.

Schlichter, H. C., l/o Leipzig; Royal Ordnance Survey Department, Bangkok, Siam.

Sindall, R. W., l/o Station Street; "Daily Chronicle" Paper Mills, Sittingbourne, Kent.

Society of Public Analysts; Journals to Dr. Sykes, 59, Friends Road, Croydon.

Starling, J. H., l/o Belvidere; 32, Craven Street, Strand, W.C.

Tweedie, G. R., l/o Sturry; 3, Granville Terrace, East Cliff, Ramsgate.

Warne, Thos., l/o Lostock Gralam; 26, Bridge Street, Wodnesbury, Staffordshire.

Waterhouse, Robt., l/o Sheffield; 101, Leadenhall Street, London, E.C.

Weed, H. T., l/o Allegheny; 298, Herkimer Street, Brooklyn, N.Y., U.S.A.

Wild, W. E., l/o Cheetham Hill; Ousel Nest Cottage, Bromley Cross, near Bolton.

Wilson, Cecil H., l/o Grimssthorpe Road; 31, Miln Road, Burngreave Road, Sheffield.

Wright, L. T., l/o Hazlitt Road; 1, Victoria Street, Westminster, S.W.

Wuth, Dr. A., l/o Wiesbaden; 3, Hohenstrasse, Stuttgart, Germany.

Wyle, J. R., l/o Prescot; Hill House, Wavertree, near Liverpool.

## Deaths.

Pinkney, Robt., 18, Bread Street Hill, E.C. Sept. 29th.

Topley, Wm., F.R.S., Geological Survey Office, 23, Jernyn Street, London, S.W.

## London Section.

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E. J. Bevan.	S. Rideal.
W. G. Blazden.	A. Gordon Salamon.
E. Grant Herper.	A. Shearer.
T. A. Lawson.	Wm. Thorp.
R. Messel.	T. Tyrer.
H. de Meuthal.	Frank Wilson.
E. O'Neil.	

*Hon. Local Secretary:* John Heron,  
74, North Side, Clapham Common, S.W.

### SESSION 1894-95.

Monday, November 5th:—

The late Dr. C. R. Alder Wright, F.R.S. The Paper to be read by Mr. Watson Smith, F.I.C., F.C.S. "The Composition and Constitution of Certain Alloys."

Mr. F. W. Read, F.I.C., F.C.S. "Note on the Oxidation of Lensed Oil."

Monday, December 3rd:—

Dr. Kuhn of Paris. "The Sterilisation of Water."

Dr. D. H. Atfield, M.A., Ph.D. "An Investigation of the Natural Sodium Sulphate Lakes of Wyoming, U.S.A."

## Liverpool Section.

UNIVERSITY COLLEGE, BROWNLOW STREET.

*Chairman:* E. Carey.

*Vice-Chairman:* H. Brunner.

*Committee:*

J. Campbell Brown.	G. Schack-Sommer.
Jos. C. Gamble.	E. Scott.
C. L. Higgins.	A. Smetham.
C. A. Kohn.	Frank Tate.
E. K. Muspratt.	H. Tate, jun.
W. Norris Jones.	

*Hon. Treasurer:* W. P. Thompson.

*Hon. Local Secretary:*

Dr. Lewis Bailey, University College, Liverpool.

### SESSION 1894-95.

November 7th.—Chairman's Address. "The Invasion of the Inventor."

December 5th.—Mr. Eric E. Watson. "The Calculation and Composition of Blast-Furnace Charges, as applied to the Smelting of Lead and Silver Ores."

## Newcastle Section.

*Chairman:* G. T. France.

*Vice-Chairman:* Alf. Allhusen.

*Committee:*

P. P. Bedson.	B. S. Proctor.
T. W. Hogg.	W. W. Proctor.
T. W. Lovibond.	W. L. Reimoldson.
W. Martyn.	W. A. Rowell.
John Morrison.	T. W. Stuart.
John Pattison.	John Watson.

*Hon. Local Secretary and Treasurer:*

Samuel Shaw, Durham College of Science, Newcastle-on-Tyne.

### SESSION 1894-95.

Thursday, November 8th.—Dr. G. Lunze. "Practical Results of the Lunze-Rohrmann Plate-Towers for Condensing Hydrochloric Acid."

## Scottish Section.

*Chairman:* Robt. Irvine.

*Vice-Chairman:* J. Clark.

*Committee:*

G. Beilby.	J. S. Macarthur.
R. Cox.	E. Ostlere.
C. J. Ellis.	T. L. Patterson.
C. A. Fawsitt.	R. Pullar.
D. Harris.	J. B. Readman.
G. G. Henderson.	H. Rose.
R. A. Inglis.	E. C. C. Stanford.
W. I. Macadam.	D. R. Stewart.

*Hon. Secretary and Treasurer:*

J. Stanley Muir, Chemical Laboratory, University of Glasgow.

### SESSION 1894-95.

*Dates of Meetings.*

Glasgow.—Philosophical Society's Room, 267, Bath Street. November 6th; January 8th; March 5th; May 7th.

Edinburgh.—Philosophical Institution, 4, Queen Street. December 8th; February 5th; April 2nd.

November 6th.—Glasgow. Chairman's Address. "Shale Distillation and the Purification of the Products therefrom."

## New York Section.

*Chairman:* Alfred H. Mason.

*Vice-Chairman:* Arthur McGeorge.

*Committee:*

G. T. Bruckmann.	E. G. Love.
T. Lynton Briggs.	John McKesson.
H. Comer.	W. H. Nichols.
H. Endemann.	Francis J. Schleicher.
Jos. D. Geisler.	Jas. H. Stebbins, jun.
Jas. Hartford.	T. B. Stillman.

*Hon. Treasurer:* R. C. Woodcock.

*Hon. Local Secretary:*

Dr. H. Schweitzer, 159, Front Street, New York, U.S.A.

Notices of Papers and Communications for the Meetings should be sent to the Hon. Local Secretary.

## Journal and Patent\* Literature.

Class.	Page
I.—General Plant, Apparatus, and Machinery .....	929
II.—Fuel, Gas, and Light .....	931
III.—Destructive Distillation, Tar Products, &c. ....	938
IV.—Colouring Matters and Dyes .....	950
V.—Textiles: Cotton, Wool, Silk, &c. ....	941
VI.—Dyeing, Calico Printing, Paper Staining, and Bleaching .....	945
VII.—Acids, Alkalis, and Salts .....	947
VIII.—Glass, Pottery, and Enamels .....	948
IX.—Building Materials, Clays, Mortars and Cements..	949
X.—Metallurgy .....	950
XI.—Electro-Chemistry and Electro-Metallurgy .....	957
XII.—Fats, Oils, and Soap Manufacture .....	959
XIII.—Pigments and Paints; Resins, Varnishes, &c.; India-Rubber, &c. ....	962
XIV.—Tanning, Leather, Glue, and Size .....	964
XV.—Manures, &c. ....	964
XVI.—Sugar, Starch, Gum, &c. ....	965
XVII.—Brewing, Wines, Spirits, &c. ....	966
XVIII.—Chemistry of Foods; Sanitary Chemistry and Water Purification; Disinfectants .....	969
XIX.—Paper, Pasteboard, &c. ....	970
XX.—Fine Chemicals, Alkaloids, Essences, and Extracts	970
XXI.—Photographic Materials and Processes .....	973
XXII.—Explosives, Matches, &c. ....	973
XXIII.—Analytical Chemistry .....	975

## I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

*The Standard Wire Gauge.* Chem. Trade J., 15, 1894, 239.

THE following table constitutes the only legal wire gauge in the United Kingdom. A "Mil." equals the one-thousandth part of an inch. For convenience of comparison the equivalent in millimetres is also given.

No.	Mils.	M.M.	No.	Mils.	M.M.
0,000,000	500	12.70	10	128	3.25
000,000	464	11.78	11	116	2.95
00,000	432	10.97	12	104	2.64
0,000	401	10.16	13	92	2.34
000	372	9.45	14	80	2.03
00	348	8.84	15	72	1.83
0	324	8.23	16	64	1.63
1	300	7.62	17	56	1.42
2	276	7.01	18	48	1.22
3	252	6.40	19	40	1.016
4	232	5.80	20	36	.914
5	212	5.33	21	32	.813
6	192	4.88	22	28	.711
7	176	4.47	23	24	.610
8	160	4.06	24	22	.559
9	144	3.66	25	20	.508

\* Any of these specifications may be obtained by post by remitting 8d.—the price now fixed for all specifications, postage included—to Sir Henry Reader Lack, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

N	M	M.M.	No.	M	M.M.
26	18	1.17	30	2	.162
27	16.4	1.07	40	1.5	.122
28	14.8	.976	11	4.1	1.2
29	13.6	.915	12	1.9	.102
30	12.4	.845	31	3.6	.914
31	11.6	.795	41	3.2	.813
32	10.8	.734	15	2.8	.7071
33	10.0	.684	46	1.1	.6030
34	9.2	.634	17	2.0	.6098
35	8.4	.583	18	1.6	.6046
36	7.6	.543	49	1.2	.6005
37	6.8	.513	50	1.0	.6024
38	6.0	.482			

### PATENTS.

*An Improved Apparatus for Use in Measuring the Pressure of Gas in Guns.* A. Castenholz, Karlsruhe, Germany. Eng. Pat. 11,991, August 1, 1893.

THE invention relates to apparatus for use in measuring the pressure of gas which in the combustion of a certain quantity of powder or other explosive material, is produced in guns. An improved apparatus is described for measuring the gas pressure for small arms and cannon, characterised by a cartridge case without a base, a sleeve having the same bore and fitted gas tight, and a pressing stamp or plug adapted to move axially therein and provided with a central firing device, which said stamp or plug forced back by the explosive gases upsets or "jumps" a cylinder, the amount of upset being proportional to the pressure. A modification of the above is also claimed.—J. C. C.

*Improvements in Filter Presses.* J. Critchlow, Stoke-on-Trent. Eng. Pat. 17,024, September 11, 1893.

THE inventor proposes to provide the supporting arms of each movable plate of a filter press with rollers, running upon the side frames of the press. Also to fit to each plate similar rollers bearing horizontally against the frames. The object of the invention is to facilitate the manipulation of the press plates.—B.

*Improvements relating to Metallic Tubes used for Heating Liquids.* H. and A. J. Goldthorpe, both of Wakefield. Eng. Pat. 20,362, October 28, 1893.

TO prevent the wearing away of the apertures made for the escape of steam, in tubes used for boiling and heating, a stout nipple is screwed into the aperture. The nipple may be straight or conical, and pierced with one or more holes for the escape of steam.—E. G. C.

*Improvements in and connected with Charging Vessels with Volatile Liquids or other Substances and Hermetically Sealing the same.* J. Macrae and J. Dickson, both of London. Eng. Pat. 20,697, November 1, 1893.

A RESERVOIR containing the volatile liquid has a tubular outlet below, furnished with a nozzle connected thereto by a flexible tube, which latter can be closed by a spring clip; a movable plug regulates the supply of air and consequently the flow of liquid from the reservoir into the receiving tubes. The narrow necks of the charged tubes, which are of glass, are afterwards made red hot by means of a blow pipe and pinched together by tongs or nippers, thereby hermetically sealing the tubes; as a further precaution the closed neck is dipped in liquid gutta-percha or shellac. A

of test-tube stand is provided for holding the tubes. To facilitate the operation of sealing the tubes, the glass of which they are formed should be soft.

*Filtering Apparatus.* L. H. Bristowe, London. Eng. Pat. 20,797, November 2, 1893.

The invention relates to filtering apparatus in which the liquid to be filtered passes through radial arms perforated along one side and radiating from a central hub or hollow shaft through which the liquid enters. In the usual arrangement before the liquid can reach the outer end of the radial arm, a good deal of the working pressure is lost through part of the liquid escaping by the perforations near the central hub, to avoid which the inventor divides the arm into two compartments by means of a central longitudinal division reaching to within a short distance of the outer end. The liquid passing outwards by the unperforated compartment, arrives at the outer end of the arm before encountering any perforations, and thence travelling towards through the perforated part, is more efficiently distributed over the filtering medium.—B.

*Improvements in Acid Raisers or Steam Jet-Pumps for Raising Acids, Alkalies, and other Liquids.* C. Long, Walsall, and W. H. Burkitt, Wednesbury. Eng. Pat. 5187, March 13, 1894.

THE main object of the invention is to construct pumping apparatus having easily removable parts, especially the steam-jet, which is liable to become worn, and, by admitting too much steam, to occasion needless dilution of the liquid.

Fig. 2.

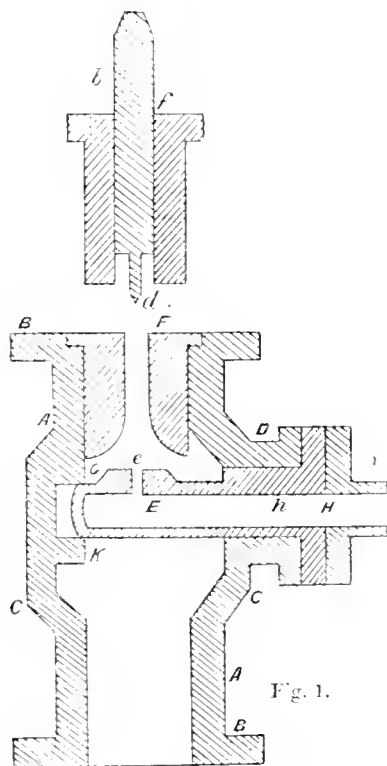


Fig. 1.

IMPROVED ACID RAISER OR STEAM-JET PUMP.

In the drawing, Fig. 1 shows the pump, consisting of a "globe-shaped" case A, reduced at the ends, with flanges B, and two flat sides C, on one of which is a branch D for reception of the steam-jet pipe E, made square or oblong

that it may not get out of position, and having a raised boss G when made for acids; H and h are flanges. A cup or rest K is provided for the jet pipe, which is thus relieved of strain.

To renew the acid raiser, the jet pipe is taken out, and the jet hole e is filled up with suitable metal by a flame. The jet pipe is then replaced, and the delivery nozzle F is removed, and the guide bush f (Fig. 2) is inserted; then by means of a turned drill b, the drill is shouldered down d so as to drill a jet hole of the desired diameter through the tused-in metal. The jet hole is thus renewed and brought into perfect line. When the apparatus is used for alkalis, a new jet hole may be drilled as described, or a new jet may be screwed into the jet pipe.—E. S.

*Improvements in Condensing Plates.* J. W. Mackenzie, London. From Brante and Company, Lyons, France. Eng. Pat. 7229, April 11, 1894.

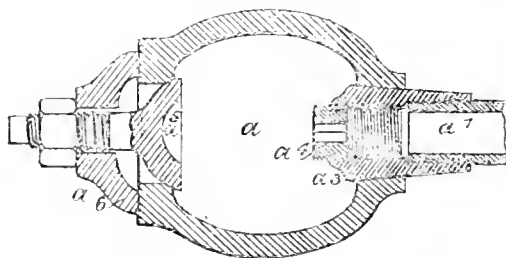
IN condensing sulphuric, nitric, hydrochloric, and acetic acids, or other products requiring large condensing surfaces, plates about 32 cm. long and 10 cm. in width and thickness are used. The plates are provided with projections, funnel-shaped openings, channels, and bores of various shapes, of which designs are shown; and the material is such as may resist the action of the substances to which they are exposed. It is stated that by a combination of such plates "all the conditions required from the Glover's and Gay-Lussac towers and from lead chambers are attained."—E. S.

*Improvements in and relating to Tubes employed for Heating, Vaporising, Refrigerating, and other Purposes.* A. Dumas, Brévannes, France. Eng. Pat. 8320, April 26, 1894.

WITHIN tubes used for heating, vaporising, and refrigerating, are placed flat, curved, or spiral wings for the purpose of giving more exposed surface and thereby increasing the efficiency of the tubes for the purposes for which they are employed.—E. G. C.

*Improvements in Steam Superheaters.* J. Grouvelle and H. Arquembourg, Paris. Eng. Pat. 13,586, July 13, 1894.

THIS invention relates to the coils of tubular zig-zag superheaters, disposed in two or more series, and so arranged as to expose those tubes which first receive the moist steam to the greatest heat in the flues. Part of the invention also refers to the device for connecting the tube ends to the steam-collectors, as indicated by the accompanying cross-section, in which a represents the section of the collecting



IMPROVEMENTS IN STEAM SUPERHEATERS.

vessel. Each tube-end a<sup>1</sup> is screwed into a conical plug a<sup>2</sup>, tightly driven into a corresponding hole in the collecting vessel. The plug has also a screw-thread at its inner end, admitting of the use of a tool for screwing the plug to the tube-end, and of the subsequent insertion of an auxiliary plug a<sup>3</sup>, whose centre is pierced with an opening of a size to determine the quantity of steam passing through the tube. A removable cap a<sup>4</sup> is fitted to the collecting vessel opposite each tube-end, as shown.—B.

*Improved Apparatus for Raising, Forcing, and Circulating Semi-Liquid and Liquid Substances.* J. Taylor, Wandsworth Common, Surrey. Eng. Pat. 13,709, July 16, 1894.

The apparatus is intended principally for dealing with semi-liquid substances, such as paper-pulp, sewage, and the like, for which ordinary pumping apparatus is unsuitable, and it consists of a modified form of centrifugal pump, with an axial inlet and tangential outflow. The vanes of the pump-wheel are spiral, and curved to a D shape in cross-section; and suitable scrapers are inserted through the casing, to clear the vanes from adherent matter. The apparatus is mounted upon a bed-plate, and the vane-wheel shaft, carrying fast and loose pulleys for driving, is supported upon suitable bearings.—E. G. C.

## II.—FUEL, GAS, AND LIGHT.

*Heating Power of Smoke.* R. R. Tatlock. Chem. News, 70, 1894, 51.

It appears to be generally imagined that a large percentage of fuel is lost in the smoke which issues so abundantly from most chimneys. A little consideration, however, will show that the loss of any large percentage of combustible matter, and consequently of heating power, is quite out of the question. This may be proved in two ways—(1) by calculation of the two sources of heating-power as shown by an analysis of coal or dross used for steam-raising, and (2) by actual analysis of the furnace gases for combustible solids and gases.

In the following paper are given the results of these two methods of observation, the same dross being analysed and also employed as fuel in a works furnace, from which smoky gases were given off which were tested for combustible matters.

1. The following is the analysis of the dross employed:—

	Per Cent.
Gas, tar, &c. ....	37.63
Fixed carbon.....	49.97
Sulphur.....	0.40
Ash.....	2.72
Water.....	9.28
	100.00
Heating-power (practical) due to gas, tar, &c.....	1.16
Heating-power (practical) due to fixed carbon ..	6.49
	7.65

The points to be observed are the relative proportions of heating-power (represented in the analysis by the number of pounds of water at 212° F. capable of being evaporated by 1 lb. of the fuel) given out respectively by the combustion of gas, tar, &c., and by the fixed carbon. These are calculated according to Playfair's well-known formula, which was practically tested on coals intended for the British navy, and which shows that while 1 lb. of fixed carbon is capable when burned of evaporating 13 lb. of water at 212° F. to dryness, 1 lb. of the gas, tar, &c., will only evaporate 3.1 lb. From these figures it appears that in the coal or dross the gas, tar, &c., only contribute 15 per cent. of the total heat given out during the combustion, and that the fixed carbon produces the remainder, or 85 per cent. In coals with less of the former ingredients and more of the latter, which is commonly the case, the proportion given out by the volatile constituents would be considerably reduced. It is thus perfectly clear that even though the whole of the volatile matters (which can alone be accountable for any loss of combustible material) escaped combustion, there could not possibly be a greater loss of heat than 15 per cent. of the whole, even in such an extreme case as this represents.

2. An analysis was made of the furnace gases given off during the burning of the dross, of which the results are given above, with the following results:—

	Grains per 100 Cubic Feet of Smoke.	Grains per 100 Cubic Feet of Furnace Gases.
	Per Cent. by Volume.	Per Cent. by Volume.
Carbonic acid.....	5.90	3.75
Carbonic oxide.....	None	None
Hydrocarbons.....	Trace	None
Nitrogen.....	77.9	79.9
Oxygen.....	15.1	14.9
	100.00	100.00

It has been asserted that carbonic oxide is given off in considerable quantity when much smoke is being produced, but it does not appear in this case; and Hempel, in his work on "Gas Analysis," comes to the conclusion that little or no combustible gases are present in furnace gases. In the volume referred to (page 205) Hempel says "furnace gases usually contain only carbon dioxide, oxygen, and nitrogen. All other gases are present in but very small amounts. In oft-repeated analysis the author has always found only traces of carbon monoxide, methane, and the heavy hydrocarbons."

That there is loss of combustible matter in the smoke is an undoubted fact, but the quantity seems also to be greatly magnified in certain random statements. In the experiment referred to above, the soot was also collected during one hour and a half with following results:—

	Grains per 100 Cubic Feet of Furnace Gases.
Carbonaceous matter .....	3.481
Ash or mineral matter .....	20.65
Total soot.....	24.13

It will be observed that the soot collected, consisted largely of mineral or incombustible matter. In several experiments to estimate the soot in furnace gases similar results to these were obtained, and the average would come very close to the quoted results of this special test.

To find how much carbonaceous matter was actually lost as smoke, it will be necessary to know the number of cubic feet of furnace gases given off by the combustion of, say, 1 ton of the dross. If the percentage of carbonic acid in the furnace gases is taken at 5 per cent., the total volume of these given off from 1 ton of dross would be about 940,000 cubic feet measured at the ordinary temperature and pressure, and this would contain 41 lb. of carbonaceous matter and 27 lb. of mineral matter. This would represent 1.83 per cent. of the volatile matters (gas, tar, &c.) given in the analysis of the dross; and if from this is now calculated the heating-power according to Playfair's formula, it will only come to 0.057. This figure, compared with the practical heating-power (7.65) of the dross, goes to show that the solid combustible matter of the smoke can only account for the very small percentage of 0.74 of the total heating power which can be obtained from the coal.

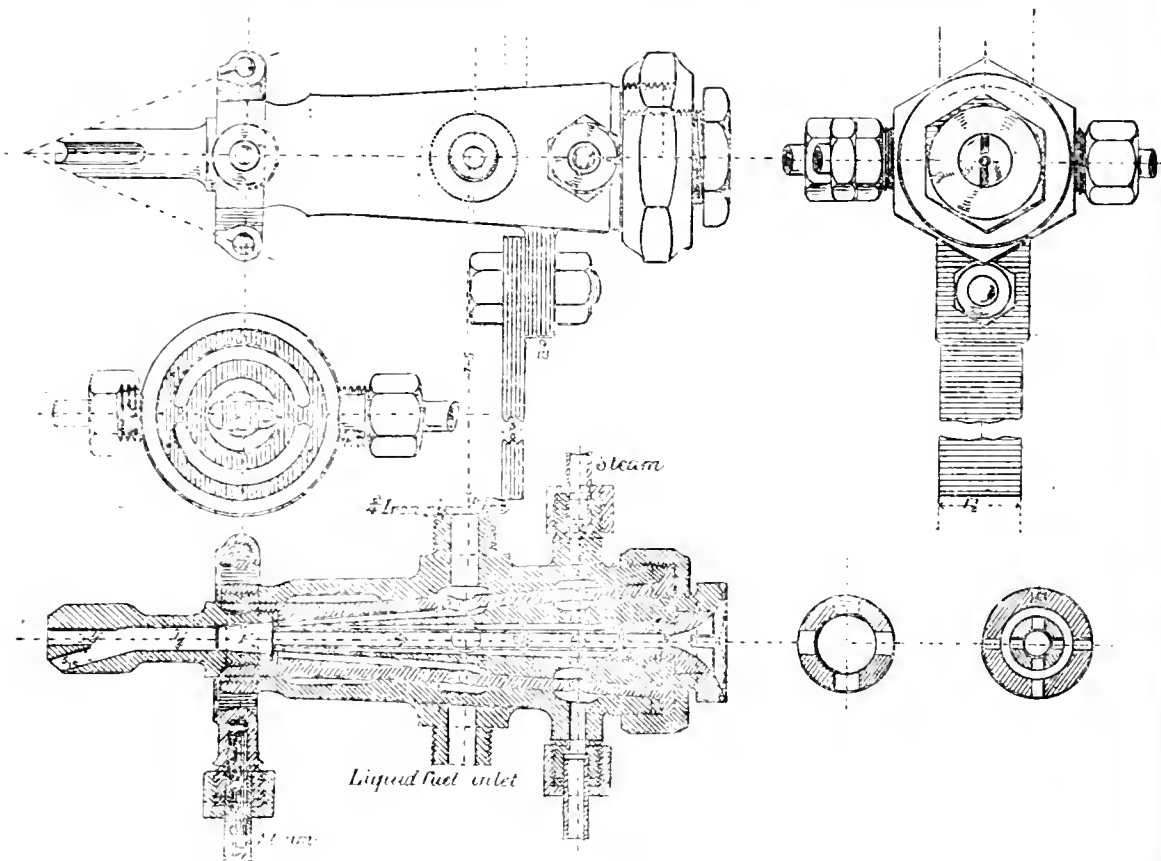
From the results of these experiments it is evident that the loss of combustible matters in smoke is very small indeed. In adopting methods of removing the smoke nuisance, it must therefore be borne in mind that there is little or no gain in burning smoke, and that other methods of dealing with the problem, such as Dalser's smoke absorption process, ought also to receive consideration.

*The Holden Liquid Fuel Burner.* Eng. and Mining J.  
1894, 101.

The accompanying drawings illustrate an apparatus and system for using liquid fuel which was devised by Mr. W. F. Holden, locomotive superintendent of the Great Eastern Railway, and which has been very successfully used there and elsewhere. Elevations and sections of the burner are shown and also its application to a boiler of the locomotive

type. It can, of course, be used with other types of boilers, with modifications in the arrangement which will readily suggest themselves.

By this system liquid fuel and air are introduced into the firebox above a thin layer of solid incandescent fuel by means of a special injector, and burnt in combination with the solid fuel, without any alteration of the firebox other than the insertion of one or more tubes through the casings,



HOLDEN'S LIQUID FUEL BURNER.

the boiler so fitted being equally suitable for the use of ordinary fuel. The use of this method results, it is claimed, in effectual combustion, entire absence of smoke, intense and regular heat, and great economy of fuel. Slack, inferior coal, lignite, cinders, wood, peat, or sawdust may with equally good results be used as solid fuel. As the air necessary for combustion need not be introduced through the fire, a very small amount of draught is required, and in the case of locomotives the orifice of the blast pipe may advantageously be enlarged from 50 to 60 per cent., reducing the wear and tear of the firebox, tubes, smokebox, and chimney, preventing the emission of sparks and ashes, and diminishing back pressure. The steam pressure in a boiler fitted with the apparatus can be regulated with great nicety, as the solid fuel required is only sufficient to form a base for ignition, and the steam pressure can be promptly increased or reduced by varying the quantity of liquid fuel. Where an engine is doing intermittent work, as in hoisting for instance, this is often a great advantage.

The chief novelty in Holden's injector consists in the provision of an annular ring near the nozzle from which small jets of steam or air issue. These small jets perform the following duties:—1. They introduce atmospheric air close to the streams of liquid fuel issuing from the nozzle, sufficient to insure perfect combustion and consequently

entire freedom from smoke. 2. They effectually break up the spray of liquid fuel, and secure thorough admixture of the hydrocarbon atoms with the air induced. 3. They diffuse the flames over the surface of the furnace or firebox and prevent local heating of the boiler plates. The internal cones can be readily removed for cleaning, &c., by unscrewing the large nut at the back. The central air tube provides means for clearing the nozzle without disconnecting the injector. On locomotives, two injectors are generally used where the grate is more than 2 ft. 6 in. wide. On stationary boilers the number will be determined by the size of the firebox.

Where steam is used intermittently it is believed that a considerable economy can be effected by the use of this system. It has been fitted with equally good results and worked by compressed air upon rivet furnaces, forge furnaces, &c. Where steam is required at a short notice and at a constant pressure, this system is found valuable for such purposes as central stations for electric light, factories, gasworks, &c. Long extended tests with this system show that 14 lb. of water are evaporated by 1 lb. of dead oil. On stationary boilers it is found that on an average 1 ton of oil does the same work as 1.75 tons of coal, and in many instances 1 ton of oil is equal to 2 tons of coal.

*Carburettling.* H. Bunte. *Journ. für Gasbeleuchtung*, **37**, 81.

THE author first discusses the causes which operate against the extended use of carburetted water-gas in Germany. Even in the best water-gas processes there is often a loss of

50 per cent. of the heat value of the coal. One chief source of this loss is the passage of undecomposed steam through the generator whenever the temperature is lowered. Experiments in this direction furnished the results here tabulated.

Mean Temperatures ° C.	Composition of the Water-Gas Produced.			Steam.		Speed of the Current of Gases per Second.
	H. Vol. per Cent.	CO. Vol. per Cent.	CO Vol. per Cent.	Decomposed per Cent.	Undecomposed per Cent.	
674	65.2	1.9	29.3	8.8	91.2	0.9
758	65.2	7.8	27.0	1.33	71.7	1.8
838	62.4	13.1	24.5	34.7	65.3	3.66
838	61.9	15.1	22.9	41.0	59.0	3.28
861	59.9	18.1	21.9	48.2	51.8	5.3
954	53.3	39.3	6.8	70.2	29.2	6.3
1,010	48.8	49.7	1.5	94.0	6.0	6.15
1,060	50.7	48.0	1.3	93.0	7.0	9.8
1,125	50.9	48.5	0.6	99.1	0.6	11.3

*Carburettling of Coal-Gas.*—Since benzene has been recovered from the gases of coke-ovens, it has come into the market at so low a price that our attention is naturally directed towards it as a means of carburettling coal-gas. The four million kilos. of benzene produced in this way up to the present would suffice, according to the author's previous experiments (*Journ. für Gasbeleucht.* **36**, 442—449; this *Journal*, 1893, 817—819) for the improvement of 800 million cubic metres of gas, by one Hefner light (= 0.886 "standard candle"). Since in Germany, at present, less than 10 per cent. of the coke ovens are provided with plant for the recovery of benzene, its production is capable of enormous extension, and there is little fear of a rise of price consequent on its employment for carburettling purposes.

The cheapness of benzene may allow of a further step being taken. Coal-gas has hitherto been produced in retorts rather than in large distillation chambers, chiefly because in the latter, methane, and especially benzene, may be decomposed in passing over heated layers of coke, and the illuminating power of the gas would thus be decreased. But now, considering that we have a cheap source of carburettling, and taking into account the lower cost of the large chambers, a slight diminution in the illuminating power may be neglected, and we may even use cheaper coal when the illuminating power is to be increased by carburettling with benzene.

This process would have many advantages over the use of carburetted water-gas, and very possibly the future development of the German gas industry will be in this direction.—R. B. B.

*The Manufacture of Coke.* R. de Soldenhoff. *Iron and Steel Inst. Autumn Meeting*, 1894. *The Ironmonger*, **68**, 1894, 381—384.

IN this posthumous article the author describes in a general way the three forms of coking used in Great Britain, viz.: (1) the bee-hive oven; (2) the Welsh or rectangular oven; and (3) the Coppée oven. Of the total quantity of coke annually made in Great Britain, 85 per cent. is made in the first-named oven and 10 per cent. in the Welsh oven. The quantity of coal made into coke is 35,000,000 tons, yielding approximately 21,000,000 tons of coke, and leaving 14,000,000 tons to be accounted for as follows:—3,500,000 tons of ashes, representing 4,250,000 tons of original shale or earthy matter extracted from the coal before coking by the process of washing, whilst 9,750,000 tons of volatile matters, hydrocarbons and nitrogen, represent the balance. As one ton of volatile or gaseous matter

is equal to about five tons of coal in heating energy, the above quantity represents about 50,000,000 tons of coal. In the question of utilising this matter for boiler purposes, the author shows that the value of the gases is about 6,000,000*l.*, of which 2,500,000*l.* only is utilised, the remainder being wasted. The gasworks practice of extracting tar and ammonia, acted as an example to the coke manufacturers, and also to the Scotch ironmasters in their blast-furnace working, to try and save the by-products. It is about 10 years ago, that the question of saving the by-products in the coking process, was revived.

The subject was brought to the front in England by Watson Smith in his paper read before the Iron and Steel Institute at Chester, and illustrated with every kind of oven then in existence for saving by-products (this *Journal*, 1885, 451. *J. Iron and Steel Inst.* 1884, 186). However, there are at present only about 200 ovens at work in Britain, 40 of which are of the Semet-Solvay type in use by Brunner, Mond, and Co., whereas in Westphalia there are 470, and in Silesia 705. Otto's installations are the most complete form in Germany, but they are very costly on account of the condensing plant being duplicate. The principal feature is that the gases are not drawn direct from the oven, as in general practice, but from a flue situated in the top of the wall on each side of the oven. This flue, by means of two dampers situated on each end of it, may be, at will, connected or disconnected from the flue situated inside of the side wall, so that when the two dampers are in their normal positions, the oven is working as a closed vessel and the gases are drawn to the condensing plant at first. If, however, from any cause it is necessary to turn the oven into one working in the presence of the oxidising atmosphere, the dampers are lifted and the burning gases are admitted into the side walls first, under the floor afterwards, and ultimately into the main flue.

The author omits the bee-hive oven improvements, because the by-products obtained by the same are inferior in quality to those obtained by the Simon-Carvés or Coppée-Otto class of oven. It has been found by Otto that one ton of coal gives 25.44 lb. of sulphate of ammonia and 60.84 lb. of tar. Taking this as a standard the author calculates that in Great Britain there is wasted annually 4,262,500*l.* worth of these two products. Beyond this there is the heating power of the gases after the extraction of the ammonia and tar, which is put down as 1,500,000*l.* The author admits that there is to be considered the cost of plant, which in the by-product oven is about three times that of the ordinary coking plant, and also the possibility of local difficulties. As regards the quality of the coke, he believes that made by the Coppée



is not superior to that of the bee-hive.  
It suggests the utilization of the heating power of the waste gases of coke ovens for the destructive distillation of shale, and the production and extraction therefrom of sulphate of ammonia.—A. W.

## PATENTS.

*Improvements in or connected with the Manufacture of Gas.*—J. H. R. Dunsmore, Liverpool. Eng. Pat. 5647, March 16, 1893.

According to this invention, gas of a given illuminating power is first heated, then mixed with the heated vapours of tar or oil, the resulting mixture being again heated. The temperature employed is about 1,700° F.

In a typical instance the apparatus is as follows:—To each bench or oven there are two sets of apparatus, each apparatus comprising three retorts. The lowermost retort is of rectangular section and is made of iron or steel; in it the tar or oil is vaporised. The central and the uppermost retorts have circular sections, and—holding, as they do, only vapours or gases—are of fireclay.

The three retorts are connected up in the following manner:—At the back of the bench, the mouthpieces of the top and bottom retorts are connected with that of the central retort, while in front of the bench is the inlet to the upper retort and the outlet from the central retort.

Coal-gas or water-gas, &c., entering the top retort at the front end, traverses it and enters the central retort at the other end. Similarly the heated tar or oil vapours, which distil from the contents of the lower retort, enter the central retort at one end, the mixed gases travelling through that retort, and passing out at the other end into the outlet pipe.

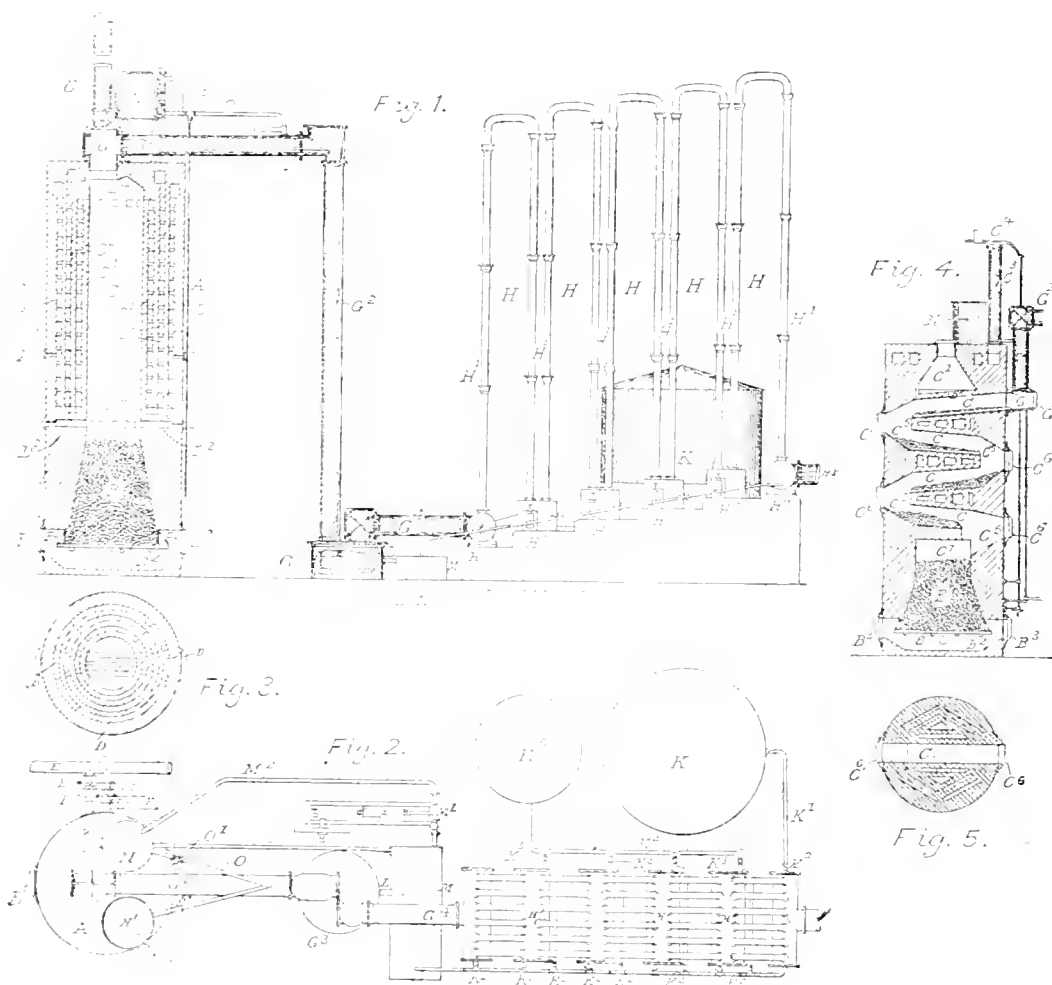
According to the drawings which accompany this specification, the retorts have similar capacities.

It is stated that gases so treated have their illuminating power raised, while at the same time their volume is increased.—E. R. B.

*Improvements in the Production of Illuminating Gas from Mineral Oils, and in Apparatus therefor, and for Producing Water-Gas.*—W. Young, Priorsford, Peebles, county Peebles. Eng. Pat. 12,355, June 23, 1893.

The inventor still further develops the system of decomposing mineral oils described in his and A. Bell's patent, Eng. Pat. 12,421 of 1892 (this Journal, 1893, 666).

In the production of water-gas and in the decomposition of hydrocarbons for the purpose of carburising water-gas or other gas, very generally the necessary heat has been obtained, for the most part, from an extraneous source; that is to say, it has not been obtained by the combustion of the elements of the hydrocarbons employed; yet most of these hydrocarbons, especially those of crude tarry oils of high densities, contain carbon in considerable excess of



APPARATUS FOR THE PRODUCTION OF ILLUMINATING GAS FROM MINERAL OILS.

that which can be rendered gaseous by destructive distillation. Moreover, this excess carbon is a source of much trouble, choking up, as it does, the passages and depositing on the walls of the apparatus and rendering the distillates thick and pitchy.

The main object of the inventor has been to overcome the difficulties enumerated, by effecting a partial combustion of this carbon, while utilising the heat so evolved for the decomposition of a further quantity of hydrocarbons and for the production of a certain quantity of water-gas. Fig. 1 is a partly sectional elevation, and Fig. 2 the plan of the author's typical apparatus, while Fig. 3 is a horizontal section of one of the decomposing chambers invented. The cycle of operations is as follows:—

At starting, hydrocarbon fluid is allowed to flow by gravitation from the tank K, so as to seal the condenser chests, H<sup>2</sup>, and the main seal box G<sup>1</sup>, and to overflow into cistern M. Hence, pump M<sup>1</sup> (Fig. 2) lifts a supply through pipe M<sup>2</sup> into the cistern N.

The combustion chamber and water-gas generator B (Fig. 1) is now charged with any convenient form of carbon, kindled, and air driven through the charge by opening valve E<sup>2</sup> (Fig. 2) and so admitting air to the ashpit B<sup>2</sup>, valve C<sup>1</sup> on the smoke-stack C<sup>3</sup> being opened, and all products of combustion escaping to it through the ports D<sup>1</sup> and the flues C<sup>1</sup>, C<sup>2</sup>, which lead to the stack C<sup>3</sup>. Valve E<sup>3</sup> is then opened and a second supply of air enters by the pipe E<sup>1</sup>, leading to the annular flue D, and so to the openings D<sup>2</sup>. The CO formed in B is lit through the port D<sup>3</sup>, and the two air supplies so adjusted that all CO is burnt to CO<sub>2</sub>.

As soon as the fuel in B and the firebrick linings of the decomposing chamber C have acquired a sufficiently high temperature, both air valves are closed, and steam is admitted under the firebars by opening F<sup>2</sup>, and the water-gas so produced is allowed to displace all air and products of combustion. Hydrocarbon liquid is now allowed to slowly flow through O and along the floor of the wide conduit G<sup>2</sup> (which is very hot) into C. Here it drops through the whole height of the chamber on to the highly heated coke, being exposed to very considerable radiant heat the whole way. The hydrocarbon is partly gasified, partly carbonised. As to the latter, some forms a hard coherent cake on the coke, while any flocculent carbon set free is caught and entangled in further falling drops of hydrocarbon.

As the temperature falls, less and less hydrocarbon is allowed to enter C, and finally it is turned off. Steam is then again admitted and sufficient water-gas so produced to drive out the hydrocarbon vapours. The steam is next turned off, C<sup>1</sup> opened, air admitted, the original working temperature restored, and a second cycle entered upon.

According to the inventor, during the carburetting, only sufficient water-gas to prevent the heavy hydrocarbon vapours from sinking through the fuel is to be employed; for, he states, "the presence of water-gas interferes with the decomposition of hydrocarbon vapour in various ways, particularly when the decomposition is effected by radiant heat."

The inventor's second modification of apparatus is to allow for the cases where very much carbonisation of the hydrocarbon occurs; while in his third modification, he employs two or more combustion and decomposing chambers in one producing arrangement. By this means he obtains a continuous process and also great facility in manipulating the ratio of water-gas to carburetted water-gas produced. The fourth modification very considerably modifies the decomposing chamber, and is designed to meet the cases of those hydrocarbons which, during decomposition, "sponge or swell up in foam," this peculiarity tending to close the spaces between the pieces of carbon in the combustion chamber, and so to impede its action. The modification in question is shown in Figs. 4 and 5, and will be seen to consist of a series of retort-like chambers C, slightly inclined and connected by the chambers C<sup>3</sup>. There are clearing doors C<sup>6</sup>, through which the deposited carbon may be raked down into the combustion chamber B from time to time. The hydrocarbon is allowed to slowly flow over the large bottom surface of the retorts in a very thin layer.

When the liquid hydrocarbons carry water or, as in the case of coal-tar, valuable benzene, this is readily separated in the condenser; for, as already explained, the flow of the hydrocarbons through the condenser chests is against that of the gas; and the gas entering G<sup>1</sup>, and even H<sup>2</sup>, is sufficiently hot to vaporise these substances, which are therefore condensed in and may be recovered from one of the chests further forward.—E. R. B.

*Improvements in Obtaining Combustible Gases, Ammonia, and Tar from Fuel, and in Apparatus therefor.* L. Mond, London. Eng. Pat. 12,410, June 24, 1893.

See under III., page 938.

*Improvements in Water-Gas Apparatus.* A. G. Glasgow, Westminster. Eng. Pat. 5588, May 1, 1894.

This invention relates to the construction of the valve necessary in those water-gas plants where the steam is conducted through the incandescent fuel alternately from the top or bottom of the generator.

Three forms of valve are minutely described and figured, and in each of them the construction is such that the valve lies entirely out of the way of the intensely-heated blast gases of the generator, while the operating mechanism is for the most part outside the apparatus. The conduits from the top and bottom of the generator are so arranged that the hot gases issuing from either of them never impinge on the opening of the other. As to the steam supply, the inventor prefers to connect the controlling mechanism of this with that of the gas valve in such a way that when the conduit from the bottom of the generator is open the top injector is automatically turned on, and when the valve is moved, shutting this conduit and therefore opening the other, it turns on the bottom injector.—E. R. B.

*Improvements in the Production of Illuminating Gas and Bye-Products from Liquid Hydrocarbons, and in Apparatus therefor.* W. Young, Peebles. Eng. Pat. 13,126, July 5, 1893.

This inventor brings forward a modified process and a new arrangement of existing coal-gas plant to enable such plant to be used for producing illuminating gas and benzene from hydrocarbon liquids in accordance with the principle of the previous invention of the author and A. Bell (see Patent 12,421 of 1892).

From suitably placed tanks regulated quantities of coal-tar and of "more refined tars and oils, such as natural petroleum and those distilled from shale at low temperatures," flow through siphon pipes to one or more of the retorts heated by a given oven.

In cases where some of the retorts are carbonising coal the temperature will be too high a one to which to expose the finer qualities of oil; a rapid current of the tar through the retort is therefore in the first instance maintained, and not until the internal temperature of the retort is reduced is the finer oil allowed to enter. "The presence in the retort of the vapour and gases from the tar has a conservative action upon the oil vapours, protecting them from over-decomposition even at the high heat employed." The gas so produced in the tar-decomposing retorts mixes in the hydraulic main with that from the coal-decomposing retorts, and any excess of distillates and condensable hydrocarbons is condensed with the coal-tar, and is thus used over again.

Near the ends and cooler parts of the retorts tar is liable to soak into the fireclay, and, by swelling during coking, is liable to crack the walls; to obviate this difficulty the inventor inserts a sheath or lining of iron or steel at these points.—E. R. B.

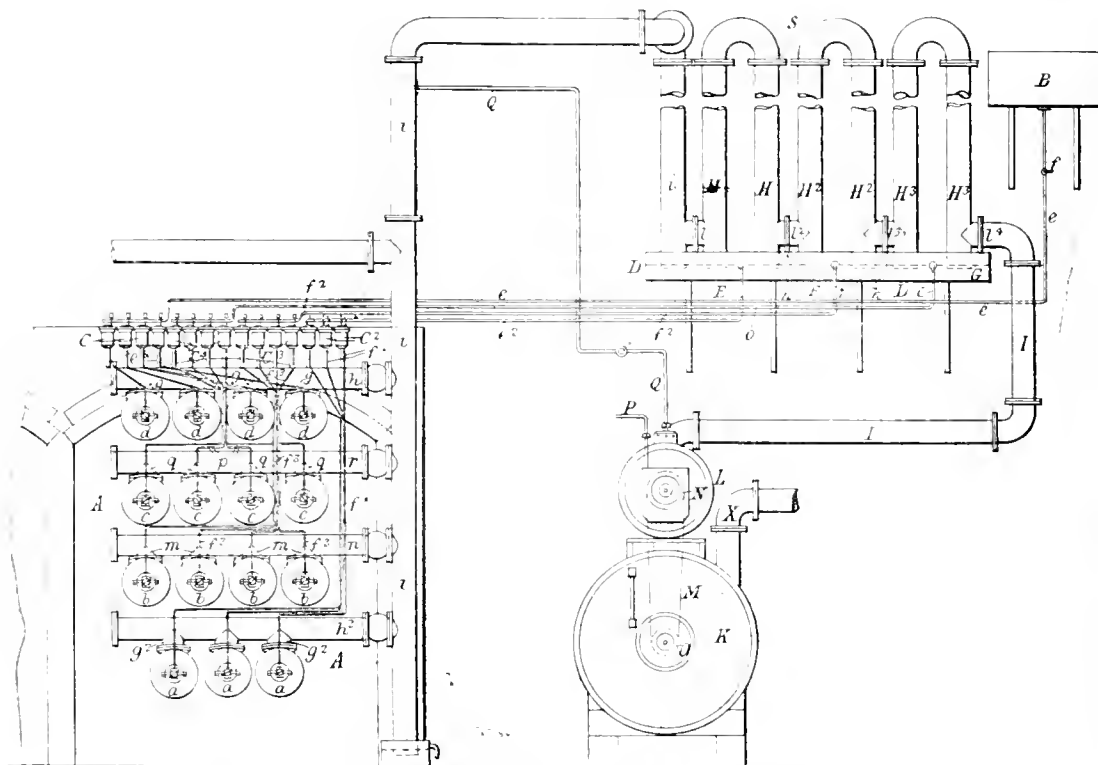
*Improvements in and Apparatus for Distilling or Cracking and Gasifying Hydrocarbon Oils and the like.* H. M. Caldwell, London, and E. Tatham and J. E. Tatham, both of Lewisham, Kent. Eng. Pat. 13,163, July 5, 1894.

The chief aim of the patentees is to accomplish the gasification of a given oil at a known suitable temperature, and to separately collect its condensable hydrocarbon by-products, so that each of these in its turn may be gasified at its proper temperature.

In the figure, A is a bench of retorts, arranged in four tiers, the retorts (*a, a, a*) of the lowest tier being maintained at the highest temperature, and those (*d, d, d*) of the

uppermost tier at the lowest temperature. The crude gas manufactured passes by *h<sup>2</sup>, n, r,* and *h* into the ascension pipe *i*, which terminates in the condenser S.

The condenser S consists of a vessel D, divided into three compartments, E, F, G, respectively. Into E dips the end of the ascension pipe *i* and the two legs of the inverted U-pipe *ll, ll*; into F dip the ends of *ll<sup>2</sup>, ll<sup>2</sup>*; into G, those of *ll<sup>3</sup>, ll<sup>3</sup>*. The connecting tubes *l, l<sup>2</sup>, l<sup>3</sup>, l<sup>4</sup>*, allow of the passage of the gas through the condenser into *l*, and thence through the meter K into X, the pipe leading to the gas-holder. The oil employed in the manufacture is contained in the tank B, and is conducted through *e* into *d, d, d*, the coolest series of retorts. As to the distillates, the



APPARATUS FOR DISTILLING OR CRACKING AND GASIFYING HYDROCARBON OILS.

heaviest of them, viz., that condensed in the descending limb of *i* and in *ll, ll*, and collected in E, is conducted through *e<sup>2</sup>* into *a, a, a*, the hottest of the retorts; the intermediate distillate, similarly collected in F, is treated in the retorts *b, b, b*; while the lightest distillate, that collected in G, flows into the still cooler retorts *c, c, c*.

C—C' are apparatus, attached to the feeding tubes, the function of which is to automatically shut off the supply of hydrocarbon to any given retort, should the outlet from such retort become choked.

Oxygen may be mixed with the products of distillation as they pass to the condenser. This gas is contained in the meter L, entering by P and leaving by Q. The oxygen meter is operated from the gas meter K by the endless chain M, which passes round the sprocket wheels N and O on the axes of the respective meters; the relative sizes of N and O are determined by the proportion of oxygen it is desired to mix with the products of distillation.—E. R. B.

*Improvements in Electric Furnaces.* F. Chaplet, Paris. Eng. Pat. 15,577, August 16, 1893.

A cavity is made on the upper side of a mass of refractory material, and fitting above this is a similar block with a

hollow on its under side, which thus forms a domed roof over the first cavity. A refractory retort, muffle, or tube projects into or across one axis of this chamber, and a pair of carbon electrodes project across an axis at right angles to the first, the ends of the electrodes passing through opposite walls of the chamber to rolling supports outside, by which the length of the arc may be regulated. Several pairs of electrodes may be used, if required, to produce higher temperatures, or for local heating. The tube contains the substance to be heated, which may be fed in at one end and run out of the other in a fused condition, into a receptacle outside, or into a well within the furnace, the tube being sloped at an angle varying with the fluidity of the fused mass. Or, the muffle may be used for cupelling or any other required purpose.—W. G. M.

*Improvements in Apparatus for Supplying Liquid Fuel to Furnaces.* J. C. Etchells, Heaton Chapel. Eng. Pat. 19,110, October 11, 1893.

For the purpose of supplying liquid fuel to furnaces in the form of a spray, provided with air-inlets around the steam-nozzle, through which a current of air enters, and, together with the steam, mixes with the fuel before reaching

the fire. The apparatus includes a nozzle and valve for the admission of steam, and at right-angles a second nozzle and valve are arranged, through which the liquid fuel enters. From the nozzle the steam enters a chamber of larger diameter, through the walls of which are a number of air-inlets, somewhat similar to those found in a Bunsen's burner. Into this chamber, a short distance nearer the furnace, the fuel enters, and meeting with the current of air and steam, is broken up into a spray, which is carried along a tube into the furnace, and striking a deflecting plate, is thrown down upon the hearth of the furnace. The quantity of fuel is regulated by a valve and hand-wheel, as is also the steam-supply. Accompanying the specification is a detail drawing of the apparatus.—S. P. E.

*Improvements in Gas Producers.* C. D. Abel, London. From the Gas Motoren Fabrik Deutz, Kolu-Deutz, Germany. Eng. Pat. 21,089, November 6, 1893.

THE inventors' producer differs from the ordinary type in that at the bottom and immediately above the grate it is enlarged, the chamber so formed being rectangular in horizontal section. This chamber has openings of its full width on opposite sides of the producer, closed by air-tight doors. To ensure further the protection of the doors from too direct heat and so to assist them in the maintenance of an air-tight closure, protective shields lined with firebrick, like the doors, are slid into the openings so as to lie about midway between the very highly heated fuel and the doors; a non-conducting layer of air therefore always existing between the shield and the door.

The structure detailed above allows of the rapid clearance of clinker and of any caked material adhering to the sides of the producer, and thus, as the inventors state, strongly slagging or caking fuel can be used for the manufacture of combustible gas with the same facility as anthracite.

A further improvement consists in the employment of several steam injectors, an arrangement which permits of a very accurate adjustment of the steam supply, according to the varying needs of the producer.—E. R. B.

*New or Improved Method of Focussing or Controlling Flame or Heat in connection with Burners, Stoves, Kilns, Melting Pots, or other Heaters provided with Combustion Chambers.* C. H. Land and H. J. Caulkins, Detroit, U.S.A. Eng. Pat. 13,106, July 6, 1894.

THIS invention relates to a method and apparatus for focussing flame and producing an intense heat (combustion being complete), in connection with burners, stoves, kilns, &c. provided with a combustion chamber, and heated with liquid or gaseous fuel. The arrangement consists in having a combustion chamber, the lower portion of which is covered by a bed with an opening in the centre. Upon this bed is placed a removable slide, also having an opening in the centre, and which further carries a removable diaphragm with as small an orifice as necessary. By means of such removable diaphragms with orifices of varying sizes the flame may be focussed. Beneath the diaphragm is placed the burner, consisting of an open pan which is fed by gravity with liquid fuel. Resting within the pan is a wire gauze screen or collar. The pan and its contents can be raised or lowered by means of a series of levers. When the surfaces above the pan are heated they radiate the heat and vaporise the fuel, which is important for obtaining the greatest efficiency; the burner being gradually lowered in proportion as the temperature rises in the combustion chamber until the maximum heat is obtained. The periphery of the opening in the movable slide forms a seating corresponding in form and size with the upper rim of the fuel pan, so that by raising the pan to the seating, the draught through the aperture at the base of the combustion chamber is completely closed, or the amount of air and flame passing through the aperture is in proportion to the height the pan or burner is raised. The air is admitted by a fine passing through the side of the burner and opening into a tubular stem that supports the burner and thence to the top of the fuel pan or point of ignition. The size of the orifice in the diaphragm used depends upon the draught

of the chimney; with a strong draught the orifice of the diaphragm should be reduced and the distance between burner and orifice proportionally decreased to obtain the proper focussing. Suitable arrangements are provided for removing any carbon that may collect upon a muffle or anything placed in the combustion chamber, also an overflow pipe from the fuel pan, and means for preventing fire outside the furnace.—S. P. E.

*A New or Improved Combustible Composition.* René Odeyn, Brussels. Eng. Pat. 11,968, July 21, 1894.

THE composition claimed by the inventor consists of 700 parts of schistous stone, crushed; 250 parts of powdered charcoal or sawdust, spent tan, or peat; and 50 parts of tar. These are well mixed together, and made into briquets.—S. P. E.

*Improvements in the Manufacture of Incombustible Burners for Liquid and Gaseous Fuel.* C. Westphal, Berlin. Eng. Pat. 11,136, July 27, 1894.

FOR the manufacture of incombustible burners for liquid and gaseous fuel, carbonate, silicate, or fluoride of calcium, clay, kaolin, tile, sand, quartz, pumice-stone, carbonate and sulphate of strontium are employed as forming the chief substance. By this invention the author claims the addition of compounds of the heavy metals, as the oxides of lead, tin, zinc, iron, manganese, &c., which is said to prevent cracking or changing in shape during the burning or drying process. For the purpose of making them porous, the inventor adds to the above mixture sulphur or some compound that volatilises when heated. The above mixture is moistened with a solution of water-glass, alum, or boric acid, and then moulded and, if necessary, burned.—S. P. E.

*Improvements in and relating to Apparatus for the Distribution and Burning of Fluid Hydrocarbons for Lighting and Heating Purposes.* J. McCownan McMurtrie, Glasgow. Eng. Pat. 16,701, September 5, 1894.

THIS apparatus is constructed for burning petroleum and other liquid hydrocarbons and water, first vaporising and then converting hydrocarbon oils and water into permanent gases, or by first vaporising the hydrocarbon or water and then superheating the vapour or steam and conducting the same to the burner. The illustration shows the general construction of the apparatus. Fig. 1 shows a side view, the lower portion being in section. Fig. 2 is a section on line  $x-x'$ , and Fig. 3 a section on line  $y'-y'$  of Fig. 2. A is the outer metal casing, which is open at the top. B is a vaporising-coil, which is connected outside with the supply-pipe  $u$  for water or hydrocarbon oil, and the inner end communicates, through a passage in the casing  $c$  and sockets  $b$  and  $d$ , with the gasifier or superheater C. This superheater communicates with the cup D of the burner by the passages  $j$  and  $i$ . An inner casing  $h$  around the coil may be employed, thus forming an annular space down which air for supporting combustion passes. The nozzle or burner is a metal cup screwed into a seating in such a way as to leave an annular chamber  $D'$ , which communicates above with the burner and below with the chamber  $k$  and supply-pipe  $n$ . The burner is connected with the stand-pipe  $m$  and pipe  $n$  by the tee-piece E, having two passages  $n'$  and  $m'$ , Fig. 2, the former leading to the chamber  $k$  and the latter to the vaporising coil. The pipes  $a$  and  $k'$  are connected to the tee-piece with coned couplings, so as to allow the burner to be inclined at any angle. The pipes  $m$  and  $n$  are connected respectively with suitable store tanks, preferably arranged one within the other, and provided with an air-pump and other necessary fittings. One tank only is required if water is obtained under pressure, in which case it is directly connected with the coil. If the apparatus is to be used for heating purposes, only a small quantity of oil is employed, and the coils B and C are allowed to get red-hot, so as to decompose the steam. Instead of water, two hydrocarbons may be employed, in which case that passing through the gasifier is converted into permanent gases, and burns with the oil at the nozzle.

The specification gives a description, accompanied by illustrations, of several modifications of the apparatus, to be employed respectively as a portable lamp, a search-light,

Fig. 1.

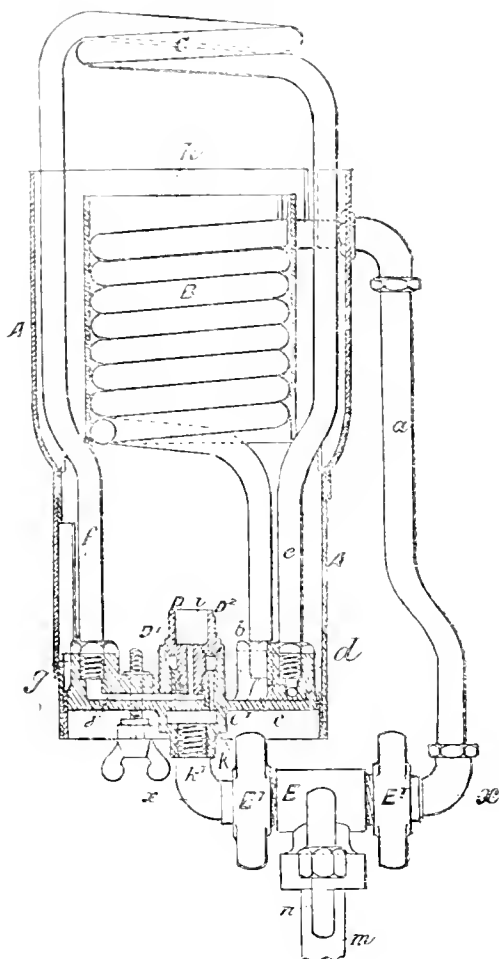


Fig. 2.

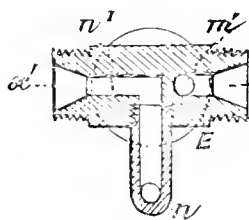
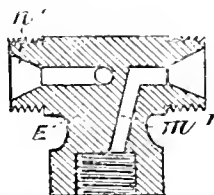


Fig. 3.



and an arrangement for a number of lamps supplied from a central reservoir.—S. P. E.

### III.—DESTRUCTIVE DISTILLATION, TAR, PRODUCTS, Etc.

*Action of Light on Phenol.* A. Bach. Monit. Scient. 42, 508—510.

This question as to whether this action is due to formation of hydrogen peroxide is answered by the author in the negative. According to his investigations it is shown that when phenol is subjected to the simultaneous action of light and moisture in an atmosphere of carbonic anhydride free from air, it assumes a brown colour much more readily than in air free from carbonic anhydride (see this Journal, 1893, 107, 112, 415 and 553).—D. B.

*The Manufacture of Coke.* R. de Soldenhoff. Iron and Steel Inst. Autumn Meeting, 1894. The Ironmonger 68, 1894, 381—384.

See under II., page 933.

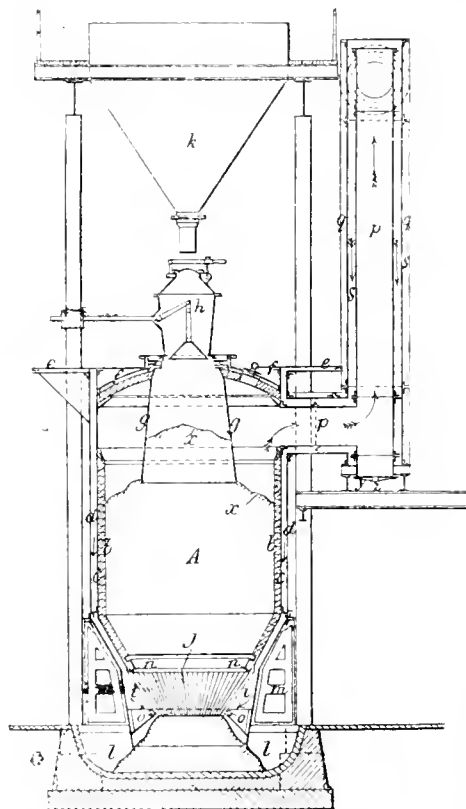
### PATENTS

*Improvements in Obtaining Combustible Gases, Ammonia, and Tar from Fuel, and in Apparatus therefor.* L. Mond, London. Eng. Pat. 12,440, June 24, 1893.

In this invention a suitable mixture of air and steam is driven by a fan or blower through a superheater of special design until it attains a temperature of at least 150°, with a pressure usually sufficient to support 6 to 9 ins. of water. It then enters a producer of particular construction, to react with the fuel contained therein. The inventor claims both process and apparatus.

With regard to the process, he states: "I believe that I have been the first to discover that, by burning fuel with a

Fig. 1.



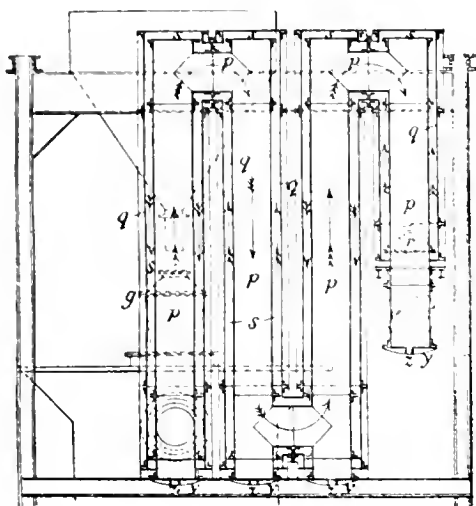
mixture of air and steam in proper proportions raised to a temperature of not less than 150° C., the quantity of ammonia obtained from the fuel is largely increased."

With regard to the apparatus, the inventor specially claims the superheater, "constructed of a series of pipes arranged vertically in zig-zag form, through which the hot gases from the producer pass, and which are surrounded by wider pipes, thus forming an annular space through which the mixture of air and steam is driven in a direction opposite to the producer gas, and the combination of this superheater with a gas-producer, substantially as described."

Fig. 1 is a median vertical section of the producer and of one unit of the superheater. Fig. 2 is a median vertical section of the superheater in a plane at right angles to the foregoing.

The air and steam mixture enters at *r* (Fig. 2), and traverses *s*, *s*, *s*, *s*, and so reaches (Fig. 1) the space

Fig. 2.



between *c* and *d*, the inner and outer casings of the producer, and passing between the bars *i* of the grate *j*, reaches the fuel in the body *A* of the producer.

The producer-gas manufactured leaves by *p*, *p*. The outer wrought-iron casing *d* of the producer is closed at the bottom by the water-lute *l*, and the inner wrought-iron casing *c*, which is lined with firebrick, has attached to it the inverted conical grate with wedge-shaped bars *j* open below, though closed during work by the heap of debris, as represented in Fig. 1. The cast-iron cylindrical apparatus *g* allows of feeding the producer from the charging chamber *A* without disturbance, only a minimum, therefore, of fuel dust entering the gas pipes. The lines *x x* show the upper limit of the fuel. The inventor admits that circular gas-producers, with surrounding annular spaces for heating the air, have been previously described, as have also various devices for heating this air by the waste heat of the producer; but, he states, "none of these are sufficient to attain the rise of temperature required." As to the process, he points out that hitherto, for obtaining ammonia from fuel with gas-producers, mixtures of superheated steam with cold air have been proposed—a method which would involve a greater expenditure "for extra fuel than would be compensated for by the increase in the ammonia obtained."

Previous patents of the inventor, relating to this subject, are 3923 of 1883 and 8973 of 1885; this Journal, 1884, 236, and 1886, 235.—E. R. B.

**A Paste or Mixture for Cleansing Various Materials and Articles.** M. Baerlein and C. Dreyfus, Manchester. Eng. Pat. 19,733, October 20, 1893.

THE paste is made by taking china clay, pipeclay, fuller's earth, French chalk, or other similar materials, either

separately or mixed in various proportions, and incorporating them with "benzol, toluol, xylol," solvent naphtha, "patent naphtha," petroleum naphtha, or carbontetrachloride, also either separately or mixed in various proportions. The paste or cream is intended for removing grease and other spots from cloth, wearing apparel, gloves, lace, pictures or prints, and metals. It is to be applied in a thin layer, left to dry, and then brushed or rubbed off. The inventors claim the paste, and also the method of using it. J. A.

## IV.—COLOURING MATTERS AND DYES.

*The Action of Light on Textile Dyes.* Remarks on J. J. Hummel's paper. M. Kischelt. *Färber Zeit.* 5, 1894, 165—168, 181—183. (See this Journal, 1894, 801—807.)

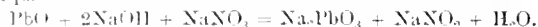
THESE remarks are based upon an extensive series of experiments carried out since 1891 at the Elberfeld colour works. In the year named all the colouring matters then known were dyed on cotton and wool and exposed simultaneously. In the autumn of 1892 the same series dyed on silk was similarly exposed, and since then the experiments have been followed up by a repetition with smaller groups of the dyes. Altogether the daily experiments fully confirmed the results obtained from the larger series. All the colouring matters were dyed to the same depth of shade without any consideration as to whether to this end 2 per cent. of the one and 1 per cent. of another dye were required. Hummel's method consists in dyeing 2 per cent. shades of each colouring matter upon cotton, wool, or silk, without consideration of the strength of each individual dye, in the author's opinion necessarily involves the error of finding too high a degree of fastness for the strong dyes, whilst the results for the weak dyes must, he thinks, be too low. Therefore they do not represent the fastness to light of the dyes, but rather are a product of the intensity of a dye and of its fastness to light. Even supposing, however, that all dyes be of the same intensity when dyed upon wool in the skein, the fact that in these tests the colours are dyed upon cloth may, the author thinks, lead to another serious error. The various dyes known exhibit great differences with regard to their capability of dyeing cloth evenly all through, so that of two dyes the one may be distributed with perfect evenness through the cloth, while the other will be almost entirely fixed on the surface. Under such circumstances an amount of fastness may be attributed to a dye which it does not actually possess. Hummel's plan of dividing each sample into six parts, one of which is kept for reference, whilst of the five others which are exposed, one is withdrawn after the expiration of each one-fifth of the whole time of exposure, is very useful. But mention should have been made, it is considered, of the remarkable difference thus observable between various dyes or classes of dyes from stage to stage during exposure. In the first group the dye disappears uniformly from beginning to end. In the second group a decided alteration of the shade becomes apparent after a few days' exposure, but then this slightly altered shade remains unaffected for a very considerable time. Such behaviour is very clearly exhibited in the case of indigo, light tints of which at first rapidly assume a somewhat greenish tone, which then remains permanent for a very long time. In the third group a reverse action takes place, i.e. the original tint remains for weeks unaffected, but as soon as the alteration sets in, a few days' further exposure will suffice for the destruction of the shade. This shows that to avoid errors the exposure must not be too short, and should not be interrupted until all the specimens under experiment begin to show decided signs of deterioration. Hummel exposed all his patterns to the same quantity of chemically active light, which was measured by means of "types." For the absolute necessity of gauging the length of exposure in this manner, instead of by time, Hummel has furnished interesting proof. According to the amount of deterioration the exposed patterns

arranged in groups, but within the groups the dyes are arranged by Hummel in the order of their fastness. Thus, as is to be seen, done in the experiments carried on at the Mulhouse colour works. Their first group comprises the fast dyes, whilst Hummel's first group contains the fugitive ones. In Hummel's first group appear the fast dyes, Eosine, Erythrosine, Phloxine, Bengal, Rose bengal, Cyanosine, Cyclamine, &c., which are not more stable or fugitive. Not more stable are safranines, Neutral red, Neutral violet, Neutral blue, and Roxamine. The rosindulines may be divided into two groups, the yellow brands ranging somewhere between the first and second group, whilst the blue brands are considerably faster. Titan, scarlet R and Patent atlas red, also belong to the first group of very fugitive dyes. Larger differences exist in the second group. The triphenylmethane colouring matters follow immediately in fastness after the first group, and are followed by the somewhat faster rhodamines. Between these two appears a considerable number of the azo dyes, especially some of the substantive dyes, such as the stilbene dyes and Congo red. Brilliant Congo stands rather higher. Ponceau SS extra is only little better than magenta. Fast red BF, Thionine, Cloth scarlet G and R, Coccine B (on silk), Ponceau acide, the Xylidine and Cumidine scarlets, the rhodamines, Pyrouine, Double brilliant scarlet G, Bordeaux COV, Archil substitute and Cloth red 3 B extra follow next. Hummel placed the Xylidine and Cumidine scarlets, Bordeaux COV, and Ponceau G in the third group, but they are much more sensitive to light than Cloth red 3 G extra, Bordeaux BX, and others. On the other hand, Cloth red 3 G extra, Fast red A, Bordeaux BX, and Fast red B are faster than they appear according to Hummel. Of the substantive dyes, Benzopurpurin 1 B, 4 B, and Brilliant Congo come next or nearly so to Fast red BF, the rhodamines next to Bordeaux COV. The rhodamines are faster on silk than on wool. The third group, containing moderately fast dyes, contains a great many azo dyes. But part of those mentioned by Hummel really belong, according to the author, to the second group. The Rosindulines 2 B and 3 B, and Azocarmine stand at the beginning of the third group, then follow Fast red A, E and D, Scarlet R and Ponceau GG are of about the same fastness as the fast reds. Next follow Palatine red, Cloth red 3 G extra, Azo-eosine, Croceine 3 BX, crystal Ponceau and finally Carmoisine B (Fast red C). Of the natural dyestuffs, cochineal on alumina and tin mordant stand between this and the next group. From this group should be removed, according to the author, the following dyes placed in it by Hummel: the Ponceaux, Cloth scarlet (into group II.), Buffalo ruby (into group II.), and Cloth red G extra (into group IV.). Also Carmoisine and cochineal were better assigned to the fourth group. Azocarmine, which Hummel judged too favourably, as the author thinks, should be removed to this group, whilst Cloth red 3 G extra, fast red A, B, and Bordeaux BX, which Hummel placed in the second group, the author places in the third. Of the substantive dyes, the deltapurpurins stand with the fast reds. The last members of the third group, Carmoisine B and cochineal, are perhaps better placed in the beginning of the fourth group, followed by Brilliant double scarlet 3 R, Brilliant Ponceau 4 R, Ponceau 5 R and 6 R, Biebrich scarlet 3 B, 7 B, Palatine scarlet, Cloth red G extra, and alizarin on alumina mordant, according to the author. All these show about the same degree of fastness. Ponceau S, the Azo-magentas, and the Chromotropes then immediately follow. Compared with Hummel's results the following differences appear, as the author thinks, in this group: Ponceau 2 G and Wool scarlet belong to Fast red; Coccine B (on silk) is about equal to the Xylidine scarlets, Cloth red GB and Archiline BB undoubtedly belong to the next group. On the other hand, Diamine fast red, Geranine G, and Anthracene red possess the same degree of fastness as Croceine scarlet, and therefore belong to this (the fourth) group. Amongst the very fast dyes, forming the fifth group, Hummel only classes alizarin, madder, Cochineal scarlet, and Chromotrope 2 R and 2 B. The pair of last-named dyes are no better than any of the other chromotrope dyes, and therefore the author considers they should have been placed along with Azo-magenta. Alizarin, although

the fastest dye on cotton, has, the author thinks, been overrated regarding its fastness on wool, where it proves inferior to several of the cloth red brand. The same may be said of madder and cochineal, which are not fast enough to appear in this class. This first class is exclusively composed of artificial dyes: Cloth red BX, Bordeaux G, Orselline BB, Cloth red G and B, with or without chrome mordant. The two last-named dyes, says the author, are not only the fastest of red dyes, but of dyestuffs in general. All the above-named dyes were dyed on wool, in some cases on silk. When dyed on cotton the results of the experiments come out very differently.—C. O. W.

#### *A Method of Preparing Lead Peroxide for the Aniline Industry.* W. Minor. *Der Techniker*, 1894, 16, 80.

To prepare lead peroxide free from hypochlorite, and answering to the requirements of the aniline manufacture, add to molten caustic soda, at the highest possible temperature, the required amount of lead monoxide with continual stirring, and then the amount of sodium nitrate necessary for oxidation. The reaction takes place according to the equation—



By extraction of the mass with hot water the lead peroxide is obtained—



It is purified by washing with water, and dried. By this method an almost chemically pure product may be prepared.—R. B. B.

#### *Colouring and other Principles contained in Mang-Koudou.* A. G. Perkin and J. J. Hummel. *J. Chem. Soc.* 65 and 66, 1894, 851—863.

THE dyeing material named *oungkoudou*, *jong-koutong*, &c., is the root-bark of *Morinda umbellata*. It occurs in Eastern commerce as small, reddish-brown, irregular rolls of bark, much wrinkled in appearance. Its cost is about 6d. per pound. It is largely used in Java for producing the fast reds in the native calico prints known as "baticks."

The material does not appear to be considered as of any special importance by the Hindoo dyers. The shrub from which the root-bark is obtained is met with in Ceylon and the hilly regions of Eastern, Southern and South-western India, as well as in the Malay Peninsula and Java.

*Literature.*—Philosophy of Permanent Colours (1813), Bancroft. Bulletin de la Soc. Ind. de Mulhouse (1832), Schwartz and Koechlin. L'Art de la Teinture des Laines (1849), Gonfreville. Monograph on the Dyestuffs and Tanning Matters of India, &c. (1878), T. Wardle. Watts' Dictionary of the Economic Products of India (1891), J. Murray.

*Summary of Results.*—In the general character of its constituents, mang-koudou more closely resembles madder than chay-root, although like the latter it contains but a single colouring matter. There are present, though only in small quantity, numerous yellow, crystalline substances, totally different from those found in chay-root, and, judging from their behaviour, more nearly allied to those isolated by Schunck from madder. Eleven distinct substances have been isolated.

*Morindin*, the glucoside of morindone, identical with that obtained by Thorpe, Greenall, and Smith from *Morinda citrifolia*. Substances of the formula—

$\text{C}_{10}\text{H}_{11}\text{O}_6$ ;  $\text{C}_{16}\text{H}_{10}\text{O}_5(\text{A})$ ;  $\text{C}_{16}\text{H}_{12}\text{O}_5$ ;  $\text{C}_{15}\text{H}_{10}\text{O}_4$ ;  $\text{C}_{16}\text{H}_{10}\text{O}_3(\text{B})$ ; also a substance crystallising in orange-red needles, in very small quantity. These six substances form the bulk of those present in the root. Besides these there are also present, a colourless crystalline wax of the formula  $\text{C}_{18}\text{H}_{32}\text{O}$ ; a small quantity of free morindone, Rubichloric acid and a considerable amount of a free acid (undetermined). No cane-sugar is present, which is a feature distinguishing this root from both chay-root and madder.

*Dyeing Properties.*—Gonfreville and Schwartz and Koechlin have written most on this subject. The authors found that wool and silk when boiled with a decoction of



mang-koudu with the addition of a little acetic acid, acquire rich orange and yellow colours due to *morinda*, but these are of little value, as they are at once reddened by alkalis. Applied in its ordinary condition on mordanted cotton, no useful result can be obtained with mang-koudu. The ground mang-koudu must first be washed by steeping it in ten times its weight of cold water for 2–3 hours. It is then collected and the treatment repeated two or three times. Its dyeing properties are then developed. Like madder and chay-root, however, it loses about 50 per cent. of its weight, there being removed saline matter, free acid, chlorogenin, the so-called pectic matter, and a large quantity of what are probably sugary or gummy matters.

After the foregoing treatment mang-koudu may be applied in silk, wool, and cotton dyeing, exactly in the same way as madder, it being merely desirable to add to the dye-bath 1–1.5 per cent. of sodium carbonate or chalk, in order to obtain darker or fuller colours. On stripe-printed calico containing the usual aluminium and iron mordants, it gives colours fast to soap and generally similar in appearance to those obtained by the use of madder, though somewhat inferior. Experiments are now in progress as to resistance to the action of light. It may be said that the dyeing properties of mang-koudu are practically identical with those of morinda-root, with which it is closely related both botanically and chemically.

—W. S.

#### A New Process of Indigo Preparation. *Färber Zeit.* 5, 294.

THE fermentation in the steeping vats, to which is due the formation of the blue colour, ceases, according to Schrottky, after 10–12 hours, and is then replaced by lactic acid fermentation and putrefying processes. To obviate this, he would add to the vats a certain proportion of nitrates to nourish the bacteria of the colour fermentation, and carbolic acid to prevent putrefaction. Schulte, the inventor of the new process (patented in India), declares, however, that fermentation of any kind is unnecessary, and avoids it by steeping the plants in water at 50–55° C. instead of at the ordinary temperature. He assumes that the bacteria of the ordinary process are really injurious, and that during fermentation foreign substances, such as albumin and resin, are extracted from the plants, and depreciate the quality of the product. Unfortunately he does not state whether the deposit in the heating vats is merely washed, or boiled with water before filtration.

Indigo prepared by the new process has already come into the market, and appears to be superior to ordinary Bengal indigo, being softer, lighter, and less crumbly. A medium Bengal indigo contains 54 per cent. of indigotin, Schulte's 73.8 per cent., finest Bengal (old process) 77 per cent., and finest Java 88 per cent. As the two first were prepared from plants of exactly similar growth, it would appear that the new process offers a considerable advantage.

—R. B. B.

#### Logwood Extract. E. Donath. *Chem. Zeit.* 18, 1894, 277.

See under XXIII., page 982.

#### PATENTS.

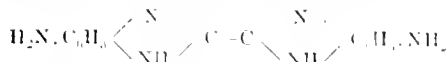
##### Improvements relating to the Manufacture of Colouring Matters. R. Vidal, Paris, France. Eng. Pat. 19,880, July 1, 1893. (Under International Convention.)

THE colouring matters referred to, which dye unmordanted fibres, are obtained by the action of sulphur on *o*- or *p*-dihydroxybenzenes in presence of ammonia or fatty amines at temperatures from 130°–200°. The following are the proportions given:—10 kilos. of quinone, 5 kilos. of sulphur, 4 kilos. of ammonium hydrochloride, and 6 kilos. of caustic soda are heated for five hours in a closed vessel to 160°–210°. The melt, when cold, is powdered and

used directly for dyeing purposes. The aqueous solution of the crude melt is precipitated almost completely by sulphuric acid with evolution of sulphuretted hydrogen. The precipitate so obtained is very soluble in alkaline sulphides, and this solution dyes vegetable fibre bluish grey, dark blue, and black. The acid filtrate, after neutralisation with caustic soda and oxidation with air or other oxidising agents, dyes wool bluish violet from a neutral and orchil red from an acid bath. The quinone may be replaced by naphthoquinone and pyrocatechol, and the ammonia by mono- or dimethylamine. The colouring matters obtained may be used in the crude state, but if purified by the sulphuric acid treatment they must, in order to render them suitable for dyeing, be mixed with 2–3 times their weight of sodium sulphide. —T. A. L.

##### Manufacture of Diamido-dibenzimidazols and Colouring Matters therefrom. O. Murray, London. From "The Farbwerke vormals Meister, Lucius, and Brüning," Höchst-on-the-Maine, Germany. Eng. Pat. 15,698, August 18, 1893.

THE compounds referred to in this specification are diamido derivatives of anhydroxanilide and its homologues (Hübner, *Annalen*, 209, 370). The lowest term of the series has the following constitution:



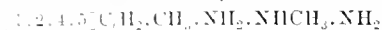
and is obtained by reducing tetra-nitro-oxanilide with tin and dilute hydrochloric acid, the proportions employed being 4 kilos. of tetranitro-oxanilide, 14 kilos. of tin, and 50 kilos. of 25 per cent. hydrochloric acid. This mixture is diluted with two or three times its bulk of water, and boiled until the whole of the tetranitro-oxanilide has dissolved, yielding a deep yellow solution. After having been freed from tin by precipitation with sulphuretted hydrogen, the filtrate is evaporated, when the easily soluble hydrochloride separates out. By dissolving it in water and adding sodium sulphate the sulphate of the base is precipitated. If the nitro compound be reduced in presence of alcohol, the sulphate may be precipitated directly by adding sulphuric acid. The base forms yellowish crystalline flakes sparingly soluble in hot water and in alcohol; it is somewhat more soluble in hot amyl alcohol, giving a solution having a green fluorescence, and is insoluble in ether, benzene, and chloroform. It separates from amyl alcohol in brilliant needles melting above 300° C. In a similar manner a diamido derivative is obtained from tetranitro-oxal-*o*-toluide. This compound is produced by the action of hot fuming nitric acid or of nitric and sulphuric acid on oxal-*o*-toluide. It is insoluble in most of the ordinary solvents, but is very sparingly soluble in hot glacial acetic acid, and melts above 300° C. In order to reduce it, 4.5 kilos. of tetranitro-oxal-*o*-toluide, 8 kilos. of tin, and 45 kilos. of 25 per cent. hydrochloric acid are heated for some time on the water-bath. After having been freed from tin the base is precipitated from the solution by sodium carbonate. The base forms a gelatinous precipitate which becomes crystalline on heating, and is sparingly soluble in water, alcohol, and amyl alcohol. The melting-point is above 300° C. The sulphate, which is rather more soluble in water, crystallises in golden needles. By the action of nitrous acid it forms a tetrazo compound which yields azo colouring matters on combination with anilines, phenols, and their derivatives. For instance, 13.2 kilos. of the diamido-dibenzimidazol are diazotised with 27 kilos. of sodium nitrite in the usual manner and run into a solution containing 25 kilos. of sodium naphthionate and 3.3 kilos. of soda. After stirring for 12 hours the formation of the colouring matter is complete. It is separated and purified in the usual manner, and dyes unmordanted cotton.

—T. A. L.



*F. M. ... and Production of New Dyes of the ...* B. Willecox, London. From "The ... Anilin and Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 18,374, September 30, 1893.

The dyestuffs referred to are obtained by the interaction of phenanthraquinone and an aromatic *o*-diamine, of which one of the amido groups is substituted by methyl, ethyl, benzyl, phenyl, t-yl, or the like. The diamine may contain other amido groups, and it has been found that such a group in the position meta to the alkylated nitrogen confers the most valuable properties on the condensation product, both as regards fastness and also brightness and purity of shades. About 15 kilos. of methyl-triamido-toluene—



20 kilos. of phenanthraquinone, and 60 kilos. of glacial acetic acid are heated together on the water-bath until no increase in the intensity of the colour is observed. About 10 kilos. of 30 per cent. hydrochloric acid are then added, and the melt is poured into 20 litres of hot water. After cooling and filtering, the dyestuff is precipitated with zinc chloride, and it is purified by repeating this treatment. It is, of course, not necessary to use separated methyl-triamido-toluene, as the solution obtained by reducing the azo-dye from a diazobenzene salt and methyl-tolylene-diamine,  $(\text{C}_6\text{H}_4\text{N}_2\text{C}_6\text{H}_4\text{NHCH}_3\text{NH}_2)_2$ , with zinc dust in acetic acid solution may also be employed. The solution thus obtained, which is kept as concentrated as possible, is poured into a mixture of glacial acetic acid and phenanthraquinone, and worked up as before. The colouring matter obtained dyes a red fast to washing. The following method is given for the preparation of a yellow dyestuff. About 17 kilos. of nitro-methyl-*p*-toluidine,  $[1,2,5]\text{H}_3\text{NO}_2\text{NHCH}_3\text{CH}_3$ , are mixed with about 170 kilos. of 30 per cent. acetic acid, and reduced with the requisite amount of zinc dust. The solution obtained is poured into hot glacial acetic acid containing 20 kilos. of phenanthraquinone. Ten kilos. of hydrochloric acid are then added, the mixture is heated for 15 minutes on the water-bath, and is allowed to cool, when the dyestuff is filtered off. It is purified by dissolving in hot water, filtering, and precipitating with hydrochloric acid. The condensation product of phenanthraquinone and amido-diphenyl-m-phenylene diamine gives a blue. About 7 kilos. of the latter base having the formula  $[1,2,4]\text{C}_6\text{H}_3\text{NH}_2\text{NHCH}_3$  are dissolved in 42 kilos. of glacial acetic acid at the ordinary temperature, 5 kilos. of phenanthraquinone are then added, and the whole is heated on the water-bath until a complete solution is obtained. The melt, which has an intense blue colour, is then poured into 20 times its weight of water, and caustic soda is added till nearly neutral, to precipitate the dye. After filtering and well washing with water, the dyestuff, which forms a resinous mass, is dried at about 70° C. It is readily soluble in alcohol and in acetone, but less soluble in water. Hydrochloric acid added to the aqueous solution precipitates the hydrochloride.—T. A. L.

*Improvements relating to the Manufacture of Dihydroxynaphthalene-sulphonic Acids and of corresponding Azo Dyes.* J. Y. Johnson, London. From "The Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 18,924, October 9, 1893.

THE sulphonic acids described are obtained by fusing with caustic soda under partial vacuum conditions, the 1,1'-amidonaphthol sulphonic acid of Eng. Pat. 9676 of 1890 (this Journal, 1891, 538) and the 1,1'-amidonaphthol disulphonic acid of Ger. Pat. 62,289. In a similar manner the azo dyestuffs corresponding to these dihydroxynaphthalene sulphonic acids are produced by heating the azo-dyestuffs from the above-mentioned amidonaphthol sulphonic acids with caustic alkalis. For the preparation of 1,1'-dihydroxynaphthalene sulphonic acid, about 30 kilos. of 1,1'-amidonaphthol sulphonic acid are heated with 20 kilos. of caustic soda in 200 litres of water in an autoclave for about 36 hours to 230° C. The melt is then made acid with hydrochloric acid and potassium chloride is added to precipitate the dihydroxynaphthalene potassium sulphonate. This salt appears to be identical with that described in Ger. Pat.

67,829. The same method can be applied to the 1,1'-amidonaphthol disulphonic acid, the same quantities being taken, and the end of the operation is determined as follows:—A sample of the melt is acidified with acetic acid and after adding sodium acetate a small quantity of diazobenzene chloride solution is run in. The dye thus formed is poured on to filter paper and spotted with soda solution. If it turns yellow, characteristic of the dihydroxynaphthalene disulphonic acid, the operation is complete, and the melt is worked up by acidifying it with hydrochloric acid and concentrating on the water-bath until a crystalline paste is obtained. After filter-pressing, the product is crystallised from 90 per cent. alcohol, from which it separates in needles. It appears to be identical with 1,1'-dihydroxynaphthalene disulphonic acid S. The strength of the caustic soda in the above examples may be increased up to 10 per cent., or a weaker solution may be employed, and similarly the temperature may be increased to 250° C., but decomposition takes place at 270° C., even with dilute lye. Regarding the production of azo derivatives of the 1,1'-dihydroxynaphthalene sulphonic acid according to this invention, the following process is given. Diazobenzene sulphonic acid from 20 kilos. of crystallised sodium sulphanilate, 30 kilos. of 30 per cent. hydrochloric acid, and 7 kilos. of sodium nitrite, is stirred into 2,400 litres of water containing 24 kilos. of 1,1'-amido-naphthol sulphonic acid. When the combination is complete the solution is raised to the boil and one-fifth the volume of brine added, when the dyestuff separates out on cooling in a crystalline form. After filter-pressing and drying it is mixed with six times its weight of 35 per cent. caustic soda lye, and heated to 120–130° C. in an open vessel or an autoclave. The conversion is complete when a test dissolves with a yellowish-red and not a bluish-red colour in water, as at the commencement of the operation. The melt is then poured into an excess of dilute sulphuric acid containing about 25 per cent. of  $\text{H}_2\text{SO}_4$ , the solution is boiled and the dyestuff precipitated with salt, after which it is filter-pressed and dried. A similar method is employed for obtaining azo dyes derived from 1,1'-dihydroxynaphthalene disulphonic acid by heating the dyestuff, obtained from a diazo salt and 1,1'-amidonaphthol disulphonic acid, with caustic soda lye as above.—T. A. L.

*The Manufacture or Production of Colouring Matters, and New Materials therefor.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 19,623, October 18, 1893.

THE present specification, which is an extension of Eng. Pat. 8898 of 1893, and of Eng. Pat. 16,780 of 1893 (this Journal, 1894, 511 and 801), describes the formation of so-called mixed 1,3-dialkyl-naphthylene diamines and their sulphonic acids, and also the production of colouring matters therefrom. By the term mixed is understood a dialkylated naphthylene diamine containing a fatty alkyl group in the one, and an aromatic alkyl group in the second amido group. The dyestuffs are obtained by condensing the *m*-dialkylated naphthylene diamines with the nitroso compounds of secondary or tertiary aromatic amines or with substances which act similarly, or else by oxidising the above-mentioned diamines with certain alkylated *p*-diamines. If necessary, the products obtained are rendered soluble by sulphonation. The production of one of the diamines referred to is described in the following example:—A mixture of 32 kilos. of 1,3,1'-naphthylamine disulphonic acid, 25 kilos. of sodium ethyl sulphate, 4.5 kilos. of soda, and 100 litres of water is heated in an autoclave for 6–8 hours at 150–170° C. The melt, after cooling, is dissolved in hot water, neutralised with sodium carbonate, filtered, and acidified with hydrochloric acid, a small quantity of salt being added. On cooling, the acid sodium salt of 1,3,1'-ethylnaphthylamine disulphonic acid separates out in colourless needles. To obtain the dialkylated naphthylene diamine sulphonic acid, 30 kilos. of the acid so formed, 90 kilos. of aniline, and 30 kilos. of aniline hydrochloride are heated for 5–6 hours at 160–170° C. Sulphur dioxide is given off during the operation, and, when cool, the melt is poured into 90 kilos. of dilute muriatic acid,

when 1,3,4'-ethylphenylnaphthylene diamine sulphonic acid separates out, and can be purified by boiling with alcohol. The following example illustrates the production of a colouring matter from this compound:—A mixture of 6.8 kilos. of 1,3,4'-ethylphenylnaphthylene diamine sulphonic acid, 9.6 kilos. of nitroso-ethylbenzyl aniline sulphonic acid, 2.7 kilos. of sodium acetate, and 20 kilos. of alcohol is heated until the nitroso compound is no longer to be detected. The melt is then poured into water, and the colouring matter salted out. A colouring matter dyeing a somewhat similar shade is obtained by substituting for the nitroso compound 12 kilos. of phenyl-azo-phenylnaphthylamine sulphonic acid (1.4) and using 40 kilos. of glacial acetic acid in place of the alcohol, and warming the mixture for four hours on the water-bath. The melt having been poured into hot water, the colouring matter is salted out. A dyestuff which gives clear bluish-red shades on wool, faster to light than similar colouring matters, is produced by heating together 8.36 kilos. of 1,3,4'-di-*p*-tolynaphthylene diamine sulphonic acid, 5.46 kilos. of azobenzene, 1.36 kilos. of sodium acetate, and 30 kilos. of glacial acetic acid at 80–110° C. until the liquid becomes red and no further increase of colour takes place. The product, which is a monosulphonic acid, is separated by pouring into water, and can be crystallised from alcohol. To convert it into a soluble dyestuff, 1 kilo. is mixed with 3–5 kilos. of fuming sulphuric acid containing 25 per cent. of anhydride, and heated at 60°–100° C. until a sample is soluble in hot water. The melt is then poured into ice water, and the sulphonic acid which separates is converted into its ammonium salt. As already mentioned in Eng. Pat. 11,892 of 1893 (this Journal, 1894, 631), certain differences are produced in the shades of these dyestuffs also, according to the solvent or diluent used in the condensation, or if the reaction be carried on at temperatures above 100° C.—T. A. L.

*Improvements in the Manufacture and Production of Mordant-dyeing Colouring Matters.* J. Y. Johnson, London. From "The Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 19,962, October 23, 1893.

THESE colouring matters are thiazine derivatives obtained from the 1,2,3', 1,2,1', and 1,2,2'-dihydroxy-naphthalene sulphonic acids or the corresponding amido-naphthol sulphonic acids by oxidising these compounds together with *p*-diamine thiosulphonic acids, and finally converting the thionated indophenol or indamine into the thionine dye either by heating with chloride of zinc, with sodium thiosulphate in dilute acetic acid, or with concentrated sulphuric acid. About 25 kilos. of amido-dimethyl-aniline thiosulphonic acid are dissolved in 600 litres of water, together with sufficient dilute ammonia to neutralise the acid, and the solution is diluted with 1,800 litres of water and 200 kilos. of 30 per cent. acetic acid. A saturated solution of 24 kilos. of 1,2,3'-dihydroxy-naphthalene sulphonic acid is added, and with constant agitation a concentrated solution of 15 kilos. of potassium bichromate, the temperature being kept at 12°–15° C. After standing some time, salt, together with a little sodium acetate, is added to precipitate the indophenol thiosulphonate. To convert this product into the colouring matter, 10 kilos. of it are boiled with 100 kilos. of a 50 per cent. zinc chloride solution. The whole is gradually evaporated until a homogeneous green mass is obtained which on pouring into water deposits the colouring matter as a violet flocculent precipitate. The dihydroxy-naphthalene sulphonic acid or the corresponding amido-naphthol sulphonic acid may be replaced by a nitroso-naphthol sulphonic acid for which the quantities are as follows:—About 12.5 kilos. of amido-dimethyl-aniline thiosulphonic acid and 14 kilos. of 1,2,3'-nitroso-naphthol sulphonic acid (the nitroso-derivative of Schäffer's acid), are dissolved in 400 litres of water, together with 9 kilos. of 20 per cent. ammonia. The solution is then acidified with 80 kilos. of 30 per cent. acetic acid, and boiled until the yellowish colour of the solution has been replaced by a dirty violet coloration. This may then be precipitated

with salt and sodium acetate as above, or the solution directly may be heated with 25 kilos. of sodium thiosulphate in 60 litres of water. After a short time the colouring matters separate out in bronzy crystals, and after filtering off is dried or used in the form of paste.—T. A. L.

*The Manufacture or Production of New Colouring Matters.* H. E. Newton, London. From "The Farbendabrike Verwalt. F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 20,579, October 31, 1894.

THE 1,1',4,4'-amidonaphtholdisulphonic acid obtained from 1,3,4'-naphthalene trisulphonic acid by nitrating, reducing, and subsequently fusing with alkalis, combines readily with benzidine, its analogues and homologues, to form symmetrical or mixed tetra- colouring matters. These colouring matters dye uncoloured cotton in shades from greenish-blue or blue to violet and brown, and can be further diazotised and combined on the fibre, giving shades very fast to washing and fading.—T. A. L.

*The Manufacture of a New Amidonaphtholdisulphonic Acid and Colouring Matters therefrom.* H. E. Newton, London. From "The Farbendabrike Verwalt. F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 20,580, October 31, 1893.

ACCORDING to the researches of Armstrong and Wynne (Proc. Chem. Soc. 1890, 1293), when 2,4'-naphthylamine sulphonic acid is treated at a low temperature with fuming sulphuric acid it is converted into two isomeric  $\beta$ -naphthylamine disulphonic acids having the constitutions—



By the further action of fuming sulphuric acid at a higher temperature it has now been found that a  $\beta$ -naphthylamine trisulphonic acid is produced having the formula—



When fused with alkalis the sulphonic acid group in the position 4' is replaced by the hydroxyl group. By heating with dilute mineral acids the sulphonic acid group in the position 1 is removed, and there results 2,2',1'-naphthylamine disulphonic acid. The new amidonaphtholdisulphonic acid is to be employed for the production of dyestuffs of the benzidine series, either to give symmetrical or mixed derivatives, and the colouring matters obtained may be further diazotised and combined on the fibre. The usual methods are given for obtaining the dyestuffs, and the following quantities are employed for the production of the new raw product:—50 kilos. of the acid sodium salt of one of the above-mentioned  $\beta$ -naphthylamine disulphonic acids, or of a mixture of the two, are mixed with 150–200 kilos. of fuming sulphuric acid containing 40 per cent. of anhydride, keeping the temperature low. The melt is then gradually warmed on the water-bath until a test when diazotised and combined with K salt gives a readily soluble orange-red dyestuff. The melt, after careful dilution, is then worked up by the usual methods into the sodium salt of the new  $\beta$ -naphthylamine trisulphonic acid. In order to convert this into the amidonaphthol disulphonic acid, 50 kilos. of the sodium  $\beta$ -naphthylamine trisulphonate, 100 kilos. of caustic soda, and 50 litres of water are heated in an autoclave to 160°–220° C. until no further increase in the amount of dyestuff occurs on testing with a diazo compound. The melt, after cooling, is then acidulated with hydrochloric acid, and the acid sodium salt of the new amidonaphthol disulphonic acid separates out in matted needles. Dilute solutions of this salt show a violet-blue fluorescence, which turns blue on the addition of alkalis. Ferric chloride added to the same solution produces a green, whilst chloride of lime gives a yellow colour. The diazo compound is orange-yellow and can be salted out: it turns violet-red with alkalis.—T. A. L.

*Improvements relating to the Manufacture of Colouring Matters.* H. H. Lake, London. From K. Ochler, Offenbach-on-the-Maine, Germany. Eng. Pat. 22,204, November 20, 1893.

BRUSHING colouring matters for unmordanted cotton are produced according to this patent by combining one molecular proportion of tetrazo-diphenyl or -ditolyl with one molecular proportion of 1,1',3,3'-amido-naphthol disulphonic acid and one molecular proportion of 2,3-dihydroxynaphthalene. The tetrazo solution from 11 kilos. of tellurine or 9.2 kilos. of benzidine, 75 kilos. of 12.5 per cent. hydrochloric acid, 500 litres of water, and 7 kilos. of sodium nitrite is run into a solution containing 18 kilos. of the acid potassium salt of the above-mentioned amido-naphthol disulphonic acid and 8 kilos. of caustic soda. After a short time a solution of 8 kilos. of 2,3-dihydroxy-naphthalene and 1 kilos. of caustic soda in 100 litres of water is run in, and having stood 24 hours the mixture is heated and the dyestuff precipitated with salt, filter-pressed, and dried. The colouring matter forms a blackish-brown powder soluble in water with a violet and in concentrated sulphuric acid with a blue colour.—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

"*Antibenzene-pyrine.*" R. Kissling. Chem. Zeit. 18, 1894, 330.

ANTIBENZENE-PYRINE, which is recommended as a means for preventing the electric excitation of the benzene (petroleum spirit) commonly used in the dry-cleaning of wearing apparel (this Journal, 1894, 385), is a yellow turbid liquid of sp. gr. 0.716. It begins to boil at 50° C., 63 per cent. distilling over at 180° C. The residue is a brownish jelly which on ignition leaves an ash, 90 per cent. of which consists of magnesia. This residue contains about 40 per cent. of fatty acids and 60 per cent. of unsaponifiable hydrocarbons boiling at from 240 to 280°. Accordingly antibenzene-pyrine consists of from 60 to 65 per cent. of benzene of sp. gr. 0.700, from 20 to 25 per cent. of hydrocarbons of specific gravity from 0.825 to 0.830, and from 10 to 12 per cent. of magnesia soap.—C. O. W.

*The Waterproofing of Materials.* A. Foerster. Färb. Zeit. 5, 287–288.

ONE method of waterproofing consists in precipitating some insoluble compound within the pores of the fabric. If, for example, the material be first steeped in the solution of a compound in which the oxide of aluminium, zinc, tin, antimony, or lead plays the part of an acid, and then be immersed in the solution of a salt of one of those metals, a precipitate of a hydroxide or hydroxides results: e.g., from potassium aluminate and zinc acetate are formed hydroxides of zinc and aluminium. Or, the first bath may contain a soluble silicate, and the second a salt of some metal which forms an insoluble silicate. All these precipitates, however, act in some way injuriously upon the fabrics, generally rendering them stiff and harsh. On the other hand, if fatty substances, such as stearin, are employed, the materials are oily and greasy to the touch.

A combination of metallic salts and fats gives a better result. Two baths are prepared: the first contains an emulsion of soap with potassium aluminate and sodium silicate; the second is a concentrated solution of aluminium acetate. By successive steeping in these baths the material is waterproofed, the precipitate consisting of aluminium silicate together with the aluminium compound of a fatty acid. Squeezing between rollers and washing complete the process. The proportions in the first bath should be 90 grms. of fatty acid and 60 grms. of silicic acid for 100 kilos. of water. The amount of aluminium acetate must be twice or thrice the quantity theoretically necessary.

Materials thus waterproofed exclude rain, but allow air to penetrate, and preserve their essential properties, unimpaired.

An interesting process is that patented by Dr. J. Hoffert, of Berlin, who makes use of the fact that gum and gelatin suffer a change under the influence of formaldehyde, losing their adhesive property and becoming completely insoluble in water. The fabric is steeped in a solution of gum or gelatin and is then subjected to the action of gaseous formaldehyde or its solution in water. Most probably this renders the fabric air- as well as waterproof, and, if so, although useful for many purposes, it is quite unsuitable for clothing. The patent specification states that fabrics so prepared are specially useful for medical bandages, owing to the well-known antiseptic properties of formaldehyde.—R. B. B.

## PATENTS.

*Improvements in Scouring, Degreasing, and Cleaning Wool and in Apparatus therefor.* Frederick N. Turney, Nottingham. Eng. Pat. 8179, April 22, 1893.

THE material to be degreased is enclosed in perforated wire-gauze boxes or trays and conveyed into suitable vapour-tight chambers, containing a series of tanks arranged stepwise, through which some volatile solvent is constantly flowing. The trays containing wool travel in a direction contrary to that of the solvent, and are regularly dipped into the tanks and withdrawn; previous to drying, the cages, if made collapsible, may pass through squeezing rollers to remove excess of solvent. The wool is then conveyed into a suitable heater and dried; the vapour given off is condensed, so as to be used over again in addition to that recovered from the tanks. Special provision is made to prevent loss of vapour, both when the wool enters and when it leaves the degreasing chamber. The above process, although specially applied to the degreasing of wool, may also be used for cotton waste, leather scraps, &c.—W. R.

*A New and Improved Process for the Treatment of Textile Vegetable Fibres, more especially those of the Urtica Family.* A. F. B. Gomess, South Kensington. Eng. Pat. 15,945, August 23, 1893.

THE cementing matter in the epidermis is mostly removed by boiling in alkalis. The epidermis is rendered brittle by boiling in a strong solution of some efflorescent salt, e.g., neutral sulphate of soda, drying; the epidermis and cementing matter are then subsequently removed by rotating the same in a suitable drum or otherwise. The fibre may then be bleached by any known means.—W. R.

*A Chemical Preparation for Cleaning and Reviving all Kinds of Clothing.* H. Mällerson, London. Eng. Pat. 17,711, September 20, 1893.

THE preparation described is a mixture of turpentine, 26½ parts; ammonia solution, 19 parts; methylated spirit, 25 parts; ether, 2½ parts; acetic ether, 2½ parts; and water, 25 parts; all by weight. The claim is "a compound for use in cleaning and reviving all kinds of clothing."—L. A.

*New or Improved Process and Machinery for Imparting a Silky Appearance to Cotton and other Yarns.* H. Jacob, Argenteuil, France. Eng. Pat. 19,095, October 11, 1893.

THE cotton or other yarn is first prepared by passing through a liquid obtained by distilling together methyl alcohol and β-naphthol disulphonate of sodium. The threads are then separated from each other by passing through thread guides and thread plates, and then through a first series of dressing boxes containing very fluid collodion, and dried by passage through a series of drying chests. The alcohol and ether given off from the collodion are suitably condensed and used for preparing further quantities of collodion. The dressing and drying is repeated a second time, using collodion of

greater viscosity; and again a third time, the collodion being still thicker. The drying chests are connected by dressing boxes, which are formed of a central tube communicating with one of the collodion distributors. To the ends of this tube are connected by union joints other tubes of the same diameter, and opening into the interior of the drying chests. These tubes only communicate with each other by small orifices, which are of the same diameter as the threads to be dressed. The collodion is forced through the tube by a piston plate resting on the collodion in the receiver, and is weighted.

The capillary tube has inlet and outlet orifices provided for the passage of the thread at the same time the collodion is forced on to it, the excess of collodion being squeezed off previous to drying.

The threads finally pass through glazing rollers which have a rotatory, also an alternating rectilinear movement, so that the whole of the thread surface is submitted to the glazing action.

The collodion may or may not be coloured by aniline dyes.—W. R.

*A Paste or Mixture for Cleansing Various Materials and Articles.* M. Baerlein and C. Dreyfus, Manchester. Eng. Pat. 19,733, October 20, 1893.

See under III., page 939.

*Improvements in Machines for Washing Fibres.* J. Lazarus and M. Jefferson, Bradford. Eng. Pat. 20,197, October 26, 1893.

THE invention consists, firstly, of an improved construction, arrangement, and combination of levers designed to ensure a relatively increased efficiency, and a powerful weighting or "nip" of the squeezing rollers.

A second improvement has reference to the propulsion of the fibre in the washing-machine. In the washing-machine a grid or tray is placed in the sud-bowl; it has a rising and falling motion, and also a frame with a corrugated or ribbed under-surface inside the grid or tray, having a reciprocating to-and-fro motion; the fibre is carried forward between the grid or tray and frame, and is passed on to the squeezing rollers.—W. R.

*Improvements in the Mode of and in Apparatus for Removing Fibre from Cotton-Seed.* R. Silcock, Poulton-le-Fylde. Eng. Pat. 24,546, December 20, 1893.

THE cotton-seed is fed into a revolving cylinder, whilst hot sulphuric acid of about 120° T. is being sprayed upon it. The cylinder is perforated so that the seeds with the larger fibrous covering upon them are retained in contact with the acid a longer time; when the external fibrous matter is destroyed, the seed is mixed with phosphate powder (preferably mineral) to neutralise the excess of acid employed. The mixture is then introduced into a second revolving cylinder, also perforated, containing emery wheels fixed on the axis and revolving at a high speed; emery blocks are also attached to the cylinder in the spaces between the wheels. The fibrous portion of the seeds becomes rubbed off and falls through the perforations. The dust formed is drawn off by an exhaust fan; it forms a valuable fertiliser.

The cotton seed, if necessary, is washed in a bean-washer, dried, and crushed.—W. R.

*Improvements in or relating to the Carbonisation of Rags, Wool, or the like.* Gebrüder Schüll, Düren. Eng. Pat. 12,047, June 21, 1894.

THE rags, wool, or the like, to be carbonised, are contained in a perforated or wire lattice-work covered drum, enclosed in a chamber. In the walls of the chamber is a system of hot-air passages for the purpose of heating the drum referred to. Acid fumes are conveyed into the chamber and penetrate into the interior of the perforated cage, which is continually rotating; the rags, &c., thus constantly present fresh surfaces to the action of the fumes. The

cotton dust is shaken out by the motion of the drum, and regularly removed from the chamber by a convenient opening. Since atmospheric air has no access to the chamber, any chance of an explosion is thus avoided.

—W. R.

## VI.—DYEING, CALICO PRINTING, PAPER-STAINING, AND BLEACHING.

*The Influence of the Structure of Fibril Fibres on their Attraction for Colouring Matters.* G. von Georgievics. Chem. Zeit. Rep. 18, 1894, 170.

THE author has endeavoured experimentally to answer the question as to whether, besides the chemical character of the substance of the textile fibre and of the colouring matter employed, also the structure of the material to be dyed plays any part in the absorption and fixation of the dye. Comparative dye-trials were carefully made with cotton and precipitated cellulose, using Benzopurpurin 4 B, also with the same materials and Benzo-azurine G and Methylene blue. The same textile materials were also tested as to their behaviour with tannin solutions. Dye-trials were further made with mercerised cotton, and similar ones with precipitated cellulose, using Benzopurpurin 1 B and Benzopurpurin G as the typical dyestuffs. The influence of structure was also tested on the absorption of dyestuff in the cases both of wool and asbestos. From these tests the conclusion is drawn that the structure of the fibre does exert an influence on the absorption of the dyestuffs employed. The fibrous nature of the material and the structure of the same not only influence the absorption of the dyestuff, but also influence the fixation of the latter on the material. These conclusions were especially confirmed through the results obtained with wool and asbestos. Both materials in a finely pulverised form, to which they had been reduced by purely chemical means, the question of chemical change of the substance being thus excluded, showed a far less capacity in such form of powder to absorb colouring matters than in that of the original fibre.—R. B. B.

*Substantive Azo Colouring Matters from 1,4-Naphthylamine Sulphonic Acid.* M. D. Bona. Farb. Zeit. 5, 335.

THIS is a study of the connection between the fastness of colouring matters and their constitution. The conclusions are: (1) That the presence of 1,4-naphthionic acid, influences favourably the fastness to alkalis; (2) It is unfavourable to fastness to acids; (3) It influences the shade of the dyestuff in the direction of red; (4) The naphthionic acid dyestuffs are inferior in fastness to light to those containing salicylic acid.—R. B. B.

*Sodium Polysulphide, a New Material for Domestic and Industrial Scouring, Bleaching, and Cleansing Purposes.* A. Dankelman. Chem. Zeit. 18, 1894, 332.

SODIUM monosulphide is largely used for the unhairing of skins, but its domestic use was prevented by reason of its corrosive nature. By treating the polysulphides of calcium with sodium carbonate, solutions of sodium polysulphide free from monosulphide are obtained, which are mixed with sodium carbonate and sold under the name of "Polysulfine." This preparation possesses a mild alkaline action and dissolves or emulsifies fatty matters adhering to the textile fibres. Especially in wool-scouring it was found that polysulfine very readily dissolves all the suint without in the least affecting the fibre.—C. O. W.

Stains. W. Cloth caused by Ammonio-Cupric  
(O. Walter. *Färb. Zeit.* 5, 1894, 164.

THE bottoms of milling and washing machines are frequently lined with brass or copper, and these metals often cause stains, especially when ammoniacal liquors are used in them, as is frequently the case. This was found to be due to the formation of ammonio-cupric oxide, which subsequently acting upon the cloth, causes an alteration of the colour-absorbing capacities of the wool, especially increasing its affinity for indigo. By providing glass bottoms in such machines this objection is avoided.—C. O. W.

*Progress in the Industry of Fast Woollen Dyeing.*  
Oesterr. Wollen- u. Leinen-Ind. 1894, 14, 220.

ALIZARIN colours are more and more gaining ground in this industry, especially blue alizarin dyestuffs, which have been much favoured during the past year by the fashions; as have also alizarin-cyanines, which latter have come to the front in producing the blues having a greenish shade with carded yarns, serges, and chevrons, they being used in conjunction with the fluochrome mordant. In certain factories aniline black is much used for loose wool, and, moreover, not with the old single bath process, but with wool previously mordanted with potassium bichromate and cream of tartar. This method permits also of the combination of alizarin black and logwood, which has been tried for the new grey mantle cloths of the German army. The Austrian Government has recently made the use of alizarin red compulsory for dyeing the breeches worn by the cavalry; further investigations are in progress on the use of alizarin colours for dyeing mantle cloths artillery brown and grey. Of the smaller States, Roumania was the first to use aniline black as a substitute for logwood.

In Germany experiments are being conducted by the Badische Anilin- und Soda-fabrik on the use of Anthracene blue in conjunction with Mordant yellow of that firm, both for grey and hunter's green. Mordant yellow has superseded the older Wool-yellow, which was less fast than fustic, as has also Cassella's Anthracene yellow, which covers better than Mordant yellow.

As in the dyeing of loose wool, so also in piece-dyeing, the use of alizarin dyestuffs is increasing, although in many factories they are being kept back by bad ingrain dyes.

The writer acknowledges the indifferent ingrain tinctorial power of the alizarin dyes, and ascribes it to the rapid formation of lakes. It may be, however, that the recently patented process, whereby the alizarin dye is first applied in an acid bath and subsequently fixed by means of chromium salts, will give a better result. It is still an open question whether in the process of mordanting with "fluochrome," which has advantages over potassium chromate, it is possible to protect the copper vessels by the introduction of strips of zinc. The writer has obtained satisfactory results on a small scale.

Among the substitutes for alizarin colours the following has been introduced:—Diamine fast red, Anthracite black, and Diamond black. The first has been used for loose wool, whereas the latter are employed principally for yarns, especially Anthracite black, which is used for dark blue and black. For navy blue, Galloeyanine is much used in spite of its fugitiveness towards light. Of new colouring matters which withstand the milling process, Chloramine yellow and the sulphocyanines are worthy of mention.

The writer finally discusses the subject of dyeing loose wool with indigo dyes; it appears that the new "hyposulphite vat" is now generally in use for bright shades, whereas for deep shades the old wood vat has advantages.

—A. R. L.

*Canaigre.* S. Kapff. *Färb. Zeit.* 5, 290—291.

In *Färb. Zeit.* 1, Nos. 18 and 19, the author published the results of his research on the tannin-containing canaigre root of New Mexico and Arizona, and concluded that the extract of the root might possibly serve to replace sumach extract. He has since made experiments in America on the large scale with unfavourable results.

A quantity of the undried root was sent from Arizona to New York and made into an extract at 30° B. This gave a clear reddish solution and worked favourably, as on the small scale. There is no objection to its use, except that of the cost, but this appears insurmountable, and is entirely due to the freight expenses. Even when the percentage of water is reduced by drying in the sun, from 58 to 25 per cent., the cost of transport still renders the price too high for competition with sumach, although the price of land is almost nothing, no cultivation is necessary, and the negro labour for collecting the roots is cheap. No one has yet shown sufficient enterprise to set up extraction plant on the spot. There is thus no possibility of the extended use of canaigre until present sources of tannin are exhausted.—R. B. B.

*The Effect of Certain Metallic Salts in the Direct Development of Insoluble Azo Colouring Matters on the Fibre.*  
Gaberti and Pecc. *Färb. Zeit.* 5, 333—335.

EXPERIMENTS were made with Paranitraniline red, Amido-azo-benzene red,  $\alpha$ -Naphthylamine grenate, Benzidine puce, and Nitrosamine (B. A. S. F.). The metallic compounds employed were various salts of iron, copper, zinc, manganese, chromium, lead, and nickel; also vanadium chloride, calcium, magnesium, and aluminium acetates, potassium ferro- and ferri-cyanide, and alkaline solutions of iron, chromium, zinc, and aluminium (e.g. sodium aluminate).

Moderately concentrated solutions of the metallic salts were added in various proportions to the printing colour prepared in the ordinary manner, which was then printed on cotton previously prepared with sodium- $\beta$ -naphthol; the fabric being subsequently dried, passed once through a Mather and Platt's steaming apparatus, and soaped.

Salts of copper exercise a remarkable effect on the shade; the printing colour containing amido-azo-benzene, paranitraniline, or nitrosamine, together with a copper salt, gave, on printing, eutech-browns, of greater or less intensity according to the quantity of copper. These shades are fast to light, soap, and acids, especially those from Cassella's paranitraniline. An example of the process of preparing the printing colour is here given: 62 grms. Paranitraniline C (Cassella) are dissolved in 200 cc. HCl at 21° B. and 155 cc. boiling water. Now add 309 cc. cold water and 300 grms. ice. Add gradually 272 cc. sodium nitrite solution (145 grms. per litre), leave for 20 minutes with occasional agitation, filter, and pour the filtered solution into a mixture of 2,200 grms. of gum tragacanth (75 grms. per litre), with 250 grms. of sodium acetate, and 186 grms. of copper nitrate (17° B.).

The browns from amido-azo-benzene and nitrosamine lose much of their brilliancy in the passage through the "Mather and Platt."  $\alpha$ -Naphthylamine grenate is rendered duller by copper salts, and benzidine puce becomes yellower.

If, instead of adding the copper compound to the printing colour, it is mixed with the sodium- $\beta$ -naphthol solution in the form of a copper soap, prepared with glycerol and ammonia, very red shades of eutech-brown are yielded with  $\alpha$ -naphthylamine, not differing greatly from brazil wood browns. With paranitraniline, &c., the shades are the same as when the copper salt is mixed with the printing colour, but the preparation with sodium- $\beta$ -naphthol and a copper soap does not answer well in practice, for the fabric thus prepared quickly becomes brown, and clear whites cannot be obtained. The printing colours containing copper salts, with the exception of the nitrosamine copper chloride mixture, remain undecomposed for a sufficient time to permit of their practical application.

Zinc chloride decomposes all azo colours, and if mixed with the printing colour a considerable evolution of gas results, with such quantities of foam that even printing tests on the small scale are impossible. A similar effect results from the use of potassium ferrocyanide.

Iron salts, especially ferrous sulphate, serve as resists for the red and grenate, but do not give clear whites; in this respect they are surpassed by potassium ferrocyanide, which when applied in concentrated solution, thickened with dextrin, forms an excellent resist for Paranitraniline red.

Other metallic compounds, salts of chromium, manganese, zinc, nickel, or vanadium, merely tend to impart to the shade a more bluish tone. Alkaline chromium solutions destroy the colour. Salts of aluminium and the alkaline earths influence the shade towards yellow. Sodium zincate and aluminate destroy the colour. Potassium ferrieyanide has no apparent action.—R. B. B.

*Dyeing with Lead Chromate from the Hygienic Standpoint.* Cazeneuve. *Rev. d'Hygiène*, 1894, **16**, 382.

See under XVIII. B., page 970.

*An Improved Form of Hard Glass Beakers for Dyeing Tests.* R. Lepetit. *Färber Zeit.* **5**, 1894, 183.

See under XXIII., page 978.

## PATENTS.

*Improvements relating to the Dyeing of Dyed Cotton Yarns and the like.* W. P. Thompson, Liverpool, Manchester, and London. From T. Salzmann, Dornbirn, Austria. Eng. Pat. 21,843, November 15, 1893.

This invention is an improvement in the method of drying, which follows the dyeing process for cotton yarn, slivers, or roving, described in Eng. Pat. 17,872 of 1890 (this Journal, 1891, 918), and consists in the use of a peculiarly-constructed reel or spool. The reel consists of a wire spiral and a number of longitudinal bars of wood or other material, each bar being made in two parts, which are grooved to receive the spiral, and screwed or otherwise fastened together. On this skeleton reel the yarn is wound after dyeing, and dried preparatory to further treatment, the drying being quickly accomplished owing to the air having free access to the material from both the interior and exterior of the reel.—R. B. B.

*Machine for Loosening and Drying Yarns and Threads after Sarching, Dyeing, and like Operations.* J. T. Blass, Seiffhennersdorf, Saxony. Eng. Pat. 10,993, June 6, 1894.

The yarn is clamped between two rollers attached to a revolving arm, and, while the arm is in rotary motion, strikes an anvil block. This block consists preferably of a wooden roller mounted on an adjustable axle, the use of a cylindrical block obviating injury to the yarn by contact with sharp edges. By means of springs one of each pair of rollers can be turned and held in an upright position, to admit of the introduction and removal of the yarn.—R. B. B.

*Improvements relating to the Dyeing of Fibre.* H. H. Lake, London. From M. E. Waldstein and A. H. Peter, New York, and E. Spott, Brooklyn, U.S.A. Eng. Pat. 14,223, July 24, 1894.

Stannic acid is fixed on the fibre in the form of an insoluble stannate. After mordanting with a stannic salt in the ordinary manner, the fibre is passed through a bath containing, for example, lead acetate or an ammoniacal solution of a barium salt. The insoluble stannate thus formed has no injurious action on the fibre, whereas stannic acid tends to render it brittle and rotten.—R. B. B.

## VII.—ACIDS, ALKALIS, AND SALTS.

*The Occurrence of Perchlorate in Potassium Nitrate.* C. Haussermann. *Chem. Zeit.* 1894, **18**, 1206-1207. (See page 979.)

The author confirms the observation of Hellich that the "pure" nitre intended for gunpowder manufacture has been found invariably to contain perchlorate. The samples were almost free from chloride. The fact that no chlorate was present was ascertained by heating the solution with a copper-zinc couple and testing the resulting liquid with silver nitrate. No greater turbidity than that due to the trace of chloride originally present was produced. Further, the nitric acid distilling over on heating this nitre with excess of sulphuric acid at 100° C., was nearly free from chlorine that could be recognised by means of silver nitrate. Even the acid coming over at a temperature above 100° C. gave only a slight reaction for chlorine, but towards the end of the distillation an acid was obtained which contained a noteworthy amount of chlorine. Perchlorates are only attacked by sulphuric acid above 100° C., whereas chlorates are decomposed before 100° C. is reached.

It may be admitted that Hellich's statement that the chlorine compound constantly present in nitre is a perchlorate is correct, but it does not appear that any danger can arise from this impurity in nitre used for making gunpowder, as samples thus contaminated have been successfully employed for years. The removal of the perchlorate on a manufacturing scale could be effected by keeping the salt fused in a cast-iron vessel until no increase in the reaction for chlorine in a test-portion withdrawn from the mass from time to time, was obtained. In the event of this plan being adopted special care would have to be taken to avoid the presence of nitrite in the finished recrystallised product. The origin of the perchlorate is to be traced to the sodium nitrate used in preparing the potassium salt by double decomposition. That the perchlorate actually exists in Chili saltpetre containing 0.5-1.5 per cent. of sodium chloride, is indicated by the circumstance that the nitric acid prepared from nitrate of this description is richest in chlorine towards the end of the distillation, the amount coming over being large at the beginning of the process (decomposition of chloride) and at the end (decomposition of perchlorate), and least in the middle fraction. Perchlorate probably exists ready formed in caliche, which is known to contain iodate. In the analysis of Chili saltpetre the probable presence of perchlorate should be considered, and the return of the nitrate by difference should, the author thinks, not be regarded as adequate.—B. B.

*The Analysis of Potassium Nitrate.* A. Hellich. *Chem. Zeit.* 1894, **18**, 485.

See under XXIII., page 979.

*Sodium Polysulphide ("Polysulfine"), a new Material for Domestic and Industrial Scouring, Bleaching, and Cleansing Purposes.* A. Dankelmann. *Chem. Zeit.* **18**, 1894, 332.

See under VI., page 945.

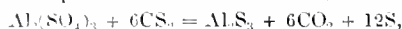
## PATENTS.

*Improvements in the Manufacture of Alumina and Aluminous Compounds and Metallic Aluminium or Alloys of Aluminium, and obtaining Chlorine, Hydrochloric Acid, Carbonate of Soda, and other By-Products resulting from this Manufacture.* D. A. Peniakoff, Paris. Eng. Pat. 20,604, April 5 (under International Convention), 1893.

Pure alumina is obtained by melting bauxite with sodium carbonate, and precipitating the cleared solution by carbonic acid. But the alkaline aluminate is obtained more economically by heating bauxite with sodium sulphate, a process which yields also sulphur dioxide and oxygen, according to

the equation:  $\text{Al}_2\text{O}_3 + \text{Na}_2\text{SO}_4 = 2\text{NaAlO}_2 + \text{SO}_2 + \text{O}$ . The effluent gases are mixed with "a proportionate quantity of hot air," and passed over previously calcined bay salt heated to about 500° C. to form sodium sulphate, chlorine being evolved mixed with the excess of nitrogen. If steam is used hydrochloric acid is obtained instead of chlorine. The sodium aluminate is decomposed, as in the first process, by carbonic acid, to obtain alumina and recover sodium carbonate. When using sodium sulphate it is preferred to add sodium sulphide in the proportion indicated by the equation,  $4\text{Al}_2\text{O}_3 + \text{Na}_2\text{S} + 3\text{Na}_2\text{SO}_4 = 8\text{NaAlO}_2 + 4\text{SO}_2$ , and pyrites may replace the sodium sulphate; in which case the sulphur dioxide may be burned into sulphuric acid. But if it is preferred to pass the gas over sodium chloride, some oxidising material capable of easy regeneration, such as barium peroxide, calcium manganite or plumbite, or cupric sulphate, is added.

Aluminium sulphide for application in obtaining aluminium is produced by passing carbon bisulphide over aluminium sulphate heated to "nasecent red," thus—



and the carbon bisulphide is regenerated by passing the sulphur vapour over incandescent carbon. The process is modified by heating aluminium sulphate with a sulphide, as sodium sulphide, in vapour of sulphur, the final products being aluminium sulphide, sodium sulphide, and sulphur dioxide. Another method consists in heating a mixture of alumina and calcium sulphide in sulphur vapour to obtain aluminium sulphide and sulphur dioxide, with recovery of the alkali sulphide. It is stated that the function of the sulphide consists in transferring the sulphur in vapour to the alumina, at the same time depriving the latter of its oxygen, so that "an extremely small quantity of sulphuret is sufficient to sulphurate considerable quantities of alumina or of sulphate of alumina."

Lead or iron may be used for reducing aluminium sulphide to obtain aluminium. It is stated that lead does not form alloys with aluminium; but that when iron is used as reducing agent, the aluminium may be obtained pure or alloyed with iron. In regenerating the lead or iron sulphide, the sulphur dioxide evolved may be utilised in obtaining sulphuric acid. Sodium may replace lead or iron in reducing aluminium sulphide. It is pointed out that aluminium sulphide being much richer in metal than the chloride or fluoride, it is better suited for electrolytic reduction, and it is stated that when fused it dissolves alumina easily, and that the mixture may be electrolysed as readily as the pure sulphide.—E. S.

*Improvements in the Construction of Ammonia Apparatus.* G. E. Davis, Salford. Eng. Pat. 19,630, October 18, 1893.

HEXAGONAL caps, steadied by wings, are placed on the division plates of the ammonia apparatus, covering a number of small apertures, instead of the central aperture covered by a cap, described in Patent 16,349, 1888 (this Journal, 1889, 884). A bent dip-pipe is used to convey the liquor from the division of the apparatus immediately above the liming chamber to a point above the liquor level, in order to prevent the entrance of lime to the upper portion of the apparatus. A perforated tray with hexagonal caps is placed at a spot above where the liquor enters the apparatus, so that the caps, when luted by steam condensed upon them, may act as washers to arrest mechanically-carried particles.—E. S.

*Improvements in the Manufacture of Cement from Chance or Ammonia-Soda Alkali Waste or the like.* B. K. Rigby, Ditton, F. A. R. Neill, St. Helens, and A. C. Carr, Rainhill. Eng. Pat. 19,705 (amended), October 19, 1893.

THE inventors have found that the waste from the ammonia-soda process, and from the Chance sulphur-recovery process, may be utilised in making cement, if first washed in a prescribed manner, and then incorporated with so much lime or other alkaline earth as will reduce to 3½ per cent., or preferably to 3 per cent., the proportion of injurious matter retained by the waste. The proportion of lime to

be added is determined by analysis of the mud, and the constituents named as injurious are calcium chloride and sulphate, sodium chloride and sulphate, iron sulphide, and free sulphur.

For washing the waste, a double tank, or one having more than two compartments, is used, into the upper part of which the waste, mixed with water, is run through a pipe having as many valved branches as there are compartments. The tank is first supplied with water to about three-fourths its height from a water pipe. The finely-divided waste gradually settles to the bottom in a washed state. The supernatant liquid is removed by pipes placed at suitable heights. The washed waste is emptied out through a mud cock by aid of a hose, or through the manhole door.

The amendment consists in the substitution of the word "Ammonia-soda waste" for "Le Blanc alkali waste," as formerly used.—E. S.

*Improvements in Acid Raisers or Steam-Jet Pumps for Raising Acids, Alkalis, and other Liquids.* C. Long, Walsall, and W. H. Burkitt, Wednesbury. Eng. Pat. 5187, March 13, 1894.

See under I., page 930.

*Improvements in Condensing Plates.* J. W. Mackenzie, London. From Brante and Co., Lyons, France. Eng. Pat. 7229, April 11, 1894.

See under I., page 930.

*Improvements in the Process of Manufacturing Carbonic Acid, and in the Production of By-Products, such as Sodium Sulphate and Magnesium Sulphate.* E. W. Enequist, New York, U.S.A. Eng. Pat. 14,228, July 24, 1894.

NITRE-CAKE (acid sodium sulphate) containing about 25 per cent. available acid, is dissolved in about twice its weight of hot water, and is brought into contact with magnesite in excess under conditions admitting of the collection of the carbonic acid evolved. The solution, containing sodium and magnesium sulphates, is drawn off into tanks, in which Glauber's salt crystallises out. The solution containing now sodium-magnesium sulphate, is concentrated to 42°–45° B., when the double salt crystallises. If it is desired to obtain a larger proportion of sodium sulphate, sodium chloride is added, either in the first stage of the process or later. The sodium-magnesium sulphate may be decomposed by an alkaline hydrate or carbonate, to obtain magnesium hydroxide or carbonate and an alkaline sulphate; or the double salt may be decomposed by an alkaline earth hydrate.—E. S.

## VIII.—GLASS, POTTERY, AND ENAMELS.

### PATENTS.

*Improvements in Moulds for Moulding Terra Cotta Blocks and like Articles.* H. L. Doulton and J. M. Carr, Lambeth. Eng. Pat. 19,445, October 16, 1893.

TERRA cotta blocks are moulded in plaster of Paris moulds, and hitherto the massive and cumbersome outer case of the mould has been made likewise of plaster of Paris; a large amount of plaster is needed for them, and when the mould is no longer required the material of which the case is made is useless. In the present invention the outer case is made of a fibrous asphalt which is readily worked when hot, and



sets firmly when cold. The material can be used over again by melting the material. The composition preferred is pitch, 1 cwt., old plaster moulds, ground, 56 to 75 lb.; cocoa fibre, 4 to 8 lb.—V. C.

*Improvements in the Manufacture of Tubes and Vessels of Covered Glass.* N. Browne, Cheapside. From G. Kastner, Dresden. Eng. Pat. 10,159, May 25, 1894.

These are improvements in tubes and receptacles, consisting of a glass-core and a protecting covering of paper or fabric surrounding it.

Glass tubes and vessels covered in the customary manner with layers of impregnated paper or other material are apt to break from shock, and if they do so break or splinter, portions of glass are liable to be carried by a liquid flowing through the tubes to parts such as stop-cock valves, whereby they are liable to do damage.

In the present invention the glass tube is separated from its envelope of paper or the like by a layer of viscous asphalt, preferably that known as "Goudron Mineral," with 3 to 5 per cent. mineral oil added. Elastic rings separate the tube from its paper envelope at the two ends. By this arrangement the liability to fracture is much diminished, and if breakage does occur the fragments of glass adhere to the viscous asphalt.—V. C.

*Process of Decorating Pottery, Glass, or similar Goods.* Percy Shelley, Longton, Staffordshire. Eng. Pat. 13,047, July 5, 1894.

The design is taken from the engraving in the usual manner, printers' oil being used either alone or mixed with non-metallic colouring matter, and the design is at once transferred to the article. The paper is then pulled off and the design (in oil) is dusted over with the colours. By this process decorative effects are obtained which have hitherto only been produced by painting, filling in, or laying on the colours by pencil or similar implement. A further advantage is that a design can be printed in various colours in which each colour can be produced in any part of the design without being superimposed upon the colour in which the design has been transferred.—V. C.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Effect of Frost on Building Construction.* Zeits. Oesterr. Ingenieur- und Architektenvereins, 1894, 7.

VARIOUS experimental walls were constructed in Vienna in the winter of 1890, and the behaviour of different cementitious materials was thus ascertained. The walls were 1 m. in length, 2 m. in height, and had a thickness of 0.3 m. They were built with common mortar, Roman cement, Portland cement, and slag cement, the water used being in some cases at the prevailing winter temperature, and in others at a temperature of 25° C. Water containing 7 per cent. of common salt was also used for some of the tests with Roman and Portland cement. The external temperature ranged from -3° C. to -16° C. The results of the experiments may be summarised as follows: Portland cement (to which no common lime had been added) gave good results, the remaining materials being inferior thereto. In the case of Roman and Portland cement the addition of salt appears to be useful.—B. B.

*Quick-Setting Cement.* H. Geyer. Thonind. Zeit. 1894, 18, 280.

CERTAIN hydraulic cements set almost instantaneously. The author found that reburning such cements at a high temperature had no influence on their rapidity of setting, but

on preparing mixtures with a varying ratio of alumina to silica, he arrived at the result (already well known) that a high content of alumina is favourable to rapid setting. He points out that it is not sufficient to know the ratio of alumina to the total silica in the raw materials, as the latter may be present as coarse quartz, and be more or less inert, thus disturbing the calculated ratio which assumes that both silica and alumina are completely combined as silicates and aluminates.—B. B.

*Improvements in Testing Hydraulic Cement.* C. Prussing. Thonind. Zeit. 1894, 18, 251.

The author submits the following propositions for consideration:—

(1.) The standard test for the constancy of volume ("soundness") of hydraulic cement can no longer be considered adequate.

(2.) Cement can be considered faultless in respect of constancy of volume when two test-pats, after 27 days' hardening, the one in cold water, the other in water at 90° C., remain hard and perfectly free from buckling or cracking.

(3.) It is desirable to insert in the standard rules for testing, a rapid test for constancy of volume, consisting in allowing test-pats to harden for 24 hours in the air, then for two hours under cold water, and finally for 22 hours under water at 90° C.

(4.) Cements, to be designated "slow-setting," should remain at least two hours after gauging without showing signs of incipient setting.

(5.) A slow-setting cement should not be considered faulty because its setting is accompanied by a notable rise of temperature. Many slow-setting cements of excellent quality begin to set after five or six hours, and then set completely in one hour, giving a rise of temperature of 5°—7° C. Provided such cements stand the pat-test, they are generally better than such as begin to set after half an hour to one hour, and finish the setting process after about seven hours, as in this gradual setting the evolution of heat is so distributed that no perceptible rise of temperature takes place.

(6.) It is not correct to judge the quality of cement merely from the 28 day cold test on tension specimens. Compression tests and hot tests after 7, 14, and 28 days should also be used.—B. B.

## PATENTS.

*Improvements in the Manufacture of Cement from Chance or Ammonia Soda, Alkali Waste, or the like.* B. K. Rigby, Ditton, F. A. R. Neill, St. Helens, and A. C. Carr, Rainhill. Eng. Pat. 19,705, October 19, 1893.

See under VII., page 948.

*Improvements in the Preservation of Wood.* E. Groebe, Koenigsstein, Germany. Eng. Pat. 14,599, July 30, 1894.

Dry wood is placed in a close vessel from which the air is exhausted: a solution of "tribasic or tetrabasic phosphate of lime in sulphurous acid" is then admitted, and the charge is submitted to a pressure of from six to eight atmospheres for about two hours. The wood thus impregnated is dried, preferably in a heated enclosed space, to admit recovery of the escaping sulphurous acid.—E. S.



# X.—METALLURGY.

*The Cyanide Process.* Walter H. Virgoe. Eng. and Mining J. 57, 1894, 533—534.

In the matter of gold-bearing ores, concentrates and tailings, only such material is adapted to the process as is, First: Low-grade, because the higher the grade of the material the stronger the solution of cyanide must be, and the greater the resulting loss of cyanide. An extraction of 80 per cent. on a 25 dollar ore, means a loss in the tailings of 5 dols. per ton. Similar extraction on 100 dollar ore means a loss per ton of 20 dols., besides a heavier loss in cyanide, owing to the necessity of using a more powerful solution. Second: The gold in the ore and concentrates must be in a fine state of division, as potassium cyanide, like chlorine, exerts a very slow solvent action on coarse particles of gold. Third: The material must be entirely free from deleterious compounds capable of absorbing the cyanide, such as copper-bearing minerals and the easily decomposable products of the oxidation of pyrites, such as ferrous and ferric sulphates and basic sulphates, &c. Fourth: The material must, above all things, possess good leaching properties at the size of mesh required for economical extraction.

Again, the manner in which the gold is mechanically carried in the material is of paramount importance. If it is contained in crystals of sphalerite or galena it cannot be extracted, and this is very frequently the case where the gold is contained in mispickel—arsenical pyrites. Some tellurium ores offer considerable difficulty, yet in certain cases, as at Cripple Creek, high percentages have been saved at a small cost of cyanide.

At the Mercuer gold mine, Fairfield, Utah, the ore is a siliceous limestone, containing magnetic oxide of iron, traces of cinnabar and gold. The latter occurs in a very fine state of division, coating the particles of magnetic oxide, and giving the ore a value of about 15·22 dols. per ton. The cost of treatment, extracting 73 per cent. by bullion returns from the refinery works, is about 2·40 dols. per ton, exclusive of royalty, superintendence, and office expenses, thus set forth:—

	Dollars.
Potassium cyanide, 1·27 lb. per ton .....	0·66
Zinc, 0·55 lb. per ton.....	0·05
Labour (7 shifts per 24 hours, 6 day and 1 night) ..	1·12
Supplies, repairs, fuel, freight.....	0·57
	<hr/> 2·40

This expense has been lowered by an increase being made in the capacity of the plant. The ore is a typical one for the process.

The process is far better adapted to the treatment of auriferous than argentiferous material. In fact, it must be admitted that the cyanide process is, comparatively speaking, unsuitable for the treatment of silver ores, and notably because argentiferous ores leach with more difficulty than gold ores in the raw state.

Again, because cyanogen has a greater affinity for gold than for silver, consequently silver ores require long treatment with strong solutions. Silver-bearing minerals are much more complex in their nature than those containing gold, and require finer crushing and agitation, and consume a larger amount of cyanide. Lastly, such silver ores as can be treated successfully by lixiviation methods can usually be more economically handled by some process in which cheaper chemicals are used, and on which no royalties or other charges have to be paid, such as the Van Patera process. However, without discussing the merits of the cyanide process as compared with others, it may be stated that the chlorides and bromides of silver are easily reduced, and extractions of over 80 per cent. have been made on antimonial silver ores, the value of which does not exceed 30 dols. to 40 dols. per ton. Difficulty has always been encountered in treating silver sulphide ores. Those in which the value is contained in galena crystals cannot be handled.

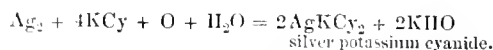
Regarding the qualities in an ore essential for successful treatment, it can be at once seen that large percentages of

extraction may be achieved at a heavy cost of cyanide, and the leaching qualities at the mesh requisite for economical extraction may be such as to render the process worse than useless owing to the length of time required to draw off the cyanide solution and succeeding wash waters. In this latter difficulty is the keynote of many of the failures in cyanide plants. Filter presses have been tried in South Africa, but without satisfactory results, owing to their cost and the power required to work them. No mechanical means have as yet been devised for the satisfactory separation of pulp and solution in the case of poor leaching ores. Such an invention would revolutionize the metallurgical world as far as the wet reduction of ore is concerned.

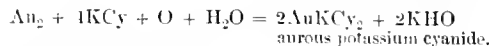
The cyanide process is very far from being a simple one. Many of its difficulties are mechanical, but there are also many chemical problems in connection with it that must be thoroughly understood, in order that a factory may be run with intelligence and economy.

The value of the alkali cyanides in the MacArthur-Forrest process of extracting gold and silver from their ores depends upon the fact that such cyanides possess the property of forming soluble double salts with these metals, viz.:—

I.

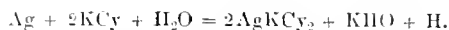


II.



It is possible that hydrogen is evolved during these reactions, as thus shown:—

III.



Since potassium cyanide costs 50 cents per pound, delivered in cases of 100 lb., the primary aim in treating all ores is to insure a maximum amount of extraction, with a minimum consumption of cyanide, consequently all sources of waste should be investigated, and, where possible, averted.

That such sources of waste may be investigated it is necessary to examine closely the reactions due to atmospheric influence liable to occur in the storage tanks; reactions occurring in the leaching vats in treating different classes of ore, also reactions occurring in the zinc precipitation boxes. Considering, first, the following sources of waste liable to occur in the supply tanks:—

1. Sources of waste due to the exposure of the solution to the effects of light and air.
2. Sources of waste due to impurities in the factory water used.

It is well known that cyanide of potassium, when exposed to the air, deliquesces and evolves hydrocyanic acid—

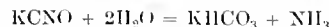


The presence of the hydrocyanic acid gas in large quantities in the atmosphere surrounding the tanks in cyanide factories is always a very noticeable feature.

The evolution of this gas may be brought about in another manner, where the surface of the solution is exposed to the air and is liable to absorb carbonic acid gas. Thus:—



Again, potassium cyanide is a very easily oxidizable compound and when in solution in contact with the atmosphere, is liable to be converted to some extent into potassium cyanate, and this again into the bicarbonate:—

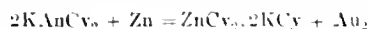


Instances are known where impurities in the factory water used, have decomposed the potassium cyanide to a very marked extent. It will be seen that water holding much carbonic acid gas in solution, or water containing soluble metallic sulphates will decompose a cyanide solution. Hence, water as pure as possible should always be used in factory solutions.

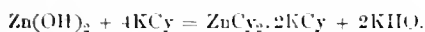
The determination of the sources of waste due to reactions in the leaching vats is a complex and difficult matter. The sources of waste in the leaching vats are similar to those in the storage tanks. There are also the losses due to consumption of cyanide by base metals in the ore treated. In regard to hematite and limonite ores the consumption of cyanide is usually low. This is also the case with sulphide of iron ores, provided the latter be free from all oxidation. The manganese gold ores of California consume a little more cyanide than the corresponding ferruginous ores. Galena and zinc blende are unattacked. Great difficulty has always been experienced in treating copper ores containing more than 2 per cent. or 3 per cent. of copper, owing to the very heavy consumption of cyanide. A quarter of one per cent. solution is invariably the best to be adopted, if ores containing copper must be treated by this process.

Pyritiferous ores are liable to contain, especially when mined above the permanent water-level, soluble salts of iron and small amounts of free sulphuric acid. It is necessary, therefore, in treating ores of this description, to first leach them with water, in order to dissolve the ferrous and ferric sulphates out, and afterward to decompose the insoluble basic sulphates with a solution of soda-lye or lime water.

In the zinc precipitation boxes not only does an amount of zinc enter into solution proportionate to the amount of gold and silver and base metals precipitated, such as Cu, Sb, As, &c., thus:—

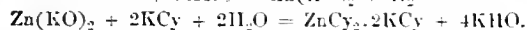
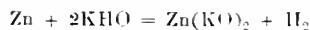


but far more, and the excess is proportionate to the decomposition of the potassium cyanide. Looking at the reactions occurring in an ordinary zinc box, reactions in part purely chemical, in part electro-chemical, involving a waste of cyanide, it is seen that as soon as the gold is deposited on the zinc shavings, galvanic action is at once established between the electro-negative gold and the electro-positive zinc, hydrogen is vigorously evolved at the negative pole and the zinc converted into hydrate, which in turn is immediately attacked by the excess of potassium cyanide, forming zinc potassium cyanide and potash:—



As this reaction is incessantly occurring the solution becomes very strongly alkaline in the lower compartments of the zinc boxes. This galvanic action is a source of a large loss of cyanide, not only by causing the formation of the double salt of zinc and potassium cyanide, but undoubtedly by causing the oxidation of a certain quantity of potassium cyanide to cyanate.

In the majority of factories the mistake is made of not discarding screens made of fine iron wire in the compartments of the precipitation boxes, because there is a strong galvanic action established immediately between the zinc and the iron so soon as the cyanide solution flows through them, hindering the deposition of gold and silver and augmenting to a marked degree the loss of cyanide through solution of the zinc, besides largely increasing the zinc slimes in the product and making them more difficult to handle. Commercial zinc is only slightly soluble in potassium cyanide, but in contact with iron it is readily dissolved, especially in the lower compartments of the precipitation boxes, where the solution is most strongly alkaline. This reaction is thus expressed—



Again, the majority of boxes in use are far too long. There is no advantage to be gained in using boxes 20, 30, and 40 ft. in length.

Precipitation takes place mainly in the upper compartments, after which the solution is unnecessarily exposed to the action of the zinc.

It is best to observe the rule that a larger number of smaller boxes is preferable in economic practice to a small number of very long boxes such as those referred to in the last paragraph.

The cyanide process has a very serious competitor, in the treatment of many classes of ore, in chlorination, "a process

involving the payment of no royalties, and which can be worked far more cheaply than formerly owing to the great reduction in the price of chemicals used."

*The Cyanide Process in the Transvaal Mines.* W. R. Feldmann. Eng. and Mining J. 58, 1894, 102-103.

There are now (about 50) plants, of an aggregate treating capacity of some 230,000 to 240,000 tons per month, at present in use for the treatment of tailings and concentrates on the Witwatersrand are of great variety, both in size and design, the general principle in all of them being, of course, the same. The objects which they have to fulfil consist briefly in: 1. Leaching of the ore, tailings, or concentrates with weak solution of cyanide of potassium for the purpose of dissolving the gold. 2. Treatment of the solution to precipitate the dissolved gold in the metallic form. In all plants we have the three main features: 1. Leaching or filter vats. 2. Launderers, for precipitation—"zinc boxes." 3. Storage vats for solutions.

In considering the treatment required for any particular ore or tailings three points are to be taken into account: the condition of the gold, whether coarse or fine; the material, whether acid or neutral; and what metals with a strong affinity for cyanogen are contained in the material.

The condition of the gold, that is its coarseness or fineness, determines the length of time required in contact with the cyanide solution to effect its dissolution. On the Witwatersrand, generally speaking, the gold is very fine, and a period of 12 hours' standing under a strong solution is usually sufficient with tailings to dissolve as much of the gold as it is commercially profitable to extract. Further treatment with weak solutions is chiefly in the nature of washing to remove the already dissolved gold. In the treatment of pyritic concentrates a very much longer contact with the solvent is required.

By an acid ore—a familiar phrase in the district—is understood the presence of the products of the partial decomposition of pyrites, usually free sulphuric acid, soluble salts, such as ferrous sulphate, or insoluble basic iron salts. All of these substances are destructive to cyanide, forming with it compounds useless in the extraction of gold.

As to other metals found in the ore, certain copper compounds are the most troublesome. It has been found, however, that in many cases this difficulty may be overcome by the use of weak solutions.

The treatment of acid ore, or rather tailings rendered acid by the partial oxidation of the crushed pyritous ore, offers more difficulty than in the case of the free-milling ore. In order to overcome the cyanide-destroying qualities of the acid or iron salts present it is necessary to have recourse to neutralisation by means of an alkali or alkaline earth, caustic soda or lime, with or without a preliminary water washing to remove such soluble "cyanicides" as may be present. If the quantity of cyanide present is large, and, by testing, a considerable proportion of it is found to be soluble in water, a preliminary water wash is generally applied. It has hitherto frequently been the custom to water wash in the same tank in which the subsequent treatment with cyanide is effected, the water wash being run to waste. In the writer's opinion this is a practice which cannot be too strongly condemned, as accounting largely for the so-called "mysterious" discrepancy between the expected yield of gold as estimated from assays made before and after the treatment and the actual return. When a water wash charged with acid out of the ore comes in contact with residual quantities of cyanide solution lying in the bottom and adhering to the sides of the tank, a certain quantity of hydrocyanic acid gas is liberated, which, diffusing through the whole tank, is capable of dissolving a not inconsiderable amount of gold from the ore. Such dissolved gold is not precipitated, even if passed through zinc, and is consequently run to waste with the water wash. If, in order to economise caustic soda and lime, water washing be adopted, it should be done in tanks which are reserved for the special purpose, and the ore should then be transferred to the cyanide treatment vats. This means a little extra cost for handling, but it

will, in most cases, amount to less than the loss of gold incurred by the other method. In order to neutralise the remaining acid, a quantity of a solution of caustic soda, of which the amount necessary must be approximately determined by the experimental test, is run on, allowed to stand for an hour or so, and then drained off into an "alkali" sump, there to be made up to strength for the next lot. Where practicable it is a good plan to mix powdered lime with the ore, as this not only saves caustic soda, but also keeps the cyanide solutions freer from suspended ferric hydrate, which, in the case of using caustic soda alone, makes them very turbid, and fouds the zinc.

The preliminary treatment having been finished, cyanide solutions are applied. The strength of the first solution may vary from 0.25 per cent. to 0.5 per cent. The number and strength of subsequent solutions and washes and the time of contact required depend entirely on the quality and nature of the ore.

The average extraction from acid ore, say from 5 or 6 dwt. stuff, may be put down at 70 per cent. Concentrates of 3 oz. value will give an extraction from 90 up to 98 per cent. in three or four weeks' treatment. With regard to the treatment in the presence of metals with an affinity for cyanogen, the principle usually adopted is to apply the weakest possible solution which has been approved by experience.

Having obtained the gold in solution, precipitation in the zinc boxes is, as a rule, a simple matter. Roughly speaking, it may be said that if solutions leaving the boxes assay more than 2 dwt. per ton the precipitation is not as it should be. This may be owing to the paucity of zinc in the boxes, which should be rectified at once, or too great speed in the flow of the solution, or in a few cases to insufficient cyanide in the solution.

Having reconverted the gold into the metallic form by passage of the solutions through the zinc shavings it is obtained as a sludgy looking precipitate, commonly known as "slimes," and the next step is to get it into marketable shape. This is done by separating from the zinc, drying, roasting, and smelting. The clean up, which takes place once or twice a month, is conducted as follows:—A small amount of clean water is run through the boxes to remove the cyanide solution, which might otherwise be injurious to the workmen. The zinc shavings are taken out after being twisted and scrubbed in the water to remove as far as possible the gold adhering to them. In some cases there is quite a thick plating on the zinc which cannot well be removed by scrubbing, but this is usually ignored, as the shavings are returned to the boxes in any case, and this plating will go into the precipitate of the next clean-up. Having removed nearly all the coarse zinc, the precipitate contained in the water is allowed to settle; an addition of a little alum will considerably assist this process. Most of the clear water is then siphoned or pumped off, and the precipitate together with the remaining small quantity of water is drawn off into a calico or linen filter or a filter press. After drying sufficiently to handle with a scoop the precipitate may be further dried in iron pots and is then ready for roasting and smelting.

The object of the roasting is to oxidise the greater portion of the zinc, which has, in the form of small chips and shavings, fallen through the zinc-box trays, so as to cause it to combine in the subsequent smelting with the fluxes, and leave the bullion fairly fine. Oxidation by the aid of the atmospheric air is sufficient, but a certain amount of the zinc oxide subsequently becomes reduced by the carbon of the plumbago melting pots and re-enters the bullion. A good method of roasting has been found to be the addition of a little nitre, say about 3 to 10 per cent. to the precipitate. It is best applied as a strong solution before drying the precipitate, so that it gets equally mixed with the whole mass.

The precipitate having been sufficiently roasted, the next step is to mix it with suitable quantities of flux, and smelt the mixture in plumbago pots. The fluxes commonly used are bicarbonate of soda, borax, and sand.

One of the great bugbears of the cyanide men on the Witwatersrand has been the treatment of slimes, by which

is meant the very fine, or in the case of free-milling ores, the clayey portion of the tailings. Many suggestions have been made for the treatment of these, but the only really practicable scheme so far appears to be to allow them to dry thoroughly, and by screening or otherwise to reduce them to a fine powder. This powder is thoroughly mixed with sandy tailings, and the mixture will usually allow of percolation fairly well. The trouble is that if these slimes go into the vats in half-dried lumps they will absorb liquid and pass away as slime. There is no chemical difficulty in the way. Another solution suggested is dry crushing and direct treatment of the powdered ore with cyanide.

An important discussion now going on relates to the question of removing the pyrites from tailings by concentration before treating the latter by cyanide.

The cost of treating tailings by the cyanide process is necessarily determined in large part by local conditions, the nature of the ore treated, and the special facilities for handling the ore. The biggest item of cost is generally for the cyanide of potassium, which probably averages on the Witwatersrand about 48 cents per ton of ore treated.

#### *The Cyanide Process.* W. R. Feldtmann. Eng. and Mining J. 53, 1894, 218—219.

MR. VIRGOE, in describing the nature of the material suitable for treatment by the cyanide process, names four points as essential:

1. He says that the ores to be treated "must be low-grade because the higher the grade of the material the stronger the solution of cyanide must be, and the greater the loss of cyanide," and points out that 20 per cent. left in a 25 dols. ore means less loss of gold than 20 per cent. left in a 100 dols. ore. While granting that the treatment of high-grade ore, concentrates, or tailings entails the use of comparatively strong solutions, and consequently increased loss of cyanide, the author would suggest that higher grade material will generally bear a greater working cost. Farther, experience on the Witwatersrand is that whereas 80 per cent. is a good extraction from low-grade tailings of say 5 dols. or so, we can treat concentrates running 60 dols. and upward to obtain a 90 per cent. to 95 per cent. extraction. As regards cost, it is mentioned that the Crown Reef Gold Mining Company is treating its concentrates (from blankets) by cyanide at a cost of about 5.50 dols. per ton.

Coming to the question of the various sources of loss of cyanide, the author states that the amount of the chemical wasted in the zinc boxes is probably not so great as Mr. Virgoe seems to think. From some experiments tried in the laboratories of the African Gold Recovery Company, it appears that, by means of pure zinc-potassium cyanide (without admixture of free potassium cyanide) gold is dissolved from ores.

Mr. Virgoe can satisfy himself, the author considers, that all the cyanogen in the zinc-potassium cyanide can be combined with an alkali, and can be determined by the silver nitrate test by simply rendering a solution of the double salt fairly, strongly alkaline, by addition of sodium or potassium hydrate.

In actual practice it is found that addition of alkali to working solutions which have become somewhat weak in alkali brings up the strength by regenerating, *i.e.*, decomposing the zinc cyanide. Potassium iodide being used as an indicator, this is not a fallacious result effected by the misleading action of free alkali on the silver nitrate test. So that, as a matter of fact, when the solutions are pretty strongly alkaline they contain no zinc as cyanide, but only as hydrate dissolved in alkali (zincate of potash, &c.). In consequence, the author would prefer to err on the side of somewhat excessively long boxes and thorough precipitation rather than too limited a precipitating surface, and consequent pumping back into the tailings or ore of solutions assaying high in gold.

*Sweep Smelting and Refining of Gold, Silver, and Platinum Metals.* Bettel. Johannesburg Chem. and Met. Soc., July 28, 1894.

THE processes in outline are as follows:—The material for smelting was mixed with Derbyshire lead slag and fluorspar, and melted in a furnace containing molten lead and leady copper matte. The material having been melted down, the copper being received into the matte together with some gold and silver, the remainder, with the exception of a small quantity passing into the slag, was retained in the molten lead. During melting, it may be remarked, a certain proportion of the lead and silver passed away in fume. The remainder of the processes consisted in extracting the gold, silver, copper, lead, platinum, palladium, and iridium from each other.

*Notes and Queries on Cyanide Works.* Caldecott. Johannesburg Chem. and Met. Soc., July 28, 1894.

IN the re-treatment of a mass of residues by the African Gold Recovery Company, from which on the average 70 per cent. of the original gold had been already extracted, the extraordinary fact was discovered that their value was no longer 15 dwts., but 9 dwts. only. Explanations as to the reason of this discrepancy were sought for in vain, until, when all the residues had been re-treated, the site they had occupied was sampled, with the result that the top 3 ins. were found to assay 38 dwts. fine gold per ton, evidently derived from the soluble gold, produced by the cyanide solution, contained in the residues, continuing to act after discharge, and being gradually washed downward by the rain. The Barrett Gold Mining Company at Kaapsche Hoop, some 30 miles west of Barberton, is probably unique, in that much of its ore is treated by cyanide, and very good extractions obtained, without preliminary crushing in a battery or other machine. The ore is of an earthy nature, containing much ferric hydrate, and is so soft that it does not require blasting for extraction, but is dug out with pick and shovel. By sifting through trommels worked by hand, the finer portion, after mixing with tailings to assist filtration, is fitted for cyanide treatment. These trommels, or cylindrical rotary screens, are also very useful in separating coarse tailings from lumps of slimes. They may be seen in operation at the Caledonia Works of the African Gold Recovery Company, near the Meyer and Charlton battery. For this purpose they possess the following advantages over the incline sieves generally used:—

1. Greater capacity: a sieve 8 ft. long and 3 ft. in diameter has a capacity of 50 tons per day, and requires six Kadlirs to work it.
2. Cleaner product: sieving of three or even four holes to the linear inch may be used.
3. Perfect mixing of lime with tailings, when this is required.
4. Delivery of lumps into trucks without further handling.

It will be generally admitted that the weight of a cubic foot of tailings depends upon, at least, the following factors, all liable to variation from time to time:—

- (1.) Nature of material, *e.g.*, whether weathered or pyritic.
- (2.) Degree of fineness; other things being equal, the finer the material the lighter it is.
- (3.) Whether or not closely packed, as in a settling pit or lower portion of a deep vat, or much tossed about, as by the operation of sifting.
- (4.) Percentage of moisture contained.

It is mentioned in Mr. W. R. Feldtmann's valuable article on gold extraction by means of cyanide (see page 952) that solutions, after leaving the zinc boxes, should not contain more than 2 dwts. per ton of fine gold. Assuming that this amount is present, it is asked if this would not account for the soluble gold in the residues, which is sometimes considered mysterious? Every ton of water introduced into the works, as moisture in the tailings charged, obviously necessitates a ton of dilute cyanide solution discharged as moisture in the residues. When a final water washing is

used, this dilute solution is, of course, contained in the lower portions of the residues only. Here we would seem to have the reason for the lower portion of the residues being richer than the upper, simply because it is moistened with gold-bearing solution instead of water, and theories as to the cyanide solution carrying rich gold slimes downwards through the charge, and depositing them near the bottom, would appear needless. As some 20,000 tons of dilute solution (8 per cent. on the total tonnage of tailings treated) is discharged monthly on the Rand as moisture in the residues, the loss involved, assuming it to contain only the dwts. per ton, would amount to 50,000 lb. per annum, and the necessity for constant watchfulness to ensure proper precipitation in the zinc-boxes by using solution of sufficient strength, sufficient zinc, and an equable slow rate of flow is obvious.

*Ore Roasting with a Blast.* L. Klotz. Chem. Zeit. 1894, 18, 953—954.

FREQUENTLY complete roasting is not required, as, for instance, with silver-bearing copper pyrites, which, in order to obtain a good matte, should contain from 10 to 12 per cent. of sulphur after roasting. It is often stated that ores should be dead roasted, and the required proportion of sulphur made up by adding raw ore. Practical men, however, know that this is not correct. The O'Hara furnace, which has lately been much used, has a production of from 24 to 36 tons in 24 hours. Its first cost and up-keep are, however, more than many mines can afford. Brückner cylinders are theoretically good, yet they have only been used in a few districts, for partially roasting easily treated ores, or for chlorinizing. For more perfect roasting, or where high temperatures are necessary, they are not suitable. A new roasting furnace has lately been designed by Bartlett, originally for making zinc white, but it is also applicable for roasting pyrites. It is rectangular in shape and has a bed of perforated cast-iron plates, through which a low pressure blast is introduced; from the upper part of the furnace a flue leads to the dust chambers.

Live coal, or burning wood, is spread over the bed, and covered with a uniform layer of a mixture of roughly broken ore and fuel, and the blast turned on. The roasting is completed in about an hour, when the roasted product, which is in the form of porous slabs, is withdrawn. After a few shovelful of coal have been thrown on the bed the furnace is ready for re-charging. The consumption of fuel, which may be coal slack, or fine coke or charcoal, is about 10 or 12 per cent. of the weight of ore roasted. Ore of all percentages of sulphur, from medium to high, can be roasted; it is not suitable for lead ores, however, as a considerable loss of lead takes place, although no loss of silver has been observed. A blast has also been tried with other forms of roasting furnaces. In applying it to the Brückner cylinder, the refractory lining was removed, and six iron ribs attached to the inside of the outer cylinder. To these a second cylinder of perforated iron was fixed. The ribs divide the space between the two cylinders into six compartments. From each of these an iron tube leads to a central distributing valve, so arranged that it only admits the blast to those compartments over which the ore lies as the cylinder rotates. In the "Pearce" furnace the stirring arms and shaft are made hollow, and the former are perforated so as to admit the blast into the midst of the mass which is being roasted.—R. B. P.

*The Manufacture of Steel in the Open Hearth.* J. A. Lencavech. Iron and Steel Institute. Autumn Meeting, 1894. The Ironmonger, 68, 1894, 366—368.

AFTER dealing with the advantages of the open hearth process, the author describes a furnace with stationary hearth erected at Aazin to produce steel of extra welding quality for tube manufacture. The bricks for the burners and roof are made of magnesite, and special precautions are taken to prevent the accumulation of dust in the chequer work by having straight passages without any projections. The chief point of the process is the injection of cold air through the sides or roof as well as the usual blast into the

from 5 to the extent of 5 to 15 per cent. of the weight of the air necessary for the complete combustion of the gas. The air jets are directed on to the metal bath, by the use of thin lined tuyeres, to lay bare the metal and to remove the glassy layer of slag or cinder from its surface so as to facilitate oxidation. The air jets likewise cause an intimate mixture of the hot air and gas as they enter the hearth, and by so doing raise the temperature of the flame and direct it on to the surface of the metallic bath. The final result of the combustion is only slightly modified by them.

At first sight a cooling effect might be expected, but the result is otherwise, as the air-jet uncovers the metal and stimulates the oxidation with the consequent combustion of the liberated carbon monoxide. Without the use of the sloping tuyere, this gas mostly escapes combustion "as it is separated from the zone of hot air by a layer of the products of combustion which only contain a very little free oxygen." Under these circumstances it only burns in the regenerators, where it is less effective than in the hearth. In the pig and scrap process the relative proportion of pig has been increased, the charge now consisting of two-fifths of hematite iron and three-fifths of old iron, scrap, &c., for the production of extra-weldable steel with 25 to 30 per cent. elongation. Previously the furnaces produced only 30 to 32 tons of steel per 24 hours, but owing to the prevention of stoppages by the use of the dust-catchers, &c., the minimum out-turn is 1,000 tons of ingots per month of 25 effective working days, or on an average, 40 tons per day. The coal consumption in the producers has at the same time been reduced from 748 lb. to 605 lb. per ton of steel made. As the object of blowing in air is to refine as much pig-iron as possible by the air as well as to increase the out-turn there should ensue less costly conditions owing to the smaller quantity of slag, and to its removal at a relatively low temperature.

The author announces his intention of making further trials with the blast at higher pressures and also with the ore process, and gives the following as the probable mode of procedure. The metal is fused at a relatively low temperature in a slightly oxidising flame to obtain the desulphurisation by the manganese contained in the pig-iron or added in the form of ferro manganese. The temperature is then raised by blowing to oxidise silicon and phosphorus. Lime is added to neutralise the last traces of phosphoric acid. Combined with the blowing it will be possible to oxidise by successive additions of hematite or other ores, which should be as pure as possible and possess calcareous gangues. It will be advantageous to reduce the use of these oxides to a minimum especially when they are difficult to obtain. The elimination of sulphur, phosphorus, and silicon is effected at temperatures lower than that at which decarburisation commences, or, in other words, at which carbonic oxide is generated. At these relatively low temperatures the basic walls do not suffer. Decarburisation can be performed with a very small minimum of ore, or the ore may be entirely omitted, for the main points for consideration are the shortening of the operation as well as obtaining economical results. The smaller the quantity of ore the less will be the amount of slag, the shorter will be the time required for repairs, and the lower will be the cost of converting the iron into steel.

In the discussion, Mr. James Riley stated that the author had given them the credit side for the hastening of the process, but he had not given the debit side for loss and expense of blowing, &c. He could not understand why they should try to accomplish in the open hearth furnace inefficiently that which could be so well done in the Bessemer converter. Mr. G. J. Snell said that in the ore process if the metal was to be oxidised by air they would not recover the metal from the oxide put in. The great advantage of the open hearth ore process was that by the reduction of the added ore as much steel was obtained from the furnace as there was pig-iron put in, which meant that they got the steel from the added ore for practically the price of the ore. This advantage would be sacrificed by the plan proposed. On the other side the process is put forward as of advantage in cases where good ore is not obtainable. —A. W.

*The Use of Caustic Lime in the Blast Furnace.* Sir Lowthian Bell, Iron and Steel Inst. Autumn Meeting, 1894. The Ironmonger, 68, 1894, 346—351.

It has been found that a preliminary calcination of limestone results in a considerable advantage in blast furnaces of about 50 ft. in height, but in the case of the modern furnaces of 80 ft. high the saving in fuel does not repay the cost incurred in calcining in the lime-kiln. The author infers from experiments that the carbon dioxide is not expelled from calcium carbonate until the temperature is reached in its descent in the furnace where the dioxide is reduced to carbon monoxide by the heated coke. If this be correct, then in the loftier furnaces the carbon dioxide from the fuel is probably absorbed by the calcined limestone in the higher and cooler portions, only to be decomposed again lower down, as in the case of the uncalcined limestone. Experiment showed that a moderate elevation of temperature greatly expedited the union of carbon dioxide with caustic lime, so that the above combination might take place extensively in the warm reducing zone.

It is found that the use of raw limestone entails an absorption of about 4,200 calories for the expulsion of carbon dioxide, and a further 4,300 calories in decomposing the liberated carbon dioxide, or 8,500 calories in all. This is much reduced by a preliminary treatment in the limekiln, although the calcination in the latter is very imperfect. The author gives a large number of figures showing the variations in calories used, &c., in different furnaces, using raw and calcined limestone. The temperature of the furnace gases, when the latter is employed, is higher than with the raw limestone by between 100° and 200° F. This results firstly from the greater weight of the raw limestone, and secondly, because the union of carbon dioxide with lime should such take place, is a source of heat.

The conditions under which heat is applied in the blast furnace are more favourable to economy than most other modes of raising the temperature of bodies. On the other hand, the action of the lime-kiln is very defective. It is not surprising, therefore, that whereas one unit of coke burnt as it is in a blast furnace only affords about 4,200 calories, one unit of coal burnt in a lime-kiln gives off nearly double this amount. The loss in the latter case by the ignition of the coal near the upper surface of the kiln is such that nearly 16,000 calories are evolved to effect that which is accomplished with about 5,000 calories in the blast furnace. As a matter, therefore, of commercial difference there is not much to choose between the coke in the furnace and coal in the kiln. The small coal used in the kiln is only half the price of coke, but of the former double the weight is required.

The author discusses the question of heat distribution and appropriation irrespective of the question of lime. An increase of 300° F. (from 1,100° to 1,400°) in the temperature of the blast by the change of stoves is shown to have very little effect on the temperature of the escaping gases—a rise of 40° only. But the temperature of combustion must rise in some proportion or another, and whether it does so to any appreciable or dangerous extent or not is an important question. From the character of the slag there does not appear to be much inconvenience as a result of the change in the temperature of the blast.

The opinion that furnaces heated with superheated air have a disposition to "hang" is generally accepted as correct. The author does not say that with a temperature of blast under 1,000° F. this difficulty is entirely overcome, but he has little doubt "that it has increased many hundred fold in the few years that fire-brick stoves have been in use at the Clarence works." Moreover, the tendency complained of "seems to increase with the age of the furnace," whilst again, with the higher temperature of blast there is greater irregularity in the quality of the iron and consequently in the consumption of coke. These irregularities are largely due to the want of uniformity in the blast temperature, which latter seems inseparable from the nature of brick stoves.

In the discussion, Mr. Chas. Woods said that for years he had economically used calcined limestone, with the result that there was a saving in coal, and that the furnace gases were better and hotter for generating steam. He, however, calcined the limestone and ironstone together in about equal

proportions. Mr. C. Cochrane, in a written criticism, takes exception to the statement that it is impossible to discriminate between the carbon dioxide due to reduction and that liberated from the limestone in the furnace, and says that it is quite easy to do so. He calculates the ratio of carbon dioxide to monoxide in a perfect working furnace and from any deviation from this works out his result. He regrets that Sir Lowthian Bell should continue to use the "fictitious standard" of "heat afforded per unit of coke," and thinks the unit of carbon should be employed.—A. W.

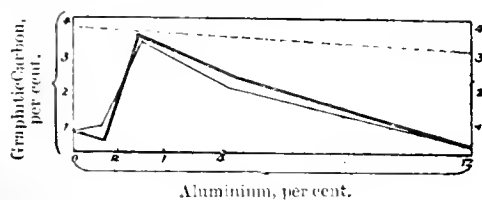
*The Influence of Aluminium upon the Carbon in Ferro-Carbon Alloys.* T. W. Hogg. Iron and Steel Inst. Autumn Meeting, 1894. The Ironmonger, 63, 1894, 355—358.

THE author gives the results of his experiments with a series of alloys containing aluminium in different proportions, with as little as possible of both silicon and manganese. Two kinds of pure Swedish pig-iron were used, one grey and the other white. The alloys were prepared by pouring the molten iron into the molten aluminium, and then pouring the mixture backwards and forwards twice. The metal was cast in two moulds, one of iron to cool rapidly, and the other of green sand to cool slowly. Alloys containing 1, 2, 4, 8, and 12 per cent. of aluminium were prepared and analyses of each were made with a view of ascertaining the effect on the carbon.

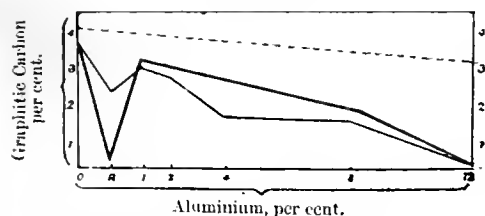
As a result, the most effectual primary change is obtained by the addition of 1 per cent., and each addition after this causes more and more of the carbon to pass into the combined state until practically the whole of it is in this condition in the 12 per cent. alloy. In all cases the rapidly-cooled alloys show a remarkable excess of graphitic carbon over that contained by the slowly-cooled alloys, and the author suggests the explanation that the aluminium may cause a precipitation of carbon in the molten alloy, and the more slowly cooled portions partially take up the carbon again by cementation. It is also observed that the percentage of total carbon in the white pig alloys is not diminished more than that produced by the addition of the aluminium, whereas in the grey-pig high-percentage alloy there is a decided loss. The addition of 12 per cent. of aluminium to the iron containing 4.33 per cent. of carbon should reduce this element to 3.80 per cent. However, only 3.44 per cent. could be found by analysis. The following curves show the results:—

DIAGRAMS ILLUSTRATING THE ALTERATIONS IN THE PROPORTION OF GRAPHITE IN PURE SWEDISH PIG-IRON AFTER ALLOYING WITH ALUMINIUM.

WHITE PIG.



GREY PIG.



Dotted lines = Total carbon.  
Thick lines = Graphite in rapidly cooled alloy.  
Thin lines = Graphite in slowly cooled alloy.  
O = Original pig.  
R = After remelting without aluminium.

It is to be observed that all tendency for carbon to be retained in the combined state is prevented in the most decided manner by the addition of 1 per cent. of aluminium, and that every increase upon this quantity tends to reverse this effect, until with a large proportion, the carbon in white pig-iron is retained in its original condition, and the graphitic carbon present in grey pig is entirely changed to the combined state.

In the case of the high-percentage alloy, there was an evolution of large quantities of gas on solidification, the alloys in this respect resembling the high-silicon alloys. The holes in the top portions of the ingots were completely filled with glistening graphite, not actually separated from the alloy, but due to decomposition of the issuing carbonic oxide gas. There is a great difference between ferro-aluminium and ferro-silicon in respect to the total carbon which they can take up, the saturation point for the latter is probably about 1.5 per cent. for an alloy containing 18 per cent. of silicon, whilst a corresponding ferro-aluminium was found by experiment to hold more than 10 per cent. (3.15 per cent.) of carbon. The effect of remelting ferro-aluminium was then tried, with the result that there was a considerable loss of aluminium with a slight increase of silicon. The addition of manganese retards the loss of aluminium, but with a great increase in the proportion of silicon. The influence of aluminium upon a high-silicon alloy does not appear to give any important results beyond showing a great change in the grain of the alloy.—A. W.

*The Alloys of Iron and Nickel.* M. H. Wedding. Moniteur Scient. 8, 1894 (Sept.), 666—674.

THE "Société d'Encouragement à l'Industrie" having decided to undertake a complete study of the alloys of iron with nickel, aluminium, and chromium, has commenced it with the preparation of a memoir detailing all the recent researches on nickel, and alloys of nickel and iron. M. Wedding, the president of this commission, finds that the chief differences between the old and modern results on nickel are due to impurities in that metal.

After tabulating the properties of nickel compared with iron, manganese, and cobalt, the author quotes largely from the researches of Riley on nickel steels (this Journal, 1889, 547 and 596).

In conclusion the commission have decided with regard to nickel steels "That despite all the energy devoted of late years to the study of nickel-iron alloys and of the good results obtained, which seem to justify the adoption of nickel steel for armour-plating, absolutely reliable results are still wanting." "We know nothing precisely as to what is the percentage of nickel and of carbon, at which we must stop to obtain a metal perfect, either from the point of view of ductility or from that of resistance. We are still less sure of the conditions which are most favourable for the melting, the casting and the working of the ingot. The researches of Riley are far from satisfying us, and the earlier work is useless."—A. W.

*The Employment of Aluminium in the Refining of certain Metals.* Moniteur Scient. 8, 1894 (Sept.), 661—665.

At present half the aluminium manufactured is used up in the iron industry. The remainder is largely used in refining nickel and copper. When added to these metals the reduction of the last traces of oxide is completed, the metals become more perfectly fluid and after cooling can be worked easily. Any alumina formed in this action is completely insoluble in the nickel or copper, and rises to the surface and thus eliminates itself. The question of how the aluminium acts in the case of steel is then reviewed. Rammelsberg found that all the aluminium was used up in deoxidising, no trace being found in the ingot obtained. At first it was thought that aluminium lowered the melting point of steel 200° to 300° C., and that its presence caused the great fluidity of the steel. Now the ingots are shown to contain no aluminium. The oxide of iron dissolved in steel renders it less fluid and more brittle, and this causes it to give off carbon dioxide, hydrogen, and nitrogen. By the use of aluminium no

in flux) is given off, but only hydrogen and nitrogen. On account of the removal of the oxide of iron with the formation of alumina. Arnold states that the carbon dioxide is decomposed by the aluminium and the carbon is again taken up by the iron, but it is difficult to see why the quantities of hydrogen and nitrogen should be at the same time reduced.

The quantity of aluminium to be added and the method of using it is then discussed. The proportion depends on the metal, and the amount of oxide of iron therein. Iron or steel with little carbon requires more than high carbon steels, and Bessemer steel more than Siemens-Martin. In practice the addition rarely exceeds 0.1 per cent. An excess causes white excrecences of alumina. So long as the quantity does not exceed 0.75 per cent. the metal will remain homogeneous and capable of hammering hot. Ingots containing too much aluminium are characterised after forging and rolling by a slight separation of aluminium as such, and of carbon as graphite, thus damaging the quality of the metal. Another objection is the considerable contraction of the ingots on cooling. It is preferable therefore to employ a little less aluminium, and to run the risk of a few gas bubbles being in the casting. It is not necessary to employ aluminium purer than 92—98 per cent., if the impurities are only silicon and iron.

Aluminium cannot replace manganese entirely, as some contend, as it cannot affect the sulphur, phosphorus, and silicon. The chief advantages of aluminium in steel are:—(1.) It dissolves much more quickly in the bath and mixes more completely. (2.) Such small quantities are required that its introduction does not lower the temperature; and (3.) Since its reduction in price it can compete with ferromanganese and ferro-silicon, provided it is employed judiciously. It favours the alloying of nickel with iron.

In refining copper the amount varies from 0.25 to 1 per cent. The aluminium diffuses itself almost instantaneously, and is a more powerful reducer than phosphorus, silicon, or manganese. It increases the resisting power of the metal without affecting sensibly its electrical conductivity. An excess has no bad effect on the quality of the final product.

Aluminium is also now employed to replace the higher priced magnesium in the refining of nickel, and the quantity used should theoretically be less than the quantity of magnesium, but this point has not yet been attained practically. In America aluminium is used in galvanising iron plates, 12 grammes to the ton of zinc being added; it renders more perfect the adherence of the zinc to the iron and gives a finer surface polish.—A. W.

*Colour Gauges for Carbon Testing.* W. G. McMillan.  
Iron and Steel Inst. Autumn Meeting. 1894.

See under XXIII., page 978.

## PATENTS.

*Improvements in the Manufacture of Alumina and Aluminous Compounds and Metallic Aluminium or Alloys of Aluminium, and obtaining Chlorine, Hydrochloric Acid, Carbonate of Soda, and other By-products resulting from this Manufacture.* D. A. Peniakoff, Paris.  
Eng. Pat. 20,604, April 5, 1893.

See under VII., page 947.

*Manufacture or Production of Substances of Extreme Hardness.* F. Chaplet, Paris. Eng. Pat. 15,453, August 14, 1893.

Dry mixtures of the required components are placed within a crucible and subjected to the heat of an electric furnace so as to produce hard crystals or amorphous masses (a) by the direct and simple union of the mixed materials; (b) by their union in the presence of an excess of metal which is subsequently removed by means of an acid; or (c) by

reduction of an oxide or salt of one of the constituents, and dissolving the resulting substance in a metal, from which it is subsequently obtained by removal of the metal.

The substances so produced consist of silicides, borides, silico-carbides, silico borides, &c., of titanium and other metals and metalloids, and are capable of scratching or cutting chrome steel, the ruby and the diamond. As an example the preparation of silicide of titanium may be referred to. Eighty-two parts of rutile in coarse powder are mixed with about 60 parts of sand or crushed quartz and about 48 parts of powdered coke or carbon. These form the materials. By the fusion, as above described, a grey mass is obtained, with a bright fracture, of great hardness, and a density of about 4.8. It contains, besides titanium and silicon, about 20—25 per cent. of carbon and varying amounts of nitrogen.—J. H. C.

*An Improved Solder for Use with Aluminium or Aluminium Alloys.* B. I. Roman, London. Eng. Pat. 17,623, September 19, 1893.

The solder consists of silver, nickel, aluminium, tin, and zinc, in the proportions as follow:—

	Per Cent
Silver.....	2
Nickel.....	5
Aluminium.....	9
Tin.....	34
Zinc.....	50

No flux is necessary, and any soldering iron or tool may be used, though one of aluminium is preferable.—J. H. C.

*An Improved Aluminium Alloy.* R. I. Roman, London.  
Eng. Pat. 19,543, October 17, 1893.

The alloy consists of copper, tungsten, aluminium, tin, and antimony, for either of the two latter manganese or nickel being at times substituted. The proportions preferred are somewhat as follows:—

	Parts.
Copper.....	0.375
Tin.....	0.105
Antimony.....	1.442
Tungsten.....	0.038
Aluminium.....	98.049
	100.000

Tungstic acid and cryolite are melted together, equal proportions being employed. When the temperature reaches 1,200° C. aluminium is added so as to produce a 10 per cent. compound of aluminium and tungsten. A second alloy is made containing equal proportions of aluminium and copper. These two alloys are then melted together with pure aluminium in the proper proportions to form the alloy required as above: tin, antimony, or their substitutes being added in the necessary proportions; or they may be left out altogether when the copper and tungstic acid originally employed are chemically pure.—J. H. C.

*Improvements in and in connection with the Extraction of Gold and Silver from Ores or Compounds containing the same.* J. C. Moutgomerie, Ayr. Eng. Pat. 20,343, October 27, 1893.

The pulverized ores are mixed with sodium or other alkaline dioxide and then treated with a solution of potassium or other cyanide, nitrate of soda or other nitrate or nitrite being added when necessary. The reactions are made to take place in closed vessels, air, oxygenated air or oxygen being supplied under pressure. The solutions so obtained are filtered through charcoal filters, which are regenerated from time to time by means of heat, superheated steam, air, or other suitable gas without removing the retained precious metals, so that the same body of charcoal may be used for filtering successive charges of solution.—J. H. C.



*Improved Means of Purifying the Effluent Waste Waters of Tin Plate Works and the like.* W. J. Pughley, Briton Ferry, Glamorganshire. Eng. Pat. 13,777, July 17, 1894.

THE "mother liquors," "scouring water," or other waste waters are passed through a series of connected filters, of which the first contains calcined limestone in pieces not more than 3 in. diameter; the second, burnt oyster or cockle shells; and the third, stick charcoal. The filters may be of any suitable material or construction, and connected by earthenware piping, or may be contiguous with perforated partitions between. It is preferred that the effluent waste water from "black pickle" should, before filtration, be deprived of part of its iron and acid by concentration and crystallisation.—E. S.

*An Improved Process of Treating Refractory Ores, and Apparatus to be Used therein.* E. A. Ashcroft, Broken Hill, New South Wales. Eng. Pat. 13,850, July 18, 1894.

THE ores are first oxidised when necessary by roasting or calcining, they are then leached, and the solutions so obtained are electrolysed. The roasting may be effected in any kind of furnace that may be preferred; the leaching is effected by the ferric-chloride process in the case of ores high in zinc and low in lead, or by the ferric-sulphate process combined with smelting for ores high in lead and low in zinc. The required solutions are obtained by an electrolytic "re-generative" at subsequent stages of the work. The electrolysis is carried on, first, in vats, using iron anodes (whereby ferrous sulphate or chloride is imparted to the solution in small quantities in place of the zinc sulphate or chloride decomposed), and then in other vats having insoluble anodes (whereby the ferrous salts previously formed are raised to the ferric state). The solutions so produced are then heated and used for the treatment of new proportions of oxidised ores. Many modifications of the process are described, with additional manipulations necessary when silver, lead, or sulphur are to be recovered.—J. H. C.

*An Improved Metallic Alloy.* J. W. Dixon and Wm. Skinner, Sheffield. Eng. Pat. 14,502, July 28, 1894.

THE alloy consists of aluminium, silver, and copper, preferably in the following proportions or approximately so:—Aluminium, 96.25; silver, 3.50; copper, 0.25 per cent. = 100.00.—J. H. C.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

*The Effects of Mechanical Stress on the Electrical Properties of Metals.* J. H. Gray and J. B. Henderson. Proc. Royal Soc. 55, 283–300.

THE first part of the paper deals with the changes of density observed. The density of a well-annealed copper wire of 98 per cent. conductivity, decreased about  $\frac{1}{2}$  per cent. for a stretching of 25 or 26 per cent. A lead wire also decreased about  $\frac{1}{2}$  per cent. just before breaking.

A copper wire drawn through 12 holes of a drawplate increased in density more than 1 per cent. A manganese-copper wire (10 per cent. of manganese) increased in density about 1 per cent. when drawn down to half its former diameter.

Twisting caused copper to increase about  $\frac{1}{2}$  per cent. for 2.3 turns per cm.

Heating the wires treated above caused no appreciable alteration.

Hammering a copper wire increased its density about 0.2 per cent.

The authors deal in the second part of their paper with the corresponding alterations of resistance, and support at some length the "Weight specific resistance," against the "Volume specific resistance." The formula used is:—

Resistance = wt. sp. res.  $\times$  length per unit wt.  $\div$  length.

Their arguments are founded, firstly, upon the fact that length per unit wt. is more easily determined accurately than the area of cross section required by the usual formula; and secondly, upon the fact first mentioned by Lord Kelvin and borne out by these experiments that wt. sp. res. varies less with mechanical treatment than vol. sp. res.

The resistance of a wire under tension was always greater than that of the same wire unloaded; the maximum increase recorded, of 1.6 per cent., being shown by a steel wire. When, however, the load was removed the resistance returned very nearly to its original value. In the latter respect copper showed much more change than either iron or steel, changing 0.7 per cent., when stretched 16 per cent., while steel did not alter more than 0.07 per cent., nor soft iron more than 0.19 per cent. This permanent alteration was found in the case of copper to be nearly proportional to the stretching, and a wire once well stretched was found to experience no further permanent alteration on being reloaded, though the temporary alteration was proportional to the load applied.

The authors conclude from their experiments that "no mechanical treatment, such as stretching, drawing . . . twisting, hammering, or combinations of these, all of which were tried, had any appreciable effect on the electrical properties of copper, iron, or steel."—E. T.

*Note on the Action of Copper Sulphate and Sulphuric Acid on Metallic Copper.* A. Schuster. Proc. Royal Soc. 55, 84.

IT is known that copper sheets experience a diminution in weight when left immersed for some time in sulphuric acid or copper sulphate, the surface of which is exposed to the air. It seemed possible that this effect was due to dissolved oxygen. To test this, rolls of copper gauze were placed in tubes containing a 20 per cent. solution of copper sulphate, 5 per cent. of sulphuric acid being added to two and 10 per cent. to the rest. The tubes were exhausted and sealed and the copper gauze remained immersed in two cases for seven days, in the rest for 14. The diminution in wt. was insignificant compared with that commonly found, and might be due either to a small quantity of oxygen remaining in the liquid, or, as suggested by Mr. Hoskyns Abrahall, to the formation of copper sulphide, in accordance with the formula:— $4\text{Cu} + \text{SO}_3 = \text{Cu}_2\text{S} + 3\text{CuO}$ . In support of the latter view traces of sulphuretted hydrogen were found when the copper was heated in hydrogen.—E. T.

*Copper Electrolysis in Vacuo.* W. Gannon. Proc. Royal Soc. 55, 1894, 66–83.

SCHUSTER and Crossley have shown (Proc. Royal Soc. 50, 344), that a heavier deposit of silver is obtained by a given quantity of electricity *in vacuo* than in air. Schuster's experiments (an account of which is given in the preceding abstract) suggested that a similar result might be found for copper. The author's experiments bear this out.

Two similar voltmeters containing equal cathode areas were placed in series so that the same current might pass through both. When fresh neutral solutions were used the excess of the deposit *in vacuo* over that in air was distinctly marked, though very variable in amount.

Gray had, however, pointed out (Phil. Mag., vols. 22 and 25) that copper deposits from neutral solutions showed just such variability.

With acid solutions the difference was clear and certain.

With a current density of 0.0027 amp. per sq. cm. 0.16 per cent. more copper was deposited *in vacuo* than in air. This excess appeared to diminish with greater current densities till with 0.01 amp. per sq. cm. (9.3 amps. per sq. ft.), or more, no practical difference could be observed. It was still found, however, that less copper *proportionately* was deposited for feeble current densities than for the stronger ones, as had been previously pointed out by Gray.



A neutral solution which had been used, did not behave, on the addition of acid, like one freshly prepared and to which acid had been added immediately. Moreover, a deposit from a neutral solution *in vacuo* was, like that obtained in air, very sandy and loose at the edges.

The acid solutions contained 1 per cent. of free acid, and a specific gravity between about 1.032 and 1.180.—E. T.

### PATENTS.

*Improvements in the Electrolytic Decomposition of Chlorides and Mixtures thereof, and in Apparatus for the purpose.* F. M. Lyte, London. Eng. Pat. 7594, April 13, 1893.

THIS invention relates to cells and anodes for use in electrolytic (fusion) processes, especially the electrolytic decomposition of fused lead chlorides into metallic lead and chlorine. Electrolysis is effected within a bell-like chamber having an outlet for gas in the crown, the mouth being sealed by dipping into molten metal, corresponding to the base of the chloride treated, which is run off as it accumulates. Electrodes are of hollow, tubular carbon, closed at bottom, open at top, having a core of metal (lead, tin), or alloy, fusible at or below the temperature at which electrolytic decomposition is to be effected. Terminal connection is made to the fusible core by an infusible conductor (iron, copper) dipping therein. Connection may be also made to the same form of hollow electrode by a longitudinally-split tube fitting spring-tight therein, the fusible metal being dispensed with.—J. C. R.

*Improvements in Plating Metallic Surfaces and also Prepared Surfaces of Non-conducting Substances by Electro-Chemical Deposition.* P. Marino, Paris. Eng. Pat. 15,264, August 10, 1893.

A MOVABLE and adjustable jacket filled with the electrolyte and containing the anode plates, is pressed against the surface to be plated in such a way that that surface forms one side of the jacket. A siphon and stop-cock for supplying or withdrawing the electrolyte are provided, and the vessel may, if preferred, be filled with an absorbent, such as felt or "sponge-piline." By using a suitable flexible joint and an elastic material for the edges of the jacket, the whole apparatus may be shifted from one part of an object to another, regardless of any variation in curve. Corners or surfaces of very variable form may be coated by means of a rolling cylinder of porous material saturated with the electrolyte, and having a metallic spindle which acts as anode.—W. G. M.

*Improvements in Electric Furnaces.* F. Chaplet, Paris. Eng. Pat. 15,577, August 16, 1893.

See under II., page 936.

*Improvements in Apparatus for the Electrolysis of Chlorides and other Salts.* J. Hargreaves, Farnworth-in-Widnes, and T. Bird, Crossington, Liverpool. Eng. Pat. 18,173, September 27, 1893.

THIS invention relates to apparatus for use in decomposing chlorides, iodides, bromides, nitrates, and other salts, adapted to operate on the principle claimed in Eng. Pat. 18,871, 1892 (this Journal, 1894, 250), the primary object being to render such apparatus less expensive, more convenient to manipulate, and more compact. Apparatus is shown having numerous vertical compartments containing the electrolyte, and a corresponding number of compartments or collecting chambers, wherein the product is obtained from parallel exposed or bare electrodes. The collecting chambers are arranged alternately with the compartments containing the electrolyte, and the anode collecting chambers alternately with the cathode collecting chambers.—J. C. R.

*Improvements in Safety Appliances to be used in Connection with Electrical Decomposing Apparatus.* J. C. Richardson, London. Eng. Pat. 19,688, October 19, 1893.

IN order to avoid the danger resulting from accumulations of explosive gas mixtures produced by deranged working of apparatus during electrolysis, either a small Bunsen gas-flame, an induction coil spark, an electrically incandescent wire, or a small quantity of spongy platinum, is placed in an apparatus connected with the electrolytic cell, with a view to induce the combination of the gases as they are evolved, and before they collect in dangerous quantities.—W. G. M.

*Improvements in or connected with Electrolytic Cells.* F. Hurter and H. Auer, Widnes, and E. K. Muspratt, Liverpool. Eng. Pat. 19,791, October 20, 1893.

THESE improvements relate to cells to be employed in the electrolysis of solutions of common salt or other chlorides. A metallic containing vessel is employed, the sides of which act as the cathode. The anode is contained in an inverted trough or bell-jar, and a plate or plates of non-conducting material is placed at a distance below the edge of the trough, and projecting on all sides beyond this edge, to prevent the hydrogen liberated at the bottom of the metallic cell from entering the anode compartment.—J. C. R.

*An Improved Process and Means used therein for the Production of Caustic Alkali.* C. T. J. Vantin, London. Eng. Pat. 20,404, October 28, 1893.

THIS process relates to electrolytic (fusion) methods of obtaining hydrates of sodium or potassium from their chlorides, molten lead being the cathode. The hydrates are obtained by treating the cathode alloys, continuously or at intervals, with steam or water vapour in a separate heated vessel, or discharging the alloy into or under water.

—J. C. R.

*Improvements in or relating to Processes and Apparatus for Obtaining and Depositing Metals by Electrolysis, and in the Treatment of the metals after leaving the Electrolytic Bath.* E. Stouls, Paris. Eng. Pat. 24,017, December 13, 1893.

FOR the production of thick electrolytic deposits, rotated copper mandrels filled with wood, and supported by *lignum vite* journals mounted in glass bearings, are used as cathodes. The mandrels are rotated in the bath on parallel axes by means of bronze or copper cranks, stayed with copper tubes, and having bronze journals working in cast-iron or *lignum vite* bearings. The mandrels are coated evenly by painting and rubbing their surfaces with a mixture of plumbago and milk, which is found preferable to the present system of applying the black-lead. The electrolyte must be free from impurities, and is therefore allowed to deposit suspended matter in wide, shallow troughs heated to from 40° to 60° C., which answer better than deep settling tanks. For the purification of the liquid air may be blown through at the temperature named. After leaving the bath the deposited metal is worked under the hammer, by drawing, or otherwise, whether treated by the Elmore process of deposition or not.—W. G. M.

*Manufacture of Coated Metallic Powders.* O. Imray, London. From J. Sachs, New York. Eng. Pat. 12,381. June 26, 1894.

THIS is a process for coating powders or fine particles of one conducting material with an electrolytic deposit of another metal. The powders are fed on to the upper end of a sloping cathode plate, resting at an angle across the bath containing the electrolyte; at some distance above the cathode, and parallel to it, is a perforated anode plate supported on a dialytic diaphragm, which may be of baked clay. Between the electrodes work a series of rotating brushes with horizontal axes, which serve at once to sweep the powders along the cathode and to press them into close

contact with it. In this manner each particle receives a sheath of electro-deposited metal, and the coated powder is discharged at the lower end of the slope through a narrow outlet, together with a little of the solution which is pumped back into the trough. By using suitable electrolyte and electrodes, graphite, iron or zinc may be given the appearance of brass, or metals may be coated with gold, silver, or other material.—W. G. M.

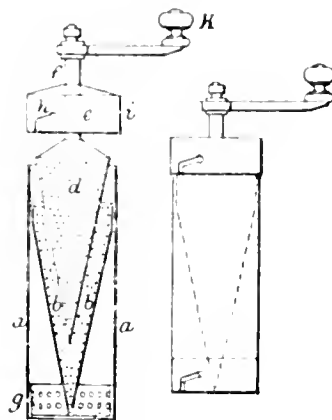
*Improvements in or connected with Electrolytic Cells, and Diaphragms therefor.* C. N. Waite, Rumford Falls, U.S.A. Eng. Pat. 13,756, July 17, 1894.

THE inventor describes an electrolytic cell for the decomposition of alkaline chlorides which, in general character, is fully explained in Eng. Pat. 5983, 1891 (this Journal, 1891, 707), granted to E. A. Le Sueur. The improvements consist in placing against diaphragms a layer of sand or equivalent comminuted material. In the drawing it is represented as placed on the anode side and resting on a diaphragm (asbestos) which, in turn, rests on a cathode of "wire cloth." "Serious explosions have resulted in cases where the injury to the diaphragm has escaped notice, and gas generated at the negative electrode has been permitted to leak through the diaphragm" (into the anode compartment), "passing into the collecting chambers." The layer of sand operates to prevent the passage of hydrogen gas into the anode compartment, and protects the diaphragm against the corrosive action of the products set free in that compartment, which in practice has made frequent renewals necessary.—J. C. R.

*Improvement in the Process of Electro-plating, and Apparatus therefor.* W. P. Thompson, Liverpool. From C. M. Barber, Cleveland, Ohio, U.S.A. Eng. Pat. 13,795, July 17, 1894.

THIS is an arrangement by which the process of electro-plating is rendered automatic; it is intended primarily for the treatment of electric light carbons. A long endless belt provided externally with a series of clips placed close together, is rotated by and around sprocket wheels, one of which is in a sliding frame in order that the slack of the belt may be taken up if necessary. Near one end of the belt, a drum with longitudinal grooves is so rotated that the carbons, which fall into the grooves successively from a hopper, are brought in front of the clips and are pressed into them, each to each, by a cam action. The belt carries them forwards and, with the aid of guide wheels, dips them successively into a cleansing vat, and a series of plating and washing vats, and then carries them through a chamber, wherein they are dried by a current of air, heated or otherwise, and thence to a releasing arrangement in which they are automatically removed from the clips. The plating vats are of metal with anodes on either side, and carry above a bar of copper, connected with the negative pole of the generator, with which the metallic clips containing the carbons make sliding contact as they are conveyed past the vats. The guide-wheels are placed with horizontal axes, one between each pair of vats, in such a way that the belt, in passing over them, lifts the carbons over the sides of the vats.—W. G. M.

cleaning of the mill after each operation is most troublesome, especially in cases in which many samples have to be examined. The crushing mill described (see Fig.) is of such a size that after the grinding operation it may be readily inserted into the Soxhlet extractor and very small quantities of the seeds are therefore sufficient for an analysis. The mill consists of a strong tinned iron cylinder *a, a*, into which is inserted the perforated cone *b, b*,



held in position by the hinged lid *c*, which is also provided with the bayonet joint *g*. In the cone *b* rests a corresponding grooved cone *d*, the shaft of which is provided with the lid *i*, and the spiral spring *e*. The lid *i* is secured upon the cylinder by means of the bayonet joint *h*, and to the end of the shaft *f* the handle *K* is fitted. In using this mill a weighed quantity (about 5 grams.) of the seed is placed in the apparatus, lid *i* is closed and the handle attached to the shaft. By now slightly raising the grinding cone the seeds fall between the surfaces of *b* and *d*, and are thoroughly crushed by turning the handle *K*. The crushed seeds fall through the perforated cone into the lower part of the cylinder. The crushing being finished, lid and handle are removed, and, by giving the bottom lid a slight turn, perforations in it are made to correspond with perforations of the cylinder, providing openings for the solution of the oil to flow from the cylinder during extraction. The mill is then wrapped in filter paper, the top remaining uncovered, and the whole inserted into a Soxhlet apparatus for extraction. After extraction the mill is ready for another operation, the dry dust from the seeds having been removed by a brush.

—C. O. W.

*Report on Castor Oils from the Indian Section of the Imperial Institute.* W. H. Deering and Boverton Redwood. (Carried out for the Research Department of the Imperial Institute, September 18, 1894.)

FROM the collection of 78 castor oils in the Imperial Institute, we selected 23 for examination, viz.: the five grades of Pirpainti oil (as representing the inland products), the same of Kotapatam oil (from coast seeds), a No. 1 and No. 2, and five No. 3 grades of inland oil other than Pirpainti, and six "good second" inland and coast oils (other than Pirpainti and Kotapatam).

These oils have been examined as to their colour, specific gravity, viscosity, rotation of plane-polarised light, the amount of potash required to neutralise the free acid present in them and for their complete saponification, the amount of bromine which would combine with them, and their solubility in alcohol. The results are given in the subjoined table.

*Viscosity.*—This was determined by means of a Redwood viscosimeter.

*Rotation of Plane-polarised Light.*—A Hofmann-Laurent polarimeter was employed. D light, and 200 mm. of oil.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*Extraction Mill.* N. Lehmann. Chem. Zeit. 18, 1894, 412.

FOR the estimation of the percentage of oil in seeds by means of extraction it is necessary to have the seeds well crushed. This has been done by grinding them in variously constructed mills, but is in every case accompanied by the loss of a certain quantity of the oil, and the necessary

**Bromine Absorption.**—A solution of bromine in dry bisulphide of carbon was used, the oil being also dissolved in bisulphide of carbon. This was preferred to Von Hübl's iodine absorption method, in which a mixed reagent (iodine and mercuric chloride) and a mixed diluent (alcohol and chloroform) are used, and chlorido-products are formed; while in the bromine bisulphide of carbon method a more definite reagent is used, with formation of bromine addition-products only.

**Solubility in Alcohol.**—One volume of oil was shaken up with three volumes of alcohol of sp. gr. 0.830 (at 60° F.). All 23 oils were wholly soluble in the alcohol; they gave clear solutions, which remained clear after standing for a couple of days.

The specific gravity, time of efflux (viscosity), amount of potash required for saponification, amount of bromine absorbed (also refractive index as indicated by the oleo-refractometer reading), point to practical identity of composition of the oils examined. Triricinolein (the glyceride of which castor oil almost wholly consists) requires by calculation from the accepted formula 18.07 per cent. potash and 51.47 per cent. bromine; the calculated amounts required by triolein, tristearin, tripalmitin (frequent constituents of oils and fats) are shown below and on next page.

	Potash required for Saponification.	Bromine required to form Saturated Compound.
	Per Cent. KHO.	Per Cent. Br.
Triricinolein, $(C_{18}H_{34}O_2)_3.C_3H_5$ ..	18.07	51.47
Tristearin, $(C_{18}H_{36}O_2)_3.C_3H_5$ .....	18.92	0
Triolein, $(C_{18}H_{34}O_2)_3.C_3H_5$ .....	19.05	54.26
Tripalmitin, $(C_{16}H_{32}O_2)_3.C_3H_5$ ..	20.89	0

The estimation of the amount of bromine absorbed tends to be rather too high. The oils, as was to be expected, contain appreciable and varying amounts of free acid; the percentage of "potash required for free acid"  $\times 5$  gives the amount of free acid. The lower grades of oil contain more free acid than the higher ones; thus the No. 1 oils contain about 0.6 per cent. to 0.8 per cent., most of the "good second" oils about 2 per cent., most of the No. 3 oils about 5 per cent. of free acid. The Kotapatam oils (except the No. 1) contain less free acid than the Pirpainti oils.

As lubricants the oils examined should be equally good, judged by their viscosity (as indicated by times of efflux) alone; but the lower grades of oil would be likely to cause considerably greater corrosion of metal bearings on account of their greater acidity.

The depth of colour increases in proceeding from the higher to the lower grades of oil; colour has probably to a great extent been relied on in grading the oils.

The rotation of the plane of polarisation by castor oil is of scientific interest. The recorded observations are conflicting; thus, Flickiger and Hanbury ("Pharmacographia," p. 513) state that they found some samples of castor oil to be right-, others left-rotating. Allen observed no rotation. Popp found specific rotatory power of castor oil  $(a) = +12.15$ , but this was deduced from the rotation of 10 per cent. solutions of castor oil in alcohol, in which case errors of observation would be greatly multiplied up. Schädler gives the rotation of castor oil (apparently for 198 mm.) as  $+9.78$ , but gives 0.9163 at 15° C. as the specific gravity of the oil; which, although probably a misprint, lessens confidence in the statement. The rotation caused by 200 mm. of castor oil under before-mentioned conditions was pretty nearly the same in all 23 oils: a dextro-rotation of about  $8.5$ . The comparatively slight variations observed with so considerable a number of castor oils of varying origin justify the belief that rotatory power is a property of the glyceride ricinolein, and not of impurities or unknown constituents of the oil; the

amount of the latter must be small (castor oil gives about 96 per cent. of total fatty acids), and would need have a most unusually high rotatory power to produce the observed effect. The power (scarcely possessed at all by other natural glyceride oils) is probably connected with the fact that ricinoleic acid is an oxy-acid.

**Turkey-Red Oil.**—Castor oil, as is well known, finds important application in Turkey-red dyeing, being converted into Turkey-red oil by the action of strong sulphuric acid and subsequent neutralisation with alkali. From the general similarity in chemical composition of the oils examined, much difference in their behaviour with sulphuric acid was not anticipated; accordingly three oils, Nos. 1,549, 2,527, 1,564 (a first, second, and third grade oil respectively) only were experimented with. The rise of temperature on stirring 50 grms. of oil with 10 cc. pure strong sulphuric acid (sp. gr. 1.844 at 15° C.) was practically the same with the three oils—about 62° C.; 63°, 61°, 60.5° C. respectively.

100 grms. of each of the three oils were treated with 33 grms. of sulphuric acid, added in drops with constant stirring, the oil being cooled externally; the rise of temperature did not in any case exceed 17° C., and the evolution of sulphurous acid was very slight. The three products were left for a couple of days; they were then greenish-brown in colour, of treacly consistence, without appreciable difference in either respect. Each was then treated with 170 grms. of water at 40° C.; after separation of the water, the washed products were of light greenish-brown colour (No. 1,549 lightest coloured, No. 2,527 next lightest, No. 1,564 darkest), and of the consistence of castor oil. They were neutralised with caustic soda solution to the extent of four-fifths of the acidity present and then solution of ammonia added in moderate excess. All three gave clear liquids; No. 1,549 was of golden-yellow colour, No. 2,527 slightly browner, No. 1,564 was the brownest—a brownish-yellow, or light yellowish-brown.

**Purgative Principle.**—The purgative principle of castor oil has not yet been identified; Tison's "ricinine" is stated by himself to be non-purgative. The greater part of the active principle appears to be left in the pressed seeds; its isolation and examination, and the estimation of the amount present in the oil and the press-cake, would be desirable.

#### Meal of Sunflower Cake. R. Theodor. Chem. Zeit. 18, 531.

SUNFLOWER cake has been found, especially in Russia, one of the best auxiliary cattle-foods. As early as the year 1856 about 100,000 centners of sunflower oil (oil of the seeds of *Helianthus annuus*) were manufactured in Russia, and its amount has increased year by year, it being esteemed as a very palatable alimentary oil. The oil was formerly obtained by hydraulic means; the residual cake is harder than any other variety of oil-cake, and for this reason apparently it has not found a wider application. Denmark and the northern countries import large quantities annually, as do also the eastern provinces of Germany, and the problem of its disintegration has been successfully solved by several manufacturers there. It is still unknown in Southern and Western Germany; now, however, that it is put on the market in the form of meal, it will doubtless soon find general application, suited, as it is, both on account of its composition and pleasant taste, for fattening cattle. The percentage of proteid varies between about 30–44 per cent., the fat between about 9–18 per cent. It is possible to prepare two qualities, one rich in proteid and poor in fat, and the other rich in fat and poor in proteid. When, for example, the somewhat finely ground meal is sifted, employing a mesh of 1 mm., that which passes through is much richer in proteid and poorer in fat than the original, whilst the reverse is true of that which remains in the sieve.—A. R. L.



*Process for the Estimation of Small Quantities of Chlorine in Fats.* R. Bandedikt and H. Zikes. *Chem. Zeit.* 18, 649.

See under XXIII., page 984.

*Detection of Castor Oil in Olive Oil.* V. Di Vetere. *Schm.* 1894, 4, 48.

See under XXIII., page 981.

#### PATENTS.

*Improvements in Soap and Washing Compounds.* John Penfold Friend Field, London. Eng. Pat. 19,475, October 17, 1893.

This specification describes (1) a soap which will cleanse linen "without the need of rubbing"; (2) a washing powder; (3) a paste for cleaning silver, metals, paint, &c. The laundry soap is a mixture of ordinary soap, soda crystals, pipeclay, and benzine, with or without indigo or ultramarine blue. The washing powder is the same as the soap, minus the benzine, and reduced to a dry powder. The paste contains caustic soda in addition to the other ingredients, but no benzine or blue. There are four claims. The first is for the employment of the above-named materials, in about the proportions stated in the specification, in the manufacture of soap; the other three are for the particular combinations needed to produce each article.—L. A.

*Improvements in the Manufacture of Soap Compositions.* M. Baerlein, Salford, Lancashire. Eng. Pat. 19,732, October 20, 1893.

The inventor claims the manufacture, use, and application of an improved soap composition, "having very much greater cleansing and detergent properties than ordinary soaps," and produced by the mixture of soap with "benzol, toluol, xylol," solvent naphtha, "patent naphtha," or mixtures of the same.—L. A.

*Improvements in or applicable to Soap in Tablets, Cakes, or Bars.* Feodor Heilbronn, Kilbarn, London. Eng. Pat. 13,104, July 6, 1894.

The improvements consist in coating the soap with a thin and preferably transparent film of gelatin, wax, spermaceti, ozokerite, or other suitable material, in order to preserve the odour, appearance, colour, &c., of the soap for an indefinite period. The coating may also be made thick, if desired, as for exhibiting soap in shop windows.—L. A.

*Improvements in Apparatus for extracting Suint from Wool and other Substances.* Emile Richard-Lagerie, Roubaix. Eng. Pat. 13,672, July 16, 1894.

IMPROVEMENTS upon Pat. 12,433, 1893 (this Journal, 1893, 1044), whereby the distribution of the liquors according to the graduated concentration of the extracts, into the different compartments of the tank or their equivalents, and the discharge of the said compartments upon the wool in the required succession, are effected automatically, instead of requiring the intervention of a workman as hitherto.

The distribution and discharge of the liquors are brought about either by utilising the change of level of the liquors by means of floats during filling and emptying, which actuate mechanism to effect same, or by making use of the rise and fall of the hydrometer according to the concentration of the liquors.

The improved apparatus is applicable for extraction and concentration of other substances besides suint; for instance, sugar from sugar cane or beet, &c.—W. R.

### XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

#### (A.)—PIGMENTS, PAINTS.

##### PATENTS.

*An Improved Process for the Production of Iron Oxide for Use as a Pigment.* C. H. M. Lyte, London. Eng. Pat. 16,638, September 4, 1893.

An ore of iron, such as burnt pyrites or the "purple ore" from the wet copper extraction process, is heated with caustic potash or soda until the mixture becomes converted into a ferrite or ferrate. It is then decomposed by boiling with water, and the red oxide washed and ground, the alkali being used again. Insoluble matter may be removed by treating the ferrate with cold water and filtering before decomposition.—F. H. L.

*A New or Improved Binding Medium for Size Colours, and Method of Manufacturing the same.* J. Clasen, Hamburg. Eng. Pat. 13,004, July 4, 1894.

HERETOFORE ordinary size has been rendered fit for distemper work by the addition of an extract of Carrageen moss, which, however, deteriorates the binding and keeping qualities of the size. The improved size is made as follows:—Ordinary size or glue, selected so as to be as rich in fat as possible, is soaked for about 14 hours in cold water so that it may become soft, and is then transferred to a boiler, where it is heated until the whole contents become a thick viscid liquid; about 2 parts of water to 1 part of size should be used. The liquid size is then tested to ascertain its richness in fat by leaving it for about 30 minutes at 90° C., whereupon the fat will have risen to the surface. Cocoa-nut oil or stearine oil is then added in a quantity inversely proportional to the content of fat in the size, namely, from 3 parts by weight of oil to 300 parts of size, for a size rich in fat, to six parts of oil to 300 parts of size, for one poor in fat. When the whole has been thoroughly mixed and cooled to about 50° C., 60 to 70 parts by weight of sago meal or potato meal are added by degrees with constant stirring; this addition supplies the acnelaginous matter to replace the Carrageen moss. The liquid mass is poured into moulds and allowed to set; a short drying serves to render the tablets ready for sale. In using the tablets they are soaked and boiled with water, and the solution added to the colour mixed with water. The surface to be painted does not require the usual preliminary soaping.—A. G. B.

#### (B.)—RESINS, VARNISHES.

##### PATENTS.

*An Improved Material or Compound adapted for Use as a Varnish, as a Substitute for India-Rubber, Gutta-Percha, and like Substances, and for other Purposes, and the Process for its Production.* C. V. King, and R. V. Jellicoe, London. Eng. Pat. 15,686, August 18, 1893.

This material consists of collodion cotton made in the usual manner, and the solvent is prepared by dehydrating methylated spirit by quick-lime or potassium carbonate, mixing it, after distillation, with 10 to 50 per cent. of methylated ether or hydrocarbon, such as benzene. The addition of castor oil and gum copal to increase flexibility, and of pigments or dyes to alter the colour, is also claimed.

—F. H. L.

*A New or Improved Composition for Coating Iron and Steel.* T. W. Elliott, Newcastle-on-Tyne. Eng. Pat. 16,800, September 7, 1893.

"VULCAN cement" is mixed with raw or boiled linseed oil, or preferably "patent boiled oil," with or without the addition of turpentine or benzoline (petroleum spirit).

—F. H. L.

*Improved Preservative Coating for Iron and Steel.*  
W. M. Walters and F. R. Stone, Liverpool. Eng. Pat.  
20,253, October 27, 1893.

A SOLUTION of tannic acid in water of about 10 per cent. strength is mixed with a 5 per cent. solution of gum arabic or dextrin, and the metal painted; ordinary paints or varnishes being afterwards applied as desired.—F. H. L.

### (C.)—INDIA-RUBBER, &c.

*The Analysis of India-Rubber Articles.* C. A. Lobry de Bruyn and F. H. van Leent. Chem. Zeit. **18**, 1894, 309.

THE determination of the specific gravity of india-rubber articles is of very little use, although it may be said that india-rubber which will float on water is free from mineral admixtures. Fatty oils and paraffin wax may be estimated in india-rubber articles by extracting them with turpentine, carbon bisulphide, or benzine (petroleum spirit), as suggested by Heinzerling, but the results are liable to be

vitiated by the presence of native resins in the india-rubber. Donath estimates in india-rubber articles only the specific gravity, ash, and percentage of sulphur. His opinion, that the value of an india-rubber article mainly depends on the quantity of india-rubber and the proportion of sulphur of vulcanisation present, altogether neglects the influence of different qualities of india-rubber, as well as the fact that articles manufactured from the same india-rubber may contain 3, or as much as 6, per cent. of sulphur without showing any difference in quality. Henriques' method for the analysis of india-rubber articles is too detailed and and elaborate for ordinary purposes, although it may be very useful in special cases. For ordinary purposes, the estimation of the mineral matters by ignition, and the estimation of the constituents soluble in alcoholic soda are sufficient, but it is useful to submit the samples, in addition to these chemical tests, to a dry-heat test for two hours at 135° C., and to a moist-heat test for four hours at 170° C., observing the general behaviour of the samples in these tests as well as their loss of weight. In this manner the following samples were examined:—

No.	Description of Sample.	Ash.	Per Cent. soluble in 6 per Cent. Alcoholic Soda.	Calculated upon Substance free from Ash.	Heated to 135° C. for		
					One Hour.	Two Hours.	Three Hours.
		Per Cent.			Loss of Weight, 0.1	Loss of Weight, ..	Loss of Weight, 0.25
1	Para rubber vulcanised with 6.4 per cent. S.	..	..	5.0	..	..	..
2	Mozambique rubber vulcanised with 5.5 per cent. S.	..	..	6.5	0.2	..	1.3
3	Borneo rubber vulcanised with 8.3 per cent. S.	..	..	36.0	1.7	..	3.6
4	Sheet .....	3-4	..	33.4	1.4	..	3.1
5	" .....	3-4	..	20.4	1.4	..	3.5
7	" .....	3-4	..	30.2	1.4	..	3.1
11	Sheet, 15 years old, 5.5 per cent. S.	..	..	..	1.7	3.1	..
13	Red sheet .....	..	..	5.8	..	1.2	..
15	Black ring .....	6.5	..	0.7	1.1	2.5	..
16	Hood .....	13.5	9.6	11.1	..	1.6	..
18	Ring .....	15.5	19.7	23.3	..	3.0	..
21	Ordnance break ring .....	19.5	2.6	3.3	0.11	..	0.4
24	Sheet, 15 years old, 3.7 per cent. S.	33.0	12.9	19.2	0.1	..	0.7
25	Floor mat .....	41.0	28.0	50.0	..	..	0.4
47	Sheet (sample) .....	57.0	4.1	9.0	..	1.7	..
53	Hose .....	63.5	1.3	1.0	..	1.2	..

Pure vulcanised Para and Mozambique rubber stands the heat tests very well, losing only from 0.15 to 0.25 per cent. in weight, while most of the other samples show much greater losses. Sample No. 21, which contains 20 per cent. of zinc-white, stood many years' wear very well. It is probably made of Para rubber, but yields no extract to alcoholic soda. White and brown rubber substitutes, on being heated for two hours to 135° C., lose from 2 to 2.5 per cent. in weight, but neither do they melt nor are they otherwise impaired in their quality. In the wet test they melt, become discoloured, and decompose. Rubber articles containing rubber substitute do not stand the moist-heat test. The quantity of resinous matters contained in india-rubber is without perceptible influence upon the result of the heat tests, but it seems that in this respect age has a detrimental effect upon india-rubber articles. The percentage of resin in an india-rubber seems to be without influence upon the durability of the articles manufactured from it.

—C. O. W.

*Analytical Methods for the Valuation of India-Rubber Goods.* C. A. Lobry de Bruyn. Chem. Zeit. **13**, 1894, 329.

See under XXIII., page 956.

*Contributions to the Analysis of India-Rubber Articles, III.* R. Henriques. Chem. Zeit. **18**, 1894, 411.

See under XXIII., page 956.

*Analytical Control of the Vulcanisation of India-Rubber.* R. Henriques. Chem. Zeit. **18**, 1894, 701.

See under XXIII., page 957.

*Contributions to the Analysis of India-Rubber Articles.* R. Henriques. Chem. Zeit. **18**, 1894, 905.

See under XXIII., page 957.

## PATENTS.

*A New Substat for India-Rubber and Gutta-Percha.* L. Le Brocquy, Dublin. Eng. Pat. 19,163, October 12, 1893. This consists of the substance known as printers' "roller composition"—consisting of glue, glycerin, and sugar—encased in a covering of ordinary india-rubber, to protect it from damp and mechanical injury. Into the composition may be introduced various substances such as tannic or chromic acid to raise its melting point, salicylic acid as a preservative, ground cork, china clay, and barium sulphate, and mica to increase its insulating capacity.—F. H. L.

*Improvements in Decolourising India-Rubber.* M. Gerber, Clermont-Ferrand, France. Eng. Pat. 19,284, October 13, 1893.

THE vulcanised rubber, containing from 5 to 8 per cent. of sulphur is heated under a pressure of three or four atmospheres with constant agitation in the presence of three and a half times its weight of toluene and one-fourth of tin. Instead of the tin, any metal or mixture of metals combining with sulphur may be employed, and the toluene may be replaced by any other indifferent solvent. The heating is continued for eight hours, when the mixture is poured off and allowed to settle.—F. H. L.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

*The Action of Tannin on Hides.* J. Pässler. Chem. Zeit. 1894, 18, 363.

HANAUER has recently resuscitated this question (Chem. Zeit. 1894, 18, 288), and Procter has replied (this Journal, 1894, 747). It appears that the dictum that tannin is incapable of tanning hides originated with Wagner, who classified tan-stuffs into physiological and pathological, denying the latter kind any function in the tanning process. Physiological tan-stuffs are, according to Wagner, such as are products of normal metabolism in the plant, and include the tanning-principles of oak-bark, pine-bark, willow-bark, valonia, myrabolans, divi-divi, algarobilla, canaigre, sumach, and of other tanning materials. Pathological tannins, on the other hand, are products of morbid metabolism in the plants, especially in those of the species *quercus*; they are generally formed as a result of the punctures of insects. Such tannins are contained in gall nuts, knopfern, and rove, and may be regarded as tannin proper, closely allied to one and the same chemical individual. Physiological tannins, on the other hand, are accompanied by other substances from which it is difficult to isolate the pure tannins.

The author tanned a sheep-skin with pure tannin and found that the product resembled leather in every respect, save that it lacked firmness and was not full. This failing has, however, nothing to do with the tannin, but is merely a result of the absence of acid in the liquor. In the ordinary tanning process the saccharine constituents of the bark or other tanstuff invariably undergo lactic and acetic fermentation, so that acids are never absent from the liquor even in "sweet-tanning." It is these acids, says the author, which impart the necessary fulness and firmness to the leather. A calf-skin which was tanned in pine extract under conditions which precluded the formation of acids, suffered from the same defects as the sheepskin tanned with pure tannin. When a pure tannin or a pathological tannin is used in conjunction with an ordinary tanstuff which contains acid-yielding constituents, a marketable leather is produced; indeed, pure tannin with an appropriate addition of lactic and acetic acids will be equally effective.

Pathological tannins are sometimes used in practice, as, for instance, that in knopfern galls which are formed

on the cups of the oak from the puncture of *cynips quercus calicis*. Rove, or Bassorah galls or apple of Sodom, are the galls produced by the puncture of a gall-wasp on the young shoots of a dwarf oak of Asia Minor. These galls are considerably larger than the usual oak-apples, and contain a high percentage of tannin; they come into the market in a crushed condition, and yield a liquor of excellent strength, the tannin being easily soluble in cold water. The common oak-apple and Chinese galls are forms of tannin too expensive for the tanner.—A. G. B.

## PATENTS.

*Improvements relating to the Manufacture of a Substitute for Leather.* H. H. Lake, London. From G. Sachsenröder, Barmen, Germany. Eng. Pat. 15,937 August 23, 1893.

ORDINARY vegetable parchment, or a combination of any suitable fabric with parchment paper manufactured according to known processes, is suitably dyed, imperfectly dried, and then conducted through a hot bath of fats, paraffin oil, wax, stearin, or the like. To promote the impregnation of the material by the fatty matter the fabric may be passed through rollers. After having been allowed to dry in the air and polished, the substitute is ready for use. Greater suppleness may be imparted to this artificial leather. It may be impregnated with some hygroscopic substance, such as glycerin or calcium chloride; in certain cases glue, casein, dextrin, &c. may be used as "covering substances" to diminish the unctuousness of the product.—A. G. B.

*An Improved Process for Colouring Tanned Hides by Means of Smoke.* A. Herrmann, Schweidnitz, Silesia. Eng. Pat. 13,414, July 11, 1894.

THE white tanned or bleached tanned hides are stretched over hurdles in a closed chamber, on the floor of which is a flat hearth designed to hold a smoking fire, the fuel being horse-dung, with or without straw. Only the grain of the leather becomes coloured by the smoke, the shade of colour depending on the duration of the process, and varying from dull light yellow to golden brown. The colour is durable and the hide remains soft.—A. G. B.

## XV.—MANURES, Etc.

*Examination of Artificial Manures.* Chem. Zeit. 1894, 18, 361.

See under XXXI., page 980.

*Detection of Adulterants in Basic Slag.* O. Böttcher. Chem. Zeit. 1894, 18, 565.

See under XXXI., page 978.

## PATENT.

*Improvements in Making Manure from Sewage and Sewage Sludge.* J. Carter, London. Eng. Pat. 22,559, November 24, 1893.

THE inventor describes in detail in this specification a process which chiefly consists in covering the drained sewage or sludge with dry ash dust, and when dry mixing by means of a steam harrow travelling on rails. Other materials besides dry ash dust may be used, such as powdered chalk, burnt clay; also soda, soot, resin, tar, and carbolic acid may be added. The whole process is carried on under a roof of corrugated iron or glass.—J. C. C.

## XVI.—SUGAR, STARCH, GUM, Etc.

*Calculation of the Rendement of Raw Sugars.* Seyffert. *Zeits. Zuckerind.* **44**, 399.

INSTEAD of the non-sugar rendement which was introduced about a year ago, and which is arrived at by deducting  $2\frac{1}{2}$  times the total non-sugar from the polarisation, the author proposes the following. He calculates, from the sugar analyses made during the past year, that the ratio of the percentage of organic matter  $O$  to the difference  $100 - P$  ( $P$  = polarisation) is almost constant, and is equal to 3 in those sugars polarising from 94 to 97. So that since  $\frac{100 - P}{O} = 3$ , it follows that  $O = \frac{100 - P}{3}$ , which latter represents therefore the normal percentage of organic matter for the particular raw sugars referred to. The author then proposes to multiply the difference between this calculated percentage of organic matter and that actually found by analysis by 2, and to deduct or add as the case may be this product from the old rendement ( $P - \text{Ash} \times 5$ ). The calculations made by the author show that the mean values are the same as those obtained from the ash rendement; they are also said to be surer and more concordant than the non-sugar rendement.—A. R. L.

*Influence of Calcium Salts on the Yield of Sugar from Raw Sugar Masseccites.* L. Szyfer. *Gaz. cukr.* **1**, 541.

ALTHOUGH the amount of lime in the masseccites examined in the experiments described in this paper, varied between 0.015 and 0.086 per cent., the yield of sugar was found to undergo no great alteration. This confirms Herzfeld's conclusion that the presence of small quantities of calcium salts does not hinder the crystallisation of the sugar.

—A. R. L.

*So-called Scum Fermentation.* *Zeits. Zuckerind.* **44**, 611.

HERZFELD states that he cannot confirm the observations of Prinsen Geerligs (this Journal, 1894, 823) as to the ready decomposability of free glucinic acid; he does not agree with the views of Prinsen Geerligs regarding the cause of scum fermentation in alkaline liquors.

Classen thinks that derivatives of invert sugar have but little to do with scum fermentation, and that the phenomenon is brought about principally by unknown organic compounds. He believes that defecation at a temperature not exceeding 69°–75° C. (Prinsen Geerligs, *loc. cit.*) is of no service, but recommends maintaining a sufficient decided alkalinity, and drawing off the liquors at not too high a temperature. The extensive laboratory experiments of Weiland show that cold defecation has in no respect advantages over hot.—A. R. L.

*Decomposition of Sugar during Evaporation.* Lexa. *Zeits. Zuckerind. in Böh.* **18**, 503.

WHEN sound, alkaline liquor is evaporated within a short time, the decomposition of the sugar is slight—perhaps not more than 0.1 per cent. The effect of high temperature is small, inasmuch as it shortens the time necessary to perform a given amount of work; the temperature in the last member of the multiple effect evaporator ought not, however, to exceed 64° C., and that in the first member should not be above 112° C. Such temperatures as 120°, 128°, and even 134°, which are ordinarily adopted in Germany, are too high and should not be used. Greater losses are occasioned by boiling than by evaporating, but exact data as to the magnitude of these losses are wanting; the quotients give no information whatever in these cases. Sugar is decomposed by heating the juices prior to evaporating them, and indeed such losses amount to 0.3–0.4 per cent.—A. R. L.

*Loss of Sugar in Manufacture.* Feldmann. *Zeits. Zuckerind. in Böh.* **18**, 468.

ACCORDING to the author the total loss of sugar during its manufacture from beetroots, up to the boiling of the masseccite, amounts to 0.7 to 1 per cent. on the roots. Of this, however, when the process is carefully controlled, only 0.3 to 0.5 per cent. can be accounted for. The unknown losses are principally due to the decomposition of sugar during the boiling and evaporating of the liquor, and are greatly influenced by the construction and management of the apparatus, as well as by the temperature, concentration, condition, and alkalinity of the liquor. Herzfeld's exact laboratory experiments are not of direct service to the industry, because in the latter the decomposition of sugar by boiling, must be greater than that observed in the laboratory.—A. R. L.

*Defecation and Filtration of Diffusion Liquor.* Mittelmann. *Bull. de l'Assoc. Chim.* **11**, 707.

THE author recommends defecation with caustic lime, and careful mechanical filtration of the crude juice, for which purpose Bouvier's apparatus, which renders it possible to filter the hot juice rapidly, is serviceable. He points out the great advantages to the industry accruing from such a mode of procedure.—A. R. L.

*Defecation and Saturation.* Beaudet. *Bull. de l'Assoc. Chim.* **11**, 658.

FROM the results of a detailed investigation carried out by the author, it would seem that defecation with caustic lime leads to the production of liquors containing less inorganic matter than those in which defecation with milk of lime or powdered slaked lime, has been adopted; the products are the better the lower the temperature of defecation and saturation. The practical application of these results is, however, beset with difficulties, and it is scarcely possible to introduce specific improvements which can be equally carried out everywhere. On this account it is necessary to prosecute exact investigations in every single factory; data of this kind are at present wanting even concerning the principal points.—A. R. L.

*Improvements in the Beetroot Sugar Industry.* Jarkovsky. *Zeits. Zuckerind. in Böh.* **18**, 436.

THE use of divided (short) diffusion batteries has not proved satisfactory in practice. The so-called Heckmann's diffusion (vacuum) process has been in use for many years in Austria, as has also the process of heating the beetroot slices previous to diffusion. Among new evaporation processes that of Classen gives the best results, and can be thoroughly recommended. The Karlik-Ehrmann rotation system has a promising future, but needs perfection at the hands of practical men.—A. R. L.

*The Drying of Potato Pulp with Molasses for Cattle-Food in North German Factories.* M. Dellbrück. *Zeit. f. Spiritusind.* **1894**, **17**, 141.

THE pulp is mixed with milk of lime and passed through filter-presses; the apparently dry mass is then carried along by a screw, which mixes it with a thin stream of molasses. On admixture it does not cake, but forms a crumbly mass, since molasses only contains about 20 per cent. of water. The mixture, after drying in the oven, is mechanically carried, along with a strong current of air, through a trough in which a reverse-acting screw arrests any undried lumps, the finer particles being carried forward ("Cyclone drying apparatus"). The air, which is then about 70° C., passes to the dust collector, leaving the dried stuff to be milled and compressed.

This method for utilising molasses is applicable:—1st. In sugar factories, where it can be mixed and dried with the root cuttings; 2, in places where the strontia recovery



process is worked: 3. in distilleries using molasses; and 4. in starch factories. The compressed material it is proposed to use as a nutritious cattle-food.—T. A. G.

*Quantitative Estimation of Carbohydrates.* E. Schulze. Chem. Zeit. 18, 527—528.

See under XXIII., page 985.

#### PATENT.

*Process for Purifying and Decolorising Saccharine Juices and Sugar Solutions generally.* C. Steffen, Vienna, Austria. Eng. Pat. 17,632, September 19, 1893.

THIS is a process for obtaining colourless sugar solution as pure as possible, and crystalline sugar therefrom, by treating the juices of beetroot and cane sugar manufacture, subsequently to defecating them, and in some cases after they have been saturated and freed from the defecation and saturation scum according to known methods, and cooled down to a temperature below that at which defecation was effected, i.e., below 60° C., and preferably between 30° C. and 40° C., with sulphurous acid in such quantities that the colouring and non-sugar substances are for the most part converted into sulphite compounds; these latter remain in solution in the acid juices, which are then subjected at the above-mentioned temperature to the action of proportionately small quantities of animal charcoal for the purpose of removing the sulphite compounds of the non-sugar substances. The free sulphurous acid is then got rid of by treating the filtered juices with the necessary quantity of lime, baryta, strontia, alumina, &c. The juices after being freed from the sulphites so precipitated are slightly alkaline or neutral, and free from colour and impurities. The application of this process to medium and thick juices of any degree of concentration, as also to all syrups of sugar manufacture, the treatment being repeated as often as necessary, and to the treatment of the clairee or fine liquors of beetroot and cane sugar refineries, and the syrups thereof, is also claimed.—A. R. L.

### XVII.—BREWING, WINES, SPIRITS, Etc.

*Estimation of Starch by Alcoholic Fermentation.* Albert Munsche. Zeits. für Spiritusind. 1894, 17, 202; 17, 209.

See under XXIII., page 985.

*Hop-Tannin.* M. Hayduck and F. Goldiner. Wochenschr. f. Brauerei, 11, 409—415.

ETTI showed (J. Chem. Soc. 1876, i, 927) that in the tannin from hops there is a tannin,  $C_{15}H_{25}O_{13}$ , and a phlobaphen of the probable formula  $C_{30}H_{46}O_{25}$ , and which he considered as probably formed by the coalescence of two molecules of the tannin with the separation of water. The authors have re-examined these substances, and studied their properties from a brewing point of view.

After extraction of the hops with ether and then with absolute alcohol, in both of which the tannin is insoluble, the tannin was extracted by repeated digestion with 70 per cent. alcohol. The alcoholic extract was then fractionally precipitated by means of a solution of lead acetate in alcohol of similar strength. The greater part of the phlobaphen was in the first brown precipitates, that of the tannin in the latter yellow precipitates. The precipitates suspended in water were treated with hydrogen sulphide, and the lead sulphide (which carried down with it most of the tannin, or phlobaphen) was extracted with 70 per cent. alcohol. The separation was completed by means of ethylic acetate, as the phlobaphen is insoluble, the tannic acid soluble, in that solvent.

The hop-tannin so obtained is a light brown amorphous powder, soluble in water, dilute alcohol, and ethyl acetate, very sparingly soluble in absolute alcohol, and insoluble in ether. A 0.2 per cent. solution in water was slightly yellow, and showed a green fluorescence. Ferric chloride produced an intense green coloration, but no precipitate: the reaction is very delicate. This tannin has the characteristics of an acid, slightly reddens blue litmus, precipitates dissolved albumin but incompletely, as the precipitate is slightly soluble in water, and (contrary to the statement of Etti) is precipitated by animal skin. It is very unstable, and, even on evaporation on the water-bath, a part is converted into an insoluble substance, mostly phlobaphen. When an alkaline carbonate is added to the solution, the latter takes a dark brown colour, and on evaporation on the water-bath the whole of the tannin is then converted into phlobaphen. The same conversion takes place rapidly if the dry tannin is heated at 140° C.

The phlobaphen forms a reddish-brown powder, partly soluble and partly insoluble in boiling water or dilute alcohol. The yellow solution gives, with ferric chloride, a dirty green precipitate: soluble albumin is precipitated by it in an insoluble form, and it is itself precipitated by animal skin. Heated at 130°, phlobaphen becomes insoluble, probably by condensation by loss of water, and this condensation takes place, to some extent, at lower temperatures, as shown by the presence of insoluble material in the phlobaphen prepared as described. It is soluble in alkaline carbonates to a brown liquid.

Both phlobaphen and tannin and their compounds with iron are slowly decomposed by continued boiling or heating.

*Estimation of Tannin in Hops.*—The author has found Schröder's modification of Löwenthal's method, in which the tannin extract is titrated with permanganate both before and after the precipitation of the tannin by skin, the best for estimating the tannin in hops. The air-dried hops are coarsely ground, then extracted six times by boiling each time with 100 cc. water in a Schröder's boiler. This latter consists of an exactly cylindrical tin vessel, into which is fitted, as a closely but evenly moving piston, a perforated plate, covered with fine wire gauze, and fitted with a handle. Ten grms. of the hops and 100 cc. water are introduced into the boiler, and the piston then inserted. The boiler is put into a water-bath at 100° for an hour, then removed, the piston pressed down slowly and firmly, and the solution, which is thus separated from the hops, poured off. A second 100 cc. of water is then added, and the heating repeated, but now for half an hour each time. The sixth extract, which only contains negligible quantities of tannin, is used to make up the bulk of the first five extractions to 500 cc. About 20 cc. of this extract is taken, 5 grms. of powdered and well-soaked skin (of which the percentage of moisture is known) added, and the whole well and often shaken up and allowed to remain for 24 hours. The skin is then filtered off, 10 cc. of a dilute indigo solution added, the whole diluted with water to about three-quarters of a litre, and titrated with permanganate. The colour, which is first blue, passes into green, and then suddenly, when the extracts and indigo are oxidised, to a bright golden yellow. The end point is very sharply indicated. Correction must be made for the moisture of the skin and the permanganate required by the indigo used as indicator. A similar titration of hop extract which has not been treated with skin is made, and the difference gives the quantity of tannin. The permanganate is standardised against a solution of pure tannin. Special trials proved that the resin present in hops does not affect this reaction, so that extraction of the hops with ether is unnecessary and best omitted, as the long digestion with ether probably decomposes some of the tannin. The same method may be used for estimating the tannin in beers, after boiling off the alcohol, but the quantity of tannin present is so small that the influence of experimental error becomes very large. The following numbers were obtained with some beers:—English ale, 0.024 per cent.; Pilsen beer, 0.018 per cent.; Munich beer, 0.012; Berlin beer, 0.012.

**Influence of Hop-Tannin on Beer.**—The phlobaphen, as already mentioned, precipitates albumin in an insoluble form. The hop-tannin, however, forms a compound soluble in boiling water, but almost insoluble in cold water. This is seen in the wort, after treatment with hops, becoming thick on cooling. The cool and cleared wort still contains some of the albumin-tannin compound, and, though more of this is precipitated during fermentation, a trace still remains in the finished beer. The influence of hop-tannin is thus to remove a part of the very unstable soluble albumin from the wort, and indirectly to act as a preservative; it has, however, no direct antiseptic properties. The bitter hop-resins, on the contrary, have a decided antiseptic power. The influence of the hop-tannin is shown in the much smaller quantity (mere traces) of soluble albumin present in strongly hopped English ale, than in the much less hopped German beers. Analyses of a number of hops for tannin also showed that the hops generally considered the best are richest in hop-tannin. The following numbers were obtained, calculated on the dry hops:—Saaz, 2.9 per cent.; Spalt, 2.25 per cent.; Neutomschel, 1.69 per cent.; Wolnzach, 1.66 per cent.; Mainburg, 1.44 per cent.—L. T. T.

**The Carbohydrates Pre-existing in Malt.** E. Jalowetz. Chem. Zeit. Rep. 18, 39.

THE author has previously stated that the carbohydrates present in malt are saccharose, maltose, dextrose, and dextrin. He now comes to the following conclusions:—(1.) Almost all the carbohydrates present can be extracted from 50 grms. of malt by treatment, six or seven times repeated, with 300 cc. of 90 per cent. alcohol, or still more completely by a two hours' hot extraction with 90 per cent. alcohol, followed by one hour's digestion with water of the malt freed from the alcohol and fat of the extraction-liquid and dried. The carbohydrates are saccharose, dextrose, levulose, and an unfermentable substance identical in rotatory and reducing properties, with maltose. (2.) By digestion of the alcohol-extracted malt with water at the ordinary temperature for one hour, followed by precipitation of the aqueous extract with alcohol, a body is obtained, levo-rotatory, not reducing Fehling's solution, and giving a violet coloration with iodine solution. This may contain the already known levo-rotatory gum of malt, with perhaps dextrin, either originally present or formed during the extraction with alcohol. (3.) By digestion of malt with water at 18°–20° C. for one hour a solution results containing the carbohydrates, together with certain transformation products of starch, of which maltose, dextrin, and dextrose can be recognised. (4.) Digestion for several hours with water at 18°–20° C. brings about inversion of the saccharose; after three hours about half of the saccharose is inverted. (5.) The amount of saccharose in (a) the alcoholic extract, (b) the aqueous extract after one hour at 20° C., and (c) the wort obtained in the usual manner in the laboratory, is almost constant, on the average 3.2 per cent. (malt containing 10 per cent. of water). (6.) The reducing carbohydrates present are dextrose (3.1 per cent.), levulose (1 per cent.), and maltose (0.7 per cent.). It is not established whether dextrin is originally present or formed during extraction. (7.) Iso-maltose is not present either in the alcoholic or the aqueous extract. (8.) The starch of the malt grain is so changed by the growth of the barley, that the breaking-down of the starch has already taken place. It is not possible to identify the results of the action of cold malt extracts or diastase solutions on starch, brought about in a mechanical way, with the results of the mashing process.

Two other ferments besides diastase are present in malt, an inverting and a dextrose-forming ferment (glycose).

—R. B. B.

**Note on the so-called "Gluten Particles" occurring in Wort, Beer, and Yeast.** H. Will. Wochenschr. f. Brauerei, 1894, 937–938.

IT is well known that bright mash-tun or copper wort becomes turbid on cooling owing to the separation of minute, highly refractive particles of nitrogenous matter.

Although a portion is deposited on the coolers, much of this gluten remains in suspension in the wort and passes partly into the yeast, partly into the new beer, from which it only separates during storage. Nevertheless, even finished beer frequently contains gluten particles; and the deposits formed in pasteurised beer, according to the author, often consist wholly of these particles in a more or less shrivelled or otherwise changed condition. Microscopically, the gluten particles separated from wort are perfectly homogeneous, and up to 2 $\mu$  in diameter. But in yeast and also stored beer they present a vacuolated appearance and resemble air-bubbles. In beer they also frequently occur in the form of minute granules. It is evident, therefore, that the gluten particles suffer some change during the process of brewing. The author has, therefore, investigated the behaviour of the gluten particles (obtained from frozen beer) towards various solvents, with the following results:—

In contact with water the contents of the particles become swollen, and dissolve slowly—more rapidly on warming—the outer membrane being left behind. Absolute alcohol exerts no solvent action, but alcohol of 65 per cent. rapidly dissolves the cell-contents. In concentrated hydrochloric acid the gluten dissolves almost completely to an intense cherry-red solution, from which, by the addition of water, a violet-red precipitate is separated. Dilute acids in general readily dissolve the contents of the particles, whilst stronger acids, and also alkaline solutions, dissolve the outer envelope as well. From the above and other considerations the author concludes that the bulk of the cell-contents of the gluten particles consists of mucidin.

Beers suffering from gluten turbidity either contain in suspension extremely minute flocculent particles, or, more frequently, possess an opalescent appearance. In the latter case, the turbidity almost entirely disappears on warming the beer to 30°–40° C., but reappears on cooling. The addition of alcohol has a similar brightening influence.

—H. T. P.

**The Influence of the Mashing Process on Attenuation.** Windisch. Wochenschr. f. Brauerei, 1894, 885.

IT is well known that the conditions of mashing largely influence the degree of attenuation of beer. In this connection the author relates an interesting practical experience. The worts in a certain brewery were found to attenuate insufficiently. In order to increase their fermentability, the mashing heats were lowered and the time of standing increased, the modified process being applied to two different malts, the one cured at 45° R., the other at 65° R. The result was that the degree of attenuation increased by 11 per cent. in the case of the low-cured malt, but remained absolutely stationary in the second instance. This peculiarity may possibly be explained on the assumption that the diastase of the second malt was so greatly restricted by the high curing heat, that the variation in the mashing process was insufficient to sensibly affect the type of extract produced.—H. T. P.

**The Treatment of Boiling (Foaming) Fermentations.**

A. Hesse. Zeits. für Spiritusind. 1894, 17, 153.

DURING 1885 the author made no less than 1,068 mashes, not one of which showed boiling fermentation. In the year following, the ordinary mash tun was replaced by an Eckert's centrifugal machine, and boiling fermentations began. These were at the time wrongly attributed to the new machine. On reverting again to the old process of mashing, the "boiling" ceased, but inferior fermentations resulted. Whilst in Hellefreund's old method the malt is all mixed at once with the whole of the potato, in the centrifugal machine the mixing is done in several portions, so that much more complete saccharification results, and to this cause the boiling fermentation is due.

The following method of procedure was adopted:—

One-third of the malt was added at the beginning of the mash, and the whole of the potatoes blown out of the vessel at a finishing temperature of 49° R. The rest of the malt was then added, and the mash cooled. A marked decrease of boiling fermentation resulted. Finding that the addition of malt to the cold mash caused no bacterial acidity

the plan was adopted of reserving part of the malt for addition at this stage. In extreme cases the malt should be added at a temperature as low as 14° R. At a temperature of 30° R. moderately vigorous boiling can be suppressed.

Boiling fermentation begins when the normal relation of maltose to dextrin exists in the mash: when this relation is upset frothing or foaming does not take place. Boiling fermentation is the normal form with good yeast and a good mash tun.—T. A. G.

*Boiling Fermentations: A Criticism of the Results obtained by A. Hesse (see preceding abstract).* G. Heinzelmann. Zeits. für Spiritusind. 1894, 17, 161.

THE author has tabulated a series of experiments conducted under different conditions, and gives the saccharometer readings at different stages of fermentation, the alcohol percentage, the petroleum required for sprinkling (to allay froth or boiling fermentation), the amount of malt used, and the result with respect to boiling fermentations. In cases where the whole of the malt was used at mashing he did not always obtain boiling fermentation, nor was it always prevented by reserving a portion for addition at a later stage. He supports the contention of Hesse that the addition of malt to the cold mash has no undesirable action on the fermentation, and does not favour acidity. The amount of malt to be used depends upon the kind of potato used, and must be determined for each kind. Thick mashes are recommended as a general means of avoiding "boiling," since diastatic action is not so intense as with thin mashes. When a small quantity of malt is used for the mash, a restricted saccharification results, and the maltose molecule is not so readily fermented, because surrounded by dextrin molecules. Consequently fermentation proceeds more regularly, and on adding malt to the cold mash, dextrin is gradually degraded to maltose and then fermented. Malt extract made by soaking malt for three hours in water, and filtering, does not give as good results as the dry malt itself.—T. A. G.

*Boiling Fermentations.* Zeits. für Spiritusind. 1894, 17, 225 (compare preceding abstracts).

A SERIES of experiments was conducted with different sorts of potatoes. The extent to which different acids influence "boiling" could not be established.

The author does not endorse Hesse's statement that "boiling" disappears when the saccharification in the first mash is unfavourably influenced, or when the malt is added in two portions, one at mashing and the other during cooling. When only one-third of the malt was used at mashing and the remainder on the cooler at 17–19° R., the primary fermentation was certainly quieter, but the effect on the after-fermentation was, on the other hand, to make it more vigorous than by the old method. Unlike Hesse, the author obtained boiling fermentations with Bellefreund's method, and claims that this system is just as bad in this respect as Eckert's. In one case boiling fermentations were got rid of by changing the yeast. Species II. had been used, and on changing it, although "boiling" (foaming) was decreased, such good fermentations were not obtained as before.

—T. A. G.

*The Influence of Fluorine Compounds on Beer Yeast.* Zeits. für Spiritusind. 1894, 17, 227.

ERRONT has shown that beer-yeast will accustom itself to gradually increasing quantities of fluorine salts until an amount can be added which would render the original yeast incapable of fermentation.

Experimenting further with different species of yeast, he finds that under the above conditions the physiological life of the yeast cells is quite changed—the power of multiplication being diminished whilst fermentative energy increases. At the same time an influence on the chemical

action is produced, which, although small, is constant under changing conditions of yeast, worts, and fermentation.

	Yeast.	
	Ordinary.	Fluorine-treated.
Alcohol per litre .....	115 cc.	111.5 cc.
Carbon dioxide .....	84.06 grms.	83.51 grms.
Ratio of alcohol to carbon dioxide.	1:0.731	1:0.749

In every case the fluorine-treated yeast gave less than the theoretical quantity of CO<sub>2</sub>.

On fermenting a glucose solution he obtained:—

	Yeast.	
	Ordinary.	Treated.
Unfermented glucose .....	0.55 per cent.	1.21 per cent.
Alcohol formed .....	7.29 "	6.67 "
Alcohol per 100 parts of fermented glucose.	50.49	48.37

The number 50.49 is almost the same as Pasteur's, whilst the second, 48.37, approaches closer to the calculated theoretical amount. The fluorine-treated yeast produces only  $\frac{1}{15}$  to  $\frac{1}{12}$  of the glycerol, and only  $\frac{1}{3}$  of the succinic acid produced by yeast under ordinary circumstances.

—T. A. G.

*The Employment of Sulphurous Acid in Malting.* Kukla. Wochenschr. f. Braueri, 1894, 885–887.

THE primary object of adding sulphurous acid to the steep liquor in malting, is the production of relatively pale malts. In addition to this, the author has made the following observations:—When using acidified water, the duration of steeping is reduced by as much as 20 hours, and subsequent growth on the floors is more rapid and regular, so that the operation at this stage may be shortened by 2–3 days. The resulting malts are stated to be more thoroughly modified, and to yield a better type of extract, although its amount is somewhat smaller. The development of mould-growth is entirely prevented. In practice, the author proceeds as follows:—The barley is at first steeped in the usual way in water alone, which is drawn off at the end of a few hours, and replaced by water, previously thoroughly mixed in a separate vessel with the required volume of sulphurous acid. After 12–14 hours the acid water is drawn off and the barley washed in running water; steeping (in water alone) may then be continued as usual. It must be remembered, however, that the operation will be finished more quickly than usual, and care be taken to avoid over-steeping.

As regards the amount of sulphurous acid to be used, this should be such that 1 cc. of the treated water requires 0.05 cc. of decinormal alkali for its neutralisation. Less may be employed, but on no account more. The precise strength of the acid used must therefore be determined by titration and the calculated amount added to the water, allowance being made for the quantity neutralised by the alkaline and earthy carbonates contained in the water (determined by direct titration, using litmus as indicator). Waters containing much calcium carbonate are unsuitable for the above process, owing to the deleterious influence on the germinating power of barley, of the calcium sulphite formed.—H. T. P.

*Alteration of the Sulphurous Acid in Wines.* E. Chuard and M. Jaccard. *Chem. Zeit.* **18**, 1894, 702.

THE sulphur treatment of wine is based upon the antiseptic properties of the sulphurous acid, a small quantity of which dissolved in the wine, prevents the development of obnoxious micro-organisms. The sulphurous acid as such, however, does not long remain permanent in a wine, part of it escaping into the atmosphere, part becoming oxidised into sulphuric acid. Recent investigations by Schmitt further showed that part of the sulphurous acid combines with the small quantities of aldehyde present in most wines. The compound thus formed received the name of aldehyde sulphurous acid, and it is characterised by a pleasant odour and indifference to the living organism. These various changes show that after a certain time, even strongly sulphured wines will only contain very small proportions of free sulphurous acid. A series of experiments showed that three to four months after sulphuring, most of the free sulphurous acid had disappeared from the wine.

—C. O. W.

*Butylic Alcohol Fermentation and the Butyl-Ferment.* Beijerinck. *Zeits. f. Spiritusind.* **17**, 233—234. From *Centralbl. f. Bakteriologie*. **15**, 171 et seq.

THE author has investigated this subject and finds that a considerable number of bacteria produce butylic alcohol more or less freely during fermentation. He proposes to give the name of *Granulobacter* to this group of organisms which he describes thus:—Wholly or temporarily anaerobic ferment-bacteria, which, when entirely anaerobic become filled with granules and then resemble the clostridium. Carbonic anhydride and hydrogen are always among their products of fermentation, but methane is always absent. The following members of this species (some of which are already known under other names) are described. *Granulobacter butylicum* (Gruber's *Bacillus amylobacter* L.) producing butylic alcohol as principal product. *G. saccharobutyricum* (*Bacillus butylicus*, Fitz.); *G. lactobutyricum*; *G. polymyxa* (Prazmowski 1890).

After discussing at length many subjects connected with fermentation, the author draws the following conclusions:—

a. There are three forms of anaerobiosis, viz., the *facultative*; the apparently *facultative* or *temporary*; and the *essential* (obligate). The facultative anaerobiosis (as for instance lactic fermentation) is independent of the presence or absence of air but requires the presence of reducible materials. The temporary anaerobiosis (yeast, &c.) depends on a reserve of combined oxygen in the cell capable of permitting considerable cell partition (20 to 30 times) before fresh free oxygen becomes necessary. The essential anaerobiosis requires total absence of free oxygen but presence of reducible materials.

b. The fermentative and reductive functions are independent of one another.

c. Alcoholic fermentation may take place in the combined presence of all three forms, and is only impossible in the presence of essential anaerobiosis alone.

d. Facultative and even essential anaerobes can live and grow apparently as aerobes in the absence of assimilable or reducible materials.

e. Fermentation does not occur without accompanying evolution of gas.—L. T. P.

*Note on the Alleged Disappearance from Beer of added Salicylic Acid and Saccharine during Protracted Storage.* J. Wauters. *Wochensch. f. Brauerei*, 1894, 910.

A NUMBER of beers, to some of which salicylic acid, to others, saccharine had been added, were kept for four years in a warm place. At the end of that period, the beers were all acid, but in no case was any difficulty experienced in detecting the added antiseptic.—H. T. P.

*Aluminium Foil as a Beer Clarifying Agent.* Windisch. *Wochensch. f. Brauerei*, 1894, 940.

A SUMMARY of various investigations concerning the practical utility of aluminium for clarifying purposes. Metallic aluminium is employed in the form of corrugated strips, 50 cm. long, and 4 cm. wide, weighing 15 grams. Apart from other considerations aluminium strips possess the advantage over wood shavings of being non-absorbent, and of being readily cleaned. The evidence as to the efficiency of these strips for clarifying purposes is somewhat conflicting, but it appears that they are, at the least, equal in this respect to wood-shavings. As regards the influence of aluminium on the properties of beer, general experience shows that the stability, flavour, condition, &c., are quite unaffected thereby. Hygienically, too, no difficulties present themselves. The amount of aluminium dissolved by beer, even after prolonged contact, is absolutely inappreciable.—H. T. P.

*The Estimation of the Acidity of Malt.* E. Prior, Bayer. *Brauerjourn.* 1894, **4**, 74.

See under XXIII., page 986.

*Sources of Error in Rose's Method of Estimating Candel Oil in Spirit.* M. Glasenapp. *Zeits. fur Spiritusind.* 1894, **17**, 169.

See under XXIII., page 982.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY AND WATER PURIFICATION, DISINFECTANTS.

### (A.)—CHEMISTRY OF FOODS.

*Estimation of the Insoluble, Non-volatile Fatty Acids.* Meillère. *J. Pharm. Chim.* **29**, 60.

See under XXIII., page 983.

### PATENTS.

*Improvements in the Treatment and Preparation of Butter, Butter Substitutes, Margarine, and Alimentary Fats or Compounds.* W. P. Thompson, Liverpool. From C. Artus, Paris, France. Eng. Pat. 20,027, October 24, 1893.

THIS invention relates to a process for the treatment and preparation of butter, butter substitutes, margarine, and alimentary fats, salted or otherwise, without the use of chemicals, with the object of preserving them from putrefaction. The butter or other like product is introduced into suitable vessels perfectly opaque and hermetically sealed and heated therein to a temperature of 60—120° C. It is thus Pastenised (sterilised). The patentee also claims a process for "reconstituting" sterilised butter or like product. It consists in vigorously agitating the butter or the like at a suitable temperature approaching that of solidification, whereby, it is said, the "liquid fatty volatile and aromatic principles dissociated during the sterilisation are emulsified or reconstituted," the solidification is then effected by rapid cooling.—A. R. L.

*Improvements in and relating to the Preservation of Food.*  
H. L. C. Paulsen, Hamburg, Germany. Eng. Pat. 13,031.  
July 5, 1894.

This invention relates to the preservation of articles of food, such as meat, fish, fowl, game, caviare, &c. The process consists in surrounding the articles of food to be preserved with a mixture of an aqueous extract prepared preferably from the following plants:—*Gelidium amansii*, *Gelidium cornutum*, *Gelidium cartilagineum*, *Gelocopeitis filicina*, and *Chondrus crispus*, glycerol and an antiseptic. The plants are steeped in water for 24 hours, and heated to 200° C. for 5–6 hours. After this the juice is strained, and when cold assumes the appearance and consistency of a jelly; it is transparent. This jelly is heated in a paraffin bath to 200° C., and to every 1,000 parts by weight there are added water (2,000 parts), glycerin (250 parts), and alum (50 parts), or boric acid (30 parts); the boiling is continued until the liquid weighs 1,800 parts, when it is cooled down to 8° C.—A. R. L.

#### (B.)—SANITARY CHEMISTRY AND WATER PURIFICATION.

*Dyeing with Lead Chromate from the Hygienic Standpoint.*  
Cazeneuve Rev. d'Hygiène, 1894, 16, 382.

THE Board of Health for the Department of the Rhone has prohibited the use of lead chromate in dyeing cotton hanks intended for reeling. This order is due to the occurrence of several cases of lead-poisoning among the women employed in this industry. The hanks are not thoroughly washed to remove excess of chromate, as is usually the case with fabrics dyed in this manner. In a room in which reeling of the yarn, and spinning of it together with metal threads were carried on, was found:—

	Per Cent. PbCrO <sub>4</sub>
In the thread.....	10.1
In the waste.....	17.9
In the dust on the floor.....	44.2

It is thus easy to understand the occurrence of lead-poisoning, and the necessity for the above prohibition.

—R. B. B.

#### PATENTS.

*Improvements in the Construction of Tanks for the Subsidence of the Precipitate or Sludge resulting from the Chemical Treatment of Sewage or other Foul Waters.*  
H. Lockwood, Manchester. Eng. Pat. 19,069, October 11, 1893.

IMPROVEMENTS are described for the construction of a tank figured in Eng. Pat. 19,308, 1892 (this Journal, 1893, 540). The claim is for a cylindrical settling tank with a conical bottom, and having an inlet opening admitting the sewage at a point considerably below the surface so as not to disturb the subsidence of the sludge. A sudge pipe is also provided, opening at or near the bottom of the conical part of the tank in such a way that the downward pressure of the supernatant liquor will force the sludge through the outlet, so as to dispense with the necessity of pumping.—J. C. C.

*Improvements in Making Manure from Sewage and Sewage Sludge.* J. Carter, London. Eng. Pat. 22,559, November 24, 1893.

See under XV., page 964.

*Improvements in Channels or Receptacles for Sewage and the like Materials and in the Method of Deodorizing or Disinfecting the same.* H. Stoffert, Hamburg, Germany. Eng. Pat. 14,733, August 1, 1894.

ALL parts of the receptacles or channels with which the sewage comes into contact have double or hollow walls forming a space in which the deodorising or disinfecting material is contained; the outer walls are made impervious to liquids and the inner walls are porous, thus allowing the disinfectant to permeate through, and come into contact with the sewage.

—J. C. C.

#### XIX.—PAPER, PASTEBOARD, Etc.

*Improved Apparatus for Raising, Forcing, and Circulating Semi-fluid and Liquid Substances.* J. Taylor, Wandsworth Common, Surrey. Eng. Pat. 13,709, July 16, 1894.

See under I., page 931.

#### XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Manufacture and Compression of Nitrous Oxide.* J. Thilo. Chem. Zeit. 18, 532.

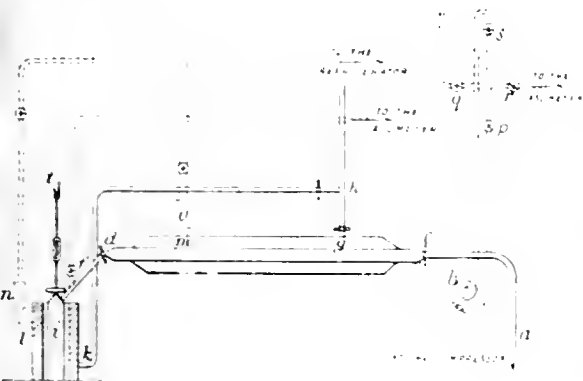
AMMONIUM nitrate (16–18 kilos.) is heated in cast-iron retorts of 30 litres capacity furnished with long narrow necks. This is at least the plan adopted in certain German works. The use of larger quantities of material is attended with danger, inasmuch as a sudden evolution of nitrous oxide may cause explosion. The connections with the washing-flasks are best made by means of caoutchouc stoppers and glass tubing. The principle impurities of the gas as thus prepared are chlorine, hydrogen chloride, and higher oxides of nitrogen. Three washing-flasks are employed for every retort; the first contains water, the second ferrous sulphate solution, and the third caustic soda solution. Whereas the object of the second flask is to render the gas innocuous by freeing it from other oxides of nitrogen, that of the third is to free it from chlorine and chlorine compounds. For the manufacture of nitrous oxide an almost absolutely pure ammonium nitrate only is admissible.

After the gas has passed through the washing-flasks, it is led into a large empty flask which is kept cool by ice, in which the greater part of the moisture of the gas is condensed, and from whence it finally reaches the condenser. It has now to pass through the second phase of the process, viz., compression and liquefaction. Two compressors, giving at most 16 atmospheres pressure, are employed, the remainder of the compression work is accomplished by cold. When the gasometer is completely filled with gas the two double-action pumps are set in motion; the gas is thus sucked out of the gasometer into a tube of 6 litres capacity, tested up to 250 atmospheres, which is surrounded with a copper jacket having inlet and outlet tubes. In this copper jacket enters a cooling fluid consisting of a mixture of carbonic and sulphurous anhydrides (Pictet's fluid) which is drawn out of the same by three other steam pumps and liquefied in a worm condenser, cooled by water at 2–3 atmospheres pressure. It has yet to be mentioned that also between the gasometer and the suction cylinders of the first machine the gas has to pass a similar refrigerator where it is cooled to –20° C. in order to free it from the last traces of water.

By means of the cooling fluid, the temperature of the tube containing the nitrous oxide remains constant at about  $-60^{\circ}$  to  $-70^{\circ}$  C., at which temperature a pressure of 12–14 atmospheres is sufficient to cause liquefaction of the gas. When the latter is accomplished, the tap of the tube is shut off from the compressor and the surplus pressure between this tap and the second compressor allowed to go back into the gasometer through a tube leading to the latter.

As the tube in which the liquefaction is accomplished contains only a little more liquid nitrous oxide than corresponds with that present in the gaseous state in the gasometer the complete evacuation of the gasometer is the signal for the end of the liquefaction. If the pressure rises above 14 atmospheres without the gasometer being empty, this indicates that the gas contains air or other gases compressible with difficulty, which can be let off by a valve placed between the high pressure compressor and the compression tube. The yield amounts generally to 85–95 per cent. of the theoretical.

The liquid nitrous oxide can now be introduced into steel flasks, which resemble those used for carbonic anhydride. These flasks are likewise surrounded with the Pictet refrigerating liquid,  $\text{SO}_2$ ,  $\text{CO}_2$ , contained in a jacket which is in connection with the cooling jacket of the compression tube for the gas, the steel flasks being connected with the compression tube itself; on the connecting tube is placed a valve with a copper tube leading back to the gasometer. In order, therefore, to fill the flasks, which must be previously rendered vacuum, these are placed in the refrigerator and hung on a balance devised for the purpose, and when sufficiently cool, a little nitrous oxide is run in and this is led back into the gasometer through the tube already mentioned. The flask is now sufficiently cool to be charged with the liquefied gas in the compression tube. The increase in weight of the flask due to the liquefied gas is indicated by the balance. The following sketch shows the complete arrangement:—



The nitrous oxide enters at *a* under a pressure of 12–14 mm. which is denoted by a manometer *b*; at *c* the gas passes into the compression tube through the cock. It is here cooled by the  $\text{SO}_2$ ,  $\text{CO}_2$  contained in the jacket. The  $\text{SO}_2$ ,  $\text{CO}_2$  enters the jacket at *g* and can also be led into the second cooling jacket surrounding the flask *i*. The entrance of the cooling liquid in the last-mentioned refrigerator is at *k*, the exit of the gas, which is aspirated by pumps and again condensed, is at *l*, and from the horizontal refrigerator at *m* the gas must yet pass the widened portions *n* and *o* of the tubes. In the tube *cd* the nitrous oxide is liquefied and led into the flask *i* through the tube *df*. At *f* the non-liquefied or re-gasified nitrous oxide is driven back into the gasometer. The cross-piece *p, q, r, s* has the following object:—When the tube *cd* is full of liquefied nitrous oxide and the gasometer is empty, the pressure which still exists between *c* and the compressor is let off into the gasometer through *p, r*. If non-compressible gases have collected, due to impurities, their presence may be recognised by opening the valve *s*,

which is in connection with the manometer *h*, they are eliminated through the valve *q*. The flask *i* hangs on a balance at *t*—A. R. L.

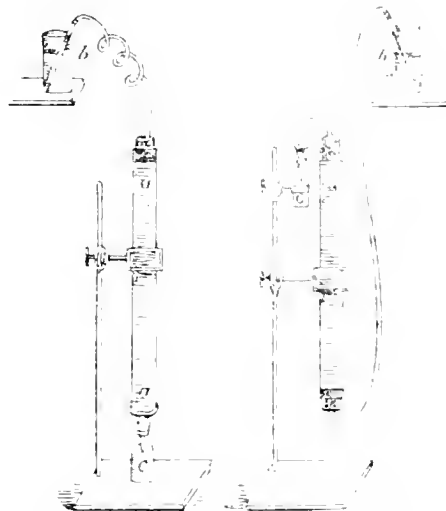
*The Recovery of Residual Tinctures from Mares.* R. H. Parker. Proc. of the Pharm. Conference, 1894 (Oxford). Chem. and Druggist, 45, 1894, 189–191.

*Displacement by Water.*—“Downward” v. “Upward.”—Two parallel experiments were conducted. In each case 2 oz. cort. cinchona flav. (in No. 10 powder) and 10 oz. proof spirit were mixed in a 12-oz. bottle, and occasionally agitated during three or four days, and poured into a glass tube percolator (2 ft. long, 1 in. wide) having muslin tied over the lower end and resting in a funnel and bottle-receiver. As soon as the mare filled the tube, and without allowing the liquid to drain away, the upper end was closed with a perforated cork carrying a tube connected with a water supply.

In one experiment the water was forced in at the top, and the displaced tincture collected in fractions at the bottom.

In the other case the water was forced in at the bottom, and the displaced tincture collected in fractions at the top.

The sketch illustrates the process:—



“DOWNWARD” v. “UPWARD” DISPLACEMENT.

- a.* Glass tube filled with mare.
- b.* Pressure water-supply.
- c.* Bottle to collect displaced tincture.

The following deductions may be made from an examination of the tables given:—

(1.) That the precision of displacement depends upon the relative affinities and miscibility of the superposed fluids at the contact surface; an alcoholic solution being more perfectly displaced by water than by alcohol, especially if each fluid holds in solution a substance insoluble in the other, as in alcoholic percolates of ginger, belladonna, aconite, &c.

(2.) In the downward displacement of alcoholic tinctures by water, diffusion of the latter into the tincture in advance of the rate of percolation takes place to an inconsiderable extent only.

*Conclusion.*—The preparation of tinctures, liniments, &c. by percolation and displacement by water, is much more economical than by screw-pressure of the mare; the process is quite reliable if carefully conducted, but is better adapted to stronger spirituous preparations than to proof spirit compounds. However, the method is unsuitable for operations on a large scale.

The chief points to be observed in order to secure good results are:—

(1.) The materials should be in fairly uniform powder: No. 20 for porous, readily permeable substances; No. 40, or finer, for drugs of denser structure.

(2.) The ingredients should be fairly macerated in the whole of the menstruum (either stirred in the percolator or agitated in a bottle) until extraction is complete.

(3.) The most important point of all is that the marc shall be entirely free from air-spaces and air-channels. To ensure this, particular attention must be paid to the method of packing the marc, and the precise moment when water should be superposed. The marc should be stirred in the percolator with sufficient menstruum to form a semi-fluid mixture; when quite uniform and free from air-bubbles it should be allowed to drain, occasionally jarring the side of the percolator against the hand until it acquires solidity; water should be immediately floated on so as not to disturb the marc. A convenient method is to pour the water through a funnel into the inverted lid of an ointment-pot, previously laid on the surface of the marc.

(4.) The displacement should not be rapid, and the percolate should be collected in relatively small fractions when the total quantity approaches completion—cloudiness, altered gravity, and colour indicating the termination of the process.

*Crystallised Guaiacol.* G. Freyss. Chem. Zeit. 1894, 18, 565—566.

LIKE other phenols of low melting-point, guaiacol gives a different value for this constant, according to whether the determination is made in a test tube or in a capillary tube. The purity should therefore be ascertained through the crystallising or solidifying point, which is always constant, even if the specimen be superfused. Pure guaiacol solidifies at 28.5, not 27 (Seifert, Chem. Zeit. Rep. 1894, 18, 19), and distils at 202.4 at 738 mm. (thermometer in vapour); a very little impurity lowers the solidifying point. Pure guaiacol has a characteristic odour; it crystallises in long, vitreous, transparent prisms, which become rose-red in sunlight. The crystals belong to the hexagonal system;  $a:c = 1:0.9933$ ; other measurements are described in the paper. For therapeutical use only very pure guaiacol should be employed. Gilbert and Maurat (Münchener med. Wochenschr., 1893, 979) found that the toxic dose of crystallised guaiacol for guinea-pigs is 0.8—0.9 gm. per kilo. of body-weight when subcutaneously injected, and 1.5 grms. when internally exhibited; experiments made with the author's pure guaiacol on a dog of 9—10 kilos. body-weight showed that 10 grms., internally exhibited, produced no ill-effect. Gilbert and Maurat have experimented with phthisical patients and have found that crystallised guaiacol, administered in capsules before meals, have had good effect. Wyss (D. med. Wochenschrift, 1894, No. 13) found that 5 cc. of commercial liquid guaiacol (which could not have been pure) was fatal to a girl of nine years and 21.7 kilos. body-weight, when internally exhibited.—A. G. B.

*Technical Analysis of Ethereal Oils.* J. Klimont. Chem. Zeit. 19, 641—642 and 672—673.

See under XXIII., page 985.

#### PATENTS.

*An Improved Method of Producing Carbon Tetrachloride.* A. Muller and H. Dubois, Rheinau, Germany. Eng. Pat. 19,628, October 18, 1893.

THE method consists in heating a mixture of sulphur chloride and carbon bisulphide with addition of metals or metallic haloids, especially iron or ferric chloride, when carbon tetrachloride is produced in accordance with the equation— $\text{CS}_2 + 2\text{SCl}_2 = \text{CCl}_4 + 6\text{S}$ . An excess of sulphur chloride renders the reaction less violent, and increases the yield.

The following example is given to illustrate the manner in which the process may be carried out:—A mixture of carbon bisulphide (76 kilos.), sulphur chloride (405 kilos.), and pulverised iron (500 grms.), is heated to about 60° C. in an iron vessel coated with lead, and provided with a lateral man-hole, a reflux condenser, and a steam coil. The reaction commences spontaneously, and at the end the mixture is heated to boiling for a short time. On cooling, the sulphur separates in large crystals, and may be reconverted into sulphur chloride or carbon bisulphide so as to be again included in the cycle of the reaction.

The liquid run off from the sulphur is subjected to fractional distillation in order to separate the carbon tetrachloride from the excess of sulphur chloride. The crude carbon tetrachloride thus obtained is further purified by the known methods.—A. R. L.

*Improvements in the Manufacture of Antiseptics.* H. E. Newton, London. From The Farbenfabriken vormals F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 19,622, October 18, 1893.

IN Eng. Pat. 3301, 1892 (this Journal, 1893, 172) the inventors claim for antiseptic, deodorising, and disinfecting purposes the use of salts of dinitro-ortho-cresol. They now find that free dinitro-ortho-cresol possesses a high degree of antibacterial power, although the metallic salts exhibit this property in only a slight degree. The solubility is about one part of dinitro-ortho-cresol in 4,000 to 5,000 parts of water, but even this strength of solution is more powerful than a 0.5 per cent. solution of the potassium salt. Stronger solutions of the body may be obtained in the solution of its potassium salt, but a 10 per cent. solution of borax will dissolve 10 per cent. of free dinitro-ortho-cresol, or dry mixtures of one part of the free body, and two parts of borax may be prepared.—J. C. C.

*A Process for Converting Compounds of the Citral Series (Geranium Series) into Isomers having a Higher Specific Weight and a lower Boiling Point than the Original Bodies.* J. C. W. F. Tiemann, Berlin, Germany. Eng. Pat. 22,114, November 18, 1893.

WHEN geranic acid,  $\text{C}_{10}\text{H}_{16}\text{O}_2$ , which is an oil boiling at 153° C., under a pressure of 13 mm., is added with agitation to 70 per cent. sulphuric acid cooled below 0° C., it is transformed into a solid isomeric acid which crystallises from ligroin. The isogeranic acid melts at 103.5° C., and boils at 138° C. under a pressure of 11 mm.

In the same manner geraniumitrile,  $\text{C}_{10}\text{H}_{15}\text{N}$ , which boils under a pressure of 10 mm. at 116° C., and has a specific gravity of 0.8709, may be transformed into the nitrile of isogeranic acid which boils at 87°—88° C. under a pressure of 11 mm., and has a specific gravity of 0.9208.

Geraniolene,  $\text{C}_{10}\text{H}_{16}$ , the hydrocarbon obtained by removing the elements of carbonic anhydride from geranic acid boils at 142°—143° C., and has a specific gravity of 1.4368. When heated with 60 per cent. sulphuric acid for 3—4 hours it is converted into isogeraniolene, which boils at 138°—140° C., and has a specific gravity of 1.4434.

Phosphoric acid or metaphosphoric acid may be substituted for sulphuric acid, but the latter gives the best results. The iso-compounds of the citral series are intended to be used for perfumery purposes.—A. R. L.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

## PATENT.

*Improvements in the Process of Making Photographic Films.* E. Edwards, London. From R. Reissner and G. C. F. Hauser, Erlangen, Germany. Eng. Pat. 18,237, September 28, 1893.

This is a process for rendering gelatin films insoluble in hot water, and consists in dissolving the gelatin in a weak solution of formaldehyde, forming films from this, and drying, the rendering insoluble of the films being effected during the drying; 0.2 part of formaldehyde being sufficient to render insoluble 1 part of gelatin.

In place of formaldehyde, a solution is claimed, the component parts of which, by their mutual influence, yield formaldehyde.—J. C. C.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*Researches on Modern Explosives.* W. Macnab and E. Ristori. Proc. Royal Society, 56, 8—19.

This paper chiefly records the results of experiments made to determine the amount of heat evolved, and the quantity and composition of the gases produced, when certain explosives and various smokeless powders now in use were fired in a closed vessel from which the air had been previously exhausted.

The explosions were carried out in a "calorimetric bomb" of Berthelot's pattern, details of the procedure are given in the paper and special care was taken to accurately determine the amount of water produced.

The principal results are given in the following tables:—

TABLE I.—INDICATING THE QUANTITY OF HEAT, also the VOLUME and ANALYSIS of the GAS developed per gram, with different SPORTING and MILITARY SMOKELESS POWDERS now in use.

Name of Explosive.	Calories Per Gram.	Permanent Gases.	Aqueous Vapour.	Total Volume of Gas calcu- lated at 0° and 760 mm.	Per Cent. Composition of Permanent Gases.					Coefficient of Potential Energy.
					CO <sub>2</sub>	CO	CH <sub>4</sub>	H.	N.	
EC powder.....	800	Ce. Per Gram. 420.	Ce. Per Gram. 154	Ce. Per Gram. 374	22.9	40.6	6.5	15.5	20.5	450
SS sporting powder } English ... {	790	584	150	734	18.2	45.4	9.7	20.0	15.7	546
Troisdorf, German.....	943	700	195	895	18.7	47.9	0.8	17.4	15.2	844
Rifleite, English.....	864	706	159	925	14.2	50.1	0.3	20.5	14.9	799
BN, French.....	833	738	168	906	13.2	53.1	0.7	19.4	13.6	755
Cordite, English manufacture....	1053	647	235	882	24.9	40.3	0.7	14.8	19.3	1105
Ballistite, German manufacture.	1291	591	231	822	33.1	35.4	0.5	10.1	20.9	1064
Ballistite, Italian and Spanish manufacture.	1317	581	245	826	35.9	32.6	0.3	9.0	22.2	1088

TABLE II.—INDICATING THE QUANTITY OF HEAT, also the VOLUME and ANALYSIS of the GAS developed per gram, with NITRO-GLYCERIN, NITRO-CELLULOSE, and with several different COMBINATIONS of these two EXPLOSIVES made at ARDEUR FACTORY.

Composition of Explosive.	Calories Per Grm.	Permanent Gas.	Aqueous Vapour.	Total Volume of Gas calcu- lated at 0° and 760 mm.	Per Cent. Composition of Permanent Gases.						Coefficient of Potential Energy.
					CO <sub>2</sub>	CO.	CH <sub>4</sub>	O.	H.	N.	
		Ce. Per Grm.	Ce. Per Grm.	Ce. Per Grm.							
A. Nitro-glycerin .....	1652	464	257	741	63.0	..	..	4.0	..	33.0	1224
B. Nitro-cellulose (nitrogen = 13.30 per cent.).	1061	673	203	876	22.3	45.4	0.5	..	14.9	16.9	920
C. { 50 per cent. nitro-cellulose (N) = 12.24 per cent. )	1349	568	249	817	36.5	32.5	6.2	..	8.4	22.1	1102
50 per cent. nitro-glycerin ....											
D. { 50 per cent. nitro-cellulose (N) = 13.30 per cent. )	1410	550	247	797	41.8	27.5	0.0	..	6.0	24.7	1124
50 per cent. nitro-glycerin ....											
E. { 80 per cent. nitro-cellulose (N) = 12.24 per cent. )	1062	675	226	901	21.7	45.4	0.1	..	15.7	17.1	957
20 per cent. nitro-glycerin .....											
F. { 80 per cent. nitro-cellulose (N) = 13.30 per cent. )	1150	637	227	864	26.6	40.8	0.1	..	12.0	20.5	1001
20 per cent. nitro-glycerin ....											
G. { 35 per cent. nitro-cellulose (N) = 13.30 per cent. )	1280	627	236	803	26.7	39.8	0.5	..	12.8	20.2	1105
5 per cent. vaseline .....											
60 " " nitro-glycerin .....											



TABLE III.—SHOWING the HEAT developed by EXPLOSIVES containing NITRO-GLYCERIN and NITRO-CELLULOSE in different proportions

Composition of Explosives.			Calories Per Grm.
Nitro-cellulose (N = 13.3 Per Cent.).	Nitro-glycerin.		
100 per cent. (dry pulp)	0		1061
100 " " (gelatinised)	0		922
90 " "	10 per cent.		1044
80 " "	20 " "		1159
70 " "	30 " "		1267
60 " "	40 " "		1347
50 " "	50 " "		1410
40 " "	60 " "		1467
0 " "	100 " "		1652
<hr/>			
Nitro-cellulose (N = 12.24 Per Cent.).	Nitro-glycerin.		
80 per cent.	20 per cent.		1062
60 " "	40 " "		1288
50 " "	50 " "		1319
40 " "	60 " "		1465
<hr/>			
Nitro-cellulose (N = 13.3 Per Cent.).	Vaseline.	Nitro-glycerin.	
55 per cent.	5 per cent.	40 per cent.	1134
35 " "	5 " "	60 " "	1280

The figures in columns headed "Coefficient of potential energy" serve as a measure of comparison of the power

of the explosives, and are the products of the number of calories by the volume of gas, the last three figures being suppressed in order to simplify the results.

The amounts of water found were calculated for comparison as volumes of H<sub>2</sub>O gas at 0° and 760 mm.

EC powder consists principally of nitrocellulose mixed with barium nitrate and a small proportion of camphor; SS, of nitroguin mixed with barium nitrate and nitrobenzene.

Troisdorf powder is gelatinised nitrocellulose; rifleite is made of gelatinised nitrocellulose, to which is added a certain proportion of nitrobenzene; BN consists mainly of gelatinised nitrocellulose.

Cordite contains 58 per cent. of nitroglycerin, 37 per cent. of gun-cotton, and 5 per cent. of vaseline.

Ballistite of Italian manufacture contains equal parts of nitrocellulose and nitroglycerin, with the addition of  $\frac{1}{2}$  per cent. of aniline.

The German ballistite contains a slightly higher percentage of nitrocellulose.

TABLE IV.—SHOWING the HEAT developed and the ANALYSIS of the PERMANENT GAS produced in a closed VESSEL from which the AIR has not been exhausted, the EXPLOSIVE being in every case BALLISTITE of ITALIAN MANUFACTURE.

Charge.	Calories per Grm.	Analysis of the Permanent Gas.			
		CO <sub>2</sub>	CO	H.	N.
2 grms. ....	1587	37.0	17.6	3.2	42.2
3 " .....	1485	36.4	22.0	4.6	37.0
4 " .....	1446	36.2	24.6	6.1	33.1
5 " .....	1415	36.2	26.0	7.2	30.6
6 " .....	1380	36.3	27.0	7.9	28.6

Traces of CH<sub>4</sub> were found, but in this series of experiments the quantity of this gas was not determined.

TABLE V.—SHOWING the ORIGINAL COMPOSITION and METAMORPHOSIS of NITRO-CELLULOSE, NITRO-GLYCERIN, and of several GUNPOWDERS made by COMBINATIONS of these two EXPLOSIVES.

Nature and Description of Explosive.	Per Cent. Composition by Weight.				Per Cent. Products of Combustion by Weight.						
	Carbon, Oxygen.		Hydro- gen, H.	Nitrogen, N.	Carbonic Acid, CO <sub>2</sub>	Carbonic Oxide, CO.	Marsh Gas, CH <sub>4</sub>	Oxygen, O.	Hydro- gen, H.	Nitrogen, N.	Water, H <sub>2</sub> O.
	C.	O.									
A. Nitro-glycerin .....	15.7	63.0	2.3	18.8	57.6	..	..	2.7	..	18.8	20.7
B. Nitro-cellulose (nitrogen=13.3)	24.58	57.63	2.73	13.6	29.27	38.52	0.24	..	0.86	13.6	16.50
C. { 50 per cent. nitro cellulose (N = 12.24 per cent.) .....	21.15	60.67	2.67	15.58	41.0	23.1	0.08	..	0.4	15.58	20.01
{ 50 per cent. nitro-glycerin .....											
D. { 50 per cent. nitro-cellulose (N = 13.30 per cent.) .....	20.47	61.23	2.49	16.35	45.3	19.0	0.00	..	0.3	16.35	19.90
{ 50 per cent. nitro-glycerin .....											
E. { 50 per cent. nitro-cellulose (N = 12.24 per cent.) .....	24.37	58.98	2.98	14.0	28.9	38.4	0.05	..	1.0	14.0	18.2
{ 20 per cent. nitro-glycerin .....											
F. { 50 per cent. nitro-cellulose (N = 13.30 per cent.) .....	23.11	58.98	2.71	15.84	33.4	32.6	0.04	..	0.7	15.84	18.2
{ 20 per cent. nitro-glycerin .....											
G. { 35 per cent. nitro-cellulose (N = 13.30 per cent.) .....	22.2	59.0	2.88	15.46	33.0	31.3	0.2	..	0.7	15.46	19.0
{ 5 per cent. vaseline .....											
H. Cordite, English manufacture..	22.91	57.72	2.95	15.19	31.76	32.68	0.32	..	0.86	15.19	18.08
K. Ballistite, Italian and Spanish manufacture.	21.47	60.83	2.68	15.80	41.11	23.76	0.12	..	0.47	15.8	19.69

—W. M.

PATENTS.

*An Improved Apparatus for Use in Measuring the Pressure of Gas in Guns.* A. Castenholz, Karlsruhe, Germany. Eng. Pat. 11,991, August 4, 1893.

See under L, page 929.

*Improvements in the Manufacture of Gunpowder.* A. Schindler, Breslau, Germany. Eng. Pat. 20,327, October 27, 1893.

THE proposed powder consists of 12 parts by weight of potassium chlorate, 3 parts of sugar, and 5 parts of anthracite, all finely ground and mixed. It is stated that anthracite, being denser and less easily combustible than ordinary charcoal, prevents the chlorate powder made in the way described from being too rapid in its action.

—W. M.

*Improvements in the Manufacture of Explosives.* H. H. Lake, London. From C. Pieper, Berlin, Germany. Eng. Pat. 23,773, December 2, 1893.

THIS invention relates to explosives composed of ammonium nitrate and dinitrobenzene or nitronaphthalene, &c., and consists in using a solvent for one or other or both of the ingredients, effecting the mixing in the wet state, and then evaporating off the solvent, care being taken that the temperature used for this purpose is not sufficiently high to melt the nitrohydrocarbon. In this way more intimate contact is ensured between the particles of the components, and the explosives thus prepared can be fired by a much smaller detonator, viz., an ordinary dynamite detonator containing 0.54 grm. of fulminate.—W. M.

*Improvements in and relating to the Method and Apparatus for Blasting.* A. W. Schwarz, Leipzig, Germany. Eng. Pat. 7098, April 10, 1894.

THIS improvement consists in making chloride, bromide, or iodide of nitrogen as required in the blasting cartridge itself, and exploding the same in a manner described in the specification.—J. C. C.

*Employment of Trinitroresorcine as Smokeless Driving Powder.* J. Hauff, Stuttgart, Germany. Eng. Pat. 9798, May 19, 1894.

THE claim of this patent is: "The employment of trinitroresorcine as smokeless driving powder, either in granular form, which is obtained from crystallisation by alcohol, pulverisation and sifting, or in grains pressed in the manner of black powder, or by appropriate gelatinising mediums brought to a colloidal form, grain or lamina, and covering single grains with a thin coating of indifferent bodies—for example, paraffin—whereby the burning goes on slowly, and therefore the explosive force is diminished."—W. M.

*Improvements in or relating to the Exploding of Blasting Charges in Mines.* L. Jaroljmeck, Prague, Bohemia. Eng. Pat. 13,714, July 16, 1894.

IN this invention the heat necessary for firing a charge in a borehole is generated in the first instance by allowing the water used as tamping to act on quicklime or a similar substance. By arrangements which are described and illustrated in the specification, the heat resulting from the hydration of the lime is caused to ignite a sensitive detonator which fires the explosive, or it melts a plug of fusible metal and allows sulphuric acid to flow from another compartment of the cartridge into the explosive, or the swelling of the lime is caused to exert pressure and rend asunder an ignition capsule which detonates the explosive.—W. M.

XXIII.—ANALYTICAL CHEMISTRY.

APPARATUS, ETC.

*Some Notes on Sampling.* David Surley. A paper read before the Glasgow City Analysts' Society, 1893-94. No. 1.

Iron Ore should always be sampled at the ship's side from waggons, as the ore is there weighed for invoice purposes, and subsequent fluctuations in the proportion of water will not then affect buyer or seller. Sampling from the "bing" at the ironworks should not be attempted, except as a last resource, as the lumps are used for making the face of the bing, the smaller ore, which is different in composition, forming the centre; as the sample at best can only be taken from the surface, a fair average cannot be obtained. In sampling iron ore the instruments required are a geological hammer, a small hand shovel, and a canvas bag to hold the sample. To draw a sample from trucks proceed thus:—Note carefully the proportions of "lumps," "rubble," and "smalls" in the waggon in which you are standing. Take your proportion of smalls from different parts of the waggon in quantities of about a teaspoonful at a time, and place these on the shovel. Take samples of the rubble and lumps in the same way. In sampling the lumps, the whole piece must be broken and chips taken from both the outside and the inside; no piece should be larger than a cherry. The quantity taken from one waggon will thus weigh about 10 oz., and is at once placed in the bag. Now go into the other waggons and proceed in precisely the same way, judging the proportions as it each waggon contained a separate cargo. The sample is next ground in small portions at a time on a planed steel plate by means of hammers, till the ore is fine enough to pass through a sieve having five meshes to the linear inch. The whole is thoroughly mixed, spread out, quartered, and two opposite quarters further ground finely enough to pass through a 10-mesh sieve. From this a fair average sample is taken to the laboratory. Sampling on a larger scale, as sometimes carried out, the sample taken even weighing as much as 2½ tons, involves more labour, and, though giving equally good, yields no better results. In a recent case the same cargo was sampled in both ways, and the two samples showed a difference of only 0.09 per cent. of iron. The author objects to "tub" sampling and to a common English practice in which the sampler estimates the proportion of lumps, rubble, and smalls by the appearance of the cargo in the ship's hold, afterwards drawing intermittent samples from, say, eight waggons in the course of the day. Joint sampling, in which representatives of the buyer and seller draw the sample jointly, is highly undesirable. It is noteworthy that the smalls of Campanil and Vena iron ores contain about 2 per cent. more iron than the lumps, whereas the lumps of Rubis ore are richer than the smalls in the same proportion.

*Pyrites Residues* from the roasting of pyrites should be sampled from waggons, as in the case of iron ore, if at all possible, as it is very irregular. Care must be taken to break the lumps, and to take portions from the centre as well as from the outside, as if there be any unburnt sulphur it will be there.

*Basic Slag* in the form of meal is simply sampled from the bags with a sampling needle in the usual way. When unground, it is sampled very carefully from each hutch as it is being conveyed to the grinding machinery. Two samples are taken: moisture is estimated in one without grinding, while the other is finely powdered for analysis.

*Pig Iron* is sampled by breaking the pig and taking borings to the depth of 1 in. from the clean fractured surface.

*Wood Pulp* is imported in bales or bundles weighing from 20 lb. to 1 cwt. In sampling, the bale is opened out and a diagonal strip is cut from one of the sheets. The portion taken out is cut into small pieces and bottled quickly to prevent loss of water from the wet pulp.

In sampling *Sulphate of Ammonia*, *Sulphate of Potash*, and salts in general, care should be taken to get an average

sample from the whole depth of the bags, as such salts drain rapidly, the bottom layer becoming wetter and the top layer drier.—L. A.

*Table for the Comparison of Hydrometers.* Reprinted from "The Chemical Trade Journal" of October 13, 1894.

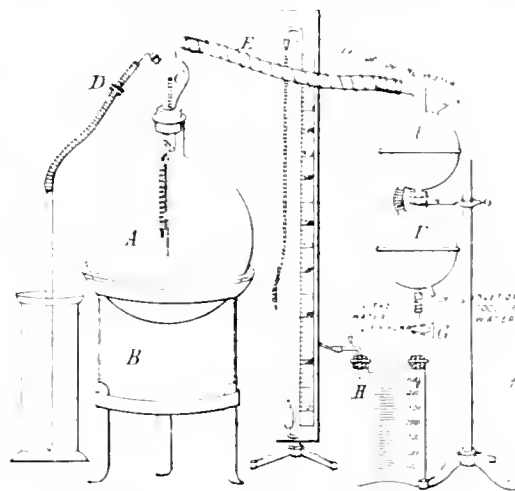
Degrees.	Specific Gravities.		
	Twaddell.	Beaumé.	Light Beaumé.
1	1'005	1'0039	..
2	1'010	1'0140	..
3	1'015	1'0212	..
4	1'020	1'0285	..
5	1'025	1'0359	..
6	1'030	1'0435	..
7	1'035	1'0510	..
8	1'040	1'0588	..
9	1'045	1'0666	..
10	1'050	1'0746	1'0000
11	1'055	1'0827	0'9931
12	1'060	1'0909	0'9863
13	1'065	1'0990	0'9795
14	1'070	1'1073	0'9730
15	1'075	1'1162	0'9664
16	1'080	1'1250	0'9600
17	1'085	1'1338	0'9536
18	1'090	1'1428	0'9473
19	1'095	1'1520	0'9411
20	1'100	1'1612	0'9350
21	1'105	1'1707	0'9290
22	1'110	1'1803	0'9230
23	1'115	1'1900	0'9171
24	1'120	1'2000	0'9113
25	1'125	1'2100	0'9055
26	1'130	1'2203	0'9000
27	1'135	1'2307	0'8944
28	1'140	1'2413	0'8888
29	1'145	1'2521	0'8834
30	1'150	1'2631	0'8780
31	1'155	1'2743	0'8727
32	1'160	1'2857	0'8674
33	1'165	1'2972	0'8622
34	1'170	1'3090	0'8571
35	1'175	1'3211	0'8520
36	1'180	1'3333	0'8470
37	1'185	1'3457	0'8421
38	1'190	1'3584	0'8372
39	1'195	1'3714	0'8323
40	1'200	1'3846	0'8275
41	1'205	1'3980	0'8228
42	1'210	1'4117	0'8181
43	1'215	1'4257	0'8135
44	1'220	1'4400	0'8089

Degrees.	Specific Gravities.		
	Twaddell.	Beaumé.	Light Beaumé.
45	1'225	1'4545	0'8044
46	1'230	1'4633	0'8000
47	1'235	1'4745	0'7955
48	1'240	1'4899	0'7912
49	1'245	1'5157	0'7863
50	1'250	1'5319	0'7826
51	1'255	1'5483	0'7783
52	1'260	1'5652	0'7736
53	1'265	1'5824	0'7700
54	1'270	1'6000	0'7659
55	1'275	1'6179	0'7619
56	1'280	1'6363	0'7578
57	1'285	1'6551	0'7539
58	1'290	1'6744	0'7500
59	1'295	1'6941	0'7461
60	1'300	1'7142	0'7422
61	1'305	1'7349	0'7384
62	1'310	1'7562	0'7346
63	1'315	1'7777	0'7309
64	1'320	1'8000	0'7272
65	1'325	1'8227	0'7236
66	1'330	1'8461	0'7200
67	1'335	1'8701	0'7164
68	1'340	1'8947	0'7128
69	1'345	1'9200	0'7093
70	1'350	1'9459	0'7058
71	1'355	1'9726	0'7024
72	1'360	2'0000	0'6990
73	1'365	..	0'6951
74	1'370	..	0'6923
75	1'375	..	0'6889
76	1'380	..	0'6857
77	1'385	..	0'6824
78	1'390	..	0'6792
79	1'395	..	0'6760
80	1'400	..	0'6728
81	1'405	..	0'6697
82	1'410	..	0'6666
83	1'415	..	0'6635
84	1'420	..	0'6605
85	1'425	..	0'6575
86	1'430	..	0'6545
87	1'435	..	0'6515
88	1'440	..	0'6486
89	1'445	..	0'6457
90	1'450	..	0'6428
91	1'455	..	0'6400
92	1'460	..	0'6371

Degrees.	Specific Gravities.			Degrees.	Specific Gravities.	
	Twaddell.	Beaume.	Light Beaume.		Twaddell.	Twaddell.
93	1.465	..	0.6343	157	1.781	1.820
94	1.470	..	0.6315	158	1.790	1.825
95	1.475	..	0.6288	159	1.795	1.830
96	1.480	..	0.6260	160	1.800	1.835
97	1.485	..	0.6233	161	1.805	1.840
98	1.490	..	0.6206	162	1.810	1.845
99	1.495	..	0.6180	163	1.815	1.850
100	1.500	..	0.6153			
101	1.505	..	0.6127			
102	1.510	..	0.6101			
103	1.515	..	0.6075			
104	1.520	..	0.6050			
105	1.525	..	0.6025			
106	1.530	..	0.6000			
107	1.535					
108	1.540					
109	1.545					
110	1.550					
111	1.555					
112	1.560					
113	1.565					
114	1.570					

*Vacuum Evaporating Apparatus for Laboratory Purposes.*  
Soxhlet. Chem. Zeit. 18, 721—722.

THE evaporation of large volumes of liquids in the chemical laboratory is a tedious and lengthy operation; if a free flame is employed there is a danger of loss of material, and there is no alternative but to use the water-bath. But many solutions—for example, sugar solutions—cannot be heated at the temperature of the water-bath without decomposition, besides which they cannot be concentrated to a syrupy condition as quickly as is desirable. The most rapid mode of procedure, in which decomposition is prevented, is evaporation under diminished pressure. By the aid of the apparatus figured below, 4 litres of water per hour may be evaporated at a temperature of 32—35 C.; or, if the condenser be surrounded by a freezing mixture, the same quantity of water per hour may be evaporated at 24—26 C.



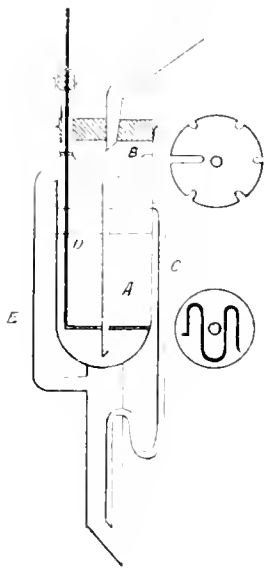
LABORATORY VACUUM EVAPORATING APPARATUS.

The glass flask A, which serves as a vacuum pan, rests on a copper water-bath B, having a rim adapted to it. The pear-shaped piece C is in connection with an arrangement for charging the flask. The caoutchouc tube E surrounded by wire spiral, serves to connect the flask with two large globular condensers of the author's well known construction. The tube G, furnished with a tap, leads to a graduated Wulff's bottle, which receives the distillate. To the second tubulus of the Wulff's bottle is attached a T-piece, one limb of which is in connection with a Korting's water-pump and the other with a mercury manometer.—A. R. L.

Degrees.	Specific Gravities.	Degrees.	Specific Gravities.
	Twaddell.		Twaddell.
115	1.575	136	1.680
116	1.580	137	1.685
117	1.585	138	1.690
118	1.590	139	1.695
119	1.595	140	1.700
120	1.600	141	1.705
121	1.605	142	1.710
122	1.610	143	1.715
123	1.615	144	1.720
124	1.620	145	1.725
125	1.625	146	1.730
126	1.630	147	1.735
127	1.635	148	1.740
128	1.640	149	1.745
129	1.645	150	1.750
130	1.650	151	1.755
131	1.655	152	1.760
132	1.660	153	1.765
133	1.665	154	1.770
134	1.670	155	1.775
135	1.675	156	1.780

*Extraction Apparatus for Liquids.* A. Kurbatow. Russ. phys.-chem. Gesellsch. 1894, 10. Chem. Zeit. Rep. 1894, 18, 101.

The apparatus shown in the figure resembles an ordinary Soxhlet's extractor for solids, save that the tube C is connected with the upper instead of the lower end of the extraction chamber A, and thus no longer functions as a siphon, but as a simple delivery tube back into the flask containing the solvent. The vapour of the solvent ascending



through the tube E, passes through slots in the sides of the funnel B to the condenser, and the resulting liquid flows down the stem of the funnel to the bottom of the liquid to be extracted, through which it rises and trickles over through C to the flask below. D is a glass rod that can be used as a stirrer without opening the apparatus.—B. B.

*An Improved Form of Hard Glass Beakers for Dyeing Tests.* R. Lepetit. Färber Zeit. 5, 1894, 183.

THE china beakers with a ring round their middle are well known, but their expense militates much against their general use. In France conical china beakers, without the ring, are much in vogue. They are considerably cheaper, and the depth to which they may be immersed in the bath is determined by their increasing circumference. These conical china beakers have several advantages over the frequently used cylindrical hard glass beakers, the most important of which is that more concentrated baths with small volumes of water can be used in them. This allows of an approach to the conditions under which the dyeing on a large scale is performed, the volume of the dye-bath used in proportion to the weight of fibre dyed in it, being relatively far greater in the experiment than in practice. This is particularly important when experimenting with slow-dyeing colours, which with the same depth of dye-bath produce from 10 to 20 per cent. stronger shades in conical than in cylindrical beakers. On the other hand, the opacity of the china beaker is certainly a drawback, as it does not admit of the ready observation of the degree of exhaustion or formation of sediments in the bath. This drawback is avoided, without losing the advantage of the conical beaker, by making these conical beakers of hard glass. The capacity of these beakers is about 400 cc., but it is important to see that the dimensions of all the beakers used for comparative tests are exactly identical.—C. O. W.

## INORGANIC CHEMISTRY.—QUALITATIVE.

*Detection of Adulterants in Basic Slag.* O. Böttcher. Chem. Zeit. 1894, 18, 565.

A NUMBER of "Thomas phosphate meals," "Thomas slags," and the like are now on the market. They are merely ground mineral phosphate coloured with powdered charcoal, &c.

Wrampelmeyer has pointed out that a microscopical examination should form the preliminary operation in detecting mineral phosphate adulteration of basic slag; natural phosphates are lighter in colour, and when considerably magnified, more rounded in structure than is basic slag, which consists of sharp-angled splinters which scatter the light. Loss on ignition, specific gravity and solubility in water should also be determined; if the first of these be low and the two last high, the slag is genuine. As a last resort the citrate solubility may be determined.

The author advocates a test for fluoride in the suspected slag, as suitable for the use of the layman. It is of importance on account of the constancy with which fluorides are present in mineral phosphates. 10—15 grms. of the slag in a beaker are covered with 15 cc. of concentrated sulphuric acid, and stirred with a glass rod; the beaker is covered with a clock glass to which a drop of water is suspended, the usual deposit of silica being detected if fluorides be present. After 5—10 minutes the clock glass is cleaned and inspected to see if etched. In this way the author has detected Florida phosphate, sand phosphate, river phosphate, Carolina phosphate, Hungarian phosphate, and a prepared phosphate, respectively, when mixed to the extent of 10 per cent. with basic slag.—A. G. B.

## INORGANIC CHEMISTRY.—QUANTITATIVE.

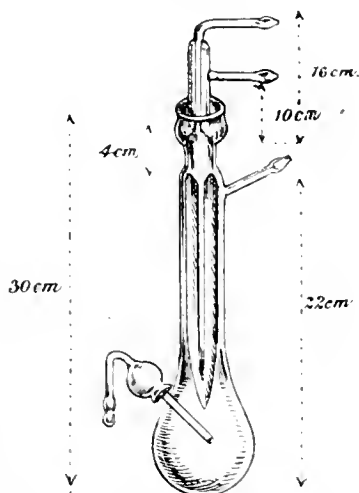
*Colour-Gauges for Carbon Testing.* W. G. McMillan. Iron and Steel Institute, Autumn Meeting, 1894.

IN respect of accuracy, rapidity, and general convenience the author considers the colorimeter, even in its existing form, to be greatly superior to the old colour tubes used in the Eggertz process for carbon determination. He, however, points out certain possibilities of error in the use of the internally illuminated (disc) chromometers, owing *inter alia* to the necessarily unequal illumination of the tubes, and lays down the following, amongst others, as the essentials of a good colour gauge:—All portions of the apparatus with which the coloured liquid shall come in contact should be of glass or glazed porcelain and free from cement. Every such part must be accessible and capable of being readily cleaned. The illumination of the two tubes must be equal and uniform. Following out these rules the author has devised two colour gauges, one internally illuminated, the other externally; both are fully described and illustrated by drawings. The former, or disc-colour gauge, has necessarily such errors as are inseparable from this form of instrument, but the author has endeavoured to simplify the mechanism. Two graduated tubes stand side by side, each with a porcelain disc near the bottom at the zero of the graduations. One of them is connected by a narrow glass tube sealed on to the bottom with a third vertical tube (wider than the others) in which a glass plunger works up and down, displacing the liquid and altering the level in the graduated tube connected with it; the plunger may be fixed at any point by means of a set screw. In the externally illuminated or transmittent colour-gauge, which the author prefers as being more scientific, and free from the errors of the disc instrument, the comparison tubes stand in a wooden box having a longitudinal slot in the bottom with a white opaque reflector beneath, so that the colour tubes are equally illuminated by direct rays, and only from beneath. The alteration in the level of the liquid is made by sliding into the comparison tubes inner glass tubes, the bottoms of which are rounded like those of the outer tubes, so that when they are in the lowest position all the liquid in the

outer tubes is displaced into reservoirs formed by enlargements in the latter, and on looking through the inner sliding tubes from above only colourless discs are seen. On raising the displacement tubes any required density of colour may be obtained, according to the position in which they are fixed; and as the two displacement tubes work independently a comparison of colour is readily made. The heights of the columns of liquid are given by indicators attached to the displacement tube and travelling over a fixed scale. To obviate the irregularities of the illuminated field introduced by round ended glass tubes, the bottoms of the outside tubes are dipped into a trough of water, and the interiors of the displacement tubes are also filled with water, the meniscus of which is flattened by a glass disc. Separate eye-piece tubes are provided. The author prefers the word *Colour-gauge* to other current terms.—W. G. M.

*The Determination of Carbon in Steel.* H. Koch. Chem. Zeit. 1894, 18, 485.

THE flask shown in the accompanying figure is used for the determination of carbon in steel by direct oxidation with chromic acid in sulphuric acid solution. Its dimensions are given in the figure. It will be seen that it consists of two principal parts, the flask proper with a side tube at its lower part for leading in air free from  $\text{CO}_2$  to sweep out the gases evolved by the oxidation of the steel, and a second side tube



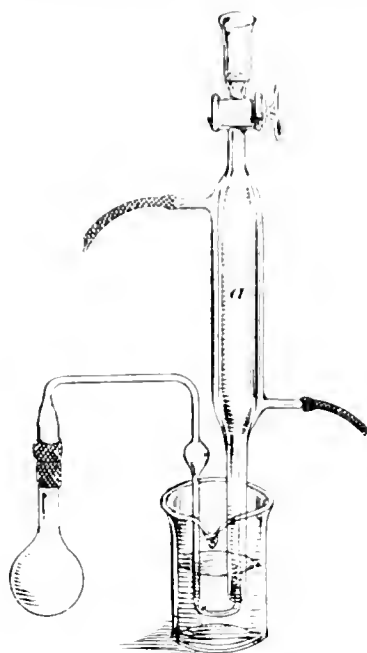
at the upper part of the neck to deliver the evolved gases into the combustion tube containing heated cupric oxide in which the oxidation of any hydrocarbons is completed. The quantities of reagents used are 8 grms. of  $\text{CrO}_3$  and 120 cc. of sulphuric acid of sp. gr. 1.62, the amount of steel (in fine borings) taken, being 2 grms.

The second main part of the apparatus is the condenser with appropriate tubes for leading the cooling water in and out, projecting down the neck of the flask and ground in at the upper end which is constricted for that purpose. By the use of a condenser thus placed, the contents of the flask may be freely boiled to effect the oxidation of the steel and the contained carbon which is to be determined. The apparatus has also been used by the author for the determination of carbon in coal.—B. B.

*Apparatus for the Valuation of Manganese Dioxide by Bunsen's Method.* C. Ullmann. Chem. Zeit. 1894, 18, 487.

THE Bunsen method for the valuation of manganese dioxide is less used than is warranted by its intrinsic merits, chiefly on account of the defects of the apparatus commonly employed. The author claims that the device shown in the figure is free from such shortcomings. The method to be employed

is obvious from the illustration. It suffices to remark that the beaker can be supported on a wooden block, and that when the whole of the chlorine has been evolved the block



can be removed and the beaker lowered before taking the burner from under the flask, thus avoiding the possibility of regurgitation. The bulb on the delivery tube of the flask serves to prevent the return to the flask of any liquid remaining in the rectangular bend. During the absorption of chlorine by the potassium iodide solution contained in *a*, the upper layer of the liquid in the beaker remains colourless. After the distillation the liberated iodine is titrated in the usual manner.—B. B.

*Detection and Separation of Arsenic in the Presence of Antimony and Tin.* F. A. Gooch and B. Hodge. Chem. Zeit. 1894, 18, 156 (Rep.).

THE sample is dissolved in hydrochloric acid and potassium iodide, and the solution distilled nearly to dryness. The distillate is condensed in hydrochloric acid, and the colour due to the iodine discharged by adding a solution of chloride of tin in hydrochloric acid. The arsenic is then precipitated by passing sulphuretted hydrogen through the solution. By this method 0.0001 grm. of arsenic can be detected in the presence of 0.4 to 0.5 grm. of antimony or tin, and 0.003 grm. of arsenic separated by it.—B. B. P.

*The Analysis of Potassium Nitrate.* A. Hellich. Chem. Zeit. 1894, 18, 485—486. (See p. 947.)

THE author, in examining a sample of potassium nitrate which contained only a trace of chlorine as chloride, tested it for the possible presence of bromate by deluging it with sugar, dissolving the residue in water, acidifying with nitric acid and adding silver nitrate solution. A precipitate was obtained which proved to be silver chloride instead of bromide. Further trials with starch, cotton, and sulphur as reducing agents in place of sugar gave similar results. On fusing the nitre *per se* in a platinum dish for some minutes, no reaction with silver nitrate in the subsequent aqueous extract was observed, but on continuing the heating and raising the temperature of the fused salt a definite reaction for a chloride was obtained; this became more marked on adding manganese dioxide to the fused salt and

heating strongly. In the remainder of the investigation the only reagents used were manganese dioxide and sulphuric acid, the freedom from chlorine of which had been previously ascertained. The plan thus modified was carried out as follows:—10 grms. of the sample of nitre were mixed with 0.5 gm. of powdered manganese dioxide and the mixture was strongly heated until the melt had become green. It was then extracted with water (50 cc.), and to it 20 cc. of dilute sulphuric acid were added. The solution was allowed to stand and was then filtered through a filter previously extracted with dilute sulphuric acid. The filtrate when tested with silver nitrate gave a white turbidity. Twenty samples of nitre were examined by this method and chlorine recognised in all, the quantity varying from that corresponding with a slight turbidity to that represented by a curdy precipitate. Examination of the precipitate obtained from a number of samples showed it to be actually silver chloride. The deduction drawn by the author is that these samples of nitre contained some chlorine compound incapable of giving a reaction with silver nitrate. Organic substances containing chlorine are excluded, as such bodies would be broken up on fusing the nitre containing them, whereas the addition of manganese dioxide and strong heating are necessary to develop the full reaction. The absence of chlorates appeared assured from the fact that a portion of the nitre treated with strong sulphuric acid developed no coloration; direct experiment showed that this test suffices for the detection of 0.1 mgrm. of potassium chlorate in 1 gm. of nitre. Another proof of the absence of chlorate was afforded by slightly colouring the nitre solution with a solution of indigo and adding sulphurous acid, when the colour of the indigo was not discharged. The author concludes that the impurity in nitre which contains chlorine is a perchlorate, the quantity amounting to about 0.25 per cent. in a sample giving evidence of considerable contamination. Definite proof of the correctness of this view can best be obtained in a nitre refinery in which fractional separation by crystallisation of large quantities of nitre is feasible. Every sample of the salt hitherto examined has shown the presence of perchlorate, which exists even in those labelled "chemically pure." The reaction was also given by a sample of nitre some 20 years old made by an obsolete method of manufacture.—B. B.

*Examination of Artificial Manures.* Chem. Zeit. 1894, 18, 361—363.

This paper is a summary of the methods now in use in the German Experiment Stations.

*Preparation of Samples.*—One-half of the sample is prepared for analysis immediately after its arrival, whilst the remainder (up to 1 kilo.) is bottled and stored in a cool room for three months, in case it be required for reference. Dry samples are sifted and mixed; moist samples, can, of course, only be mixed. Raw phosphates and bone charcoal are dried at 105°–110° C. for the estimation of moisture; if ammonia be present the proportion of this lost at the temperature of drying is determined. In the case of substances which suffer a variation of their water-content during pulverisation, a determination of moisture must be made both in the original substance and in the powdered sample, and the results of the analysis must be appropriately connected.

*Estimation of Moisture in Superphosphates.*—Ten grms. are dried for three hours at 100° C.; the loss of weight is determined as moisture.

*Estimation of Soluble Phosphoric Acid in Superphosphates.*—Twenty grms. of the sample are shaken with 800 cc. of water in a litre flask for 30 minutes; water is added until the flask is filled to the mark and the liquid is again shaken, and filtered. The phosphoric acid is estimated in the filtrate either by the citrate method or by the molybdate method, the latter being alone admissible in arbitration cases. In the citrate method 50 cc. of the solution, thus prepared, are mixed with 50 cc. of citrate solution (see below) and 25 cc. of magnesia mixture are added; the liquid is shaken for half-an-hour and filtered through a Gooch crucible. The dried precipitate is heated for 3–5 minutes in a Rössler's furnace.

When a double superphosphate is under examination, the solution must be heated with nitric acid before the phosphate is precipitated, in order that any pyrophosphate which may be present shall be converted into orthophosphate. Twenty-five cc. of the solution, prepared as described above, are diluted with 50–75 cc. of water, 10 cc. of nitric acid (sp. gr. 1.4) are added and the solution is heated for one hour on the sand-bath, an excess of ammonia is then added and sufficient nitric acid to acidify the liquid. After cooling, the citrate and magnesia mixture are added in the manner already prescribed and the precipitate is heated for 10 minutes in the Rössler's furnace.

The citrate solution is prepared by dissolving 1,100 grms. of citric acid in water, adding 4,000 cc. of 24 per cent. ammonia and diluting to 10,000 cc.

In the molybdate method, 200 cc. of the molybdate solution (see following) are added to 5 cc. of the solution containing the phosphate, the mixture is digested for three hours at 50° C. on the water-bath, cooled, and the liquid poured through a filter. The precipitate is washed by decantation with diluted molybdate solution (1:1) until the washings no longer contain lime, and ammonia is poured through the filter paper into the beaker until the portion of the precipitate on the filter and that in the beaker are dissolved. The ammoniacal solution is nearly neutralised with hydrochloric acid and 20 cc. of magnesia mixture (550 grms. of crystallised magnesium chloride and 1,050 grms. of ammonium chloride dissolved in 3,500 cc. of 24 per cent. ammonia and 6,500 cc. of water) are added drop by drop, with continued shaking, followed by 25 cc. of 25 per cent. ammonia. After two hours the precipitate is filtered, washed with 5 per cent. ammonia until the washings are free from chlorides, and burnt as usual.

The molybdate solution is prepared by dissolving 1,500 grms. of ammonium molybdate in 10 litres of water, and pouring the solution into 10 litres of nitric acid (sp. gr. 1.2).

*Estimation of Insoluble and total Phosphoric Acid.*—Five grms. of the substance in a 500 cc. flask are dissolved in 50 cc. of *aqua regia* (three parts of hydrochloric acid of sp. gr. 1.12 with one part of nitric acid of sp. gr. 1.25), or are heated with 20 cc. of nitric acid (sp. gr. 1.42) and 50 cc. of sulphuric acid (sp. gr. 1.8) for half an hour. The solution is made up to 500 cc. with water and the phosphoric acid is estimated in 50 cc. by either the citrate method, or the molybdate method.

In the case of basic slag, 10 grms. are moistened with water and treated with 5 cc. of sulphuric acid (1:1) whereby the mass becomes hard. To this 50 cc. of concentrated sulphuric acid are gradually added, and the flask is heated on the sand-bath until thick fumes are evolved. Water is added before the flask has perfectly cooled, the cold liquid is made up to 500 cc. and filtered. The phosphoric acid is determined in 50 cc. by either the citrate method or the molybdate method.

*Estimation of the fineness of Basic Slag.*—This is carried out as usual.

*Estimation of Ferric Oxide and Alumina in Raw Phosphates.*—Five grms. are dissolved in 25 cc. of nitric acid (sp. gr. 1.2) and 12.5 cc. of hydrochloric acid (1.12), and the solution is diluted to 500 cc.; 100 cc. of the filtrate are brought into a litre flask, and 25 cc. of sulphuric acid (sp. gr. 1.84) and, after five minutes shaking, 100 cc. of alcohol (95 per cent.) are added. When this liquid has cooled alcohol is added to the mark, the flask is shaken and more alcohol is added to compensate for the contraction; after half-an-hour the solution is filtered, 100 cc. of the filtrate are evaporated in a platinum dish until the alcohol has been expelled, 50 cc. of water are added, the temperature is raised to the boiling point, and ammonia is added in excess, the excess being expelled by heat. The cooled liquid is filtered and the ferric oxide and alumina are washed with hot water, ignited and weighed.

*Estimation of Nitrogen.*—The nitrogen in blood-meal, Lone-meal, flesh-meal, and the like, is determined either by the Kjeldahl method or the soda-lime method. By the former, 0.8–1.5 gm. of the finely ground substance is heated in a flask with about 1 gm. of mercury and 20 cc.

of sulphuric acid (3 vols. concentrated sulphuric acid with 2 vols. fuming sulphuric acid) until the solution is clear. The liquid is transferred to a 400 cc. flask, 100 cc. of soda solution (32° B.) and 1-1.5 gm. of zinc dust are added, and the ammonia is distilled over as usual. The addition of potassium sulphide is unnecessary.

Nitric nitrogen in mixtures is determined according to the method of Schlosing-Grandeaun or that of Lunge. The total nitrogen is determined as follows:—1 gm. of the finely ground sample is mixed with 2-3 grms. of gypsum and introduced into the reaction flask; 25 cc. of phenol sulphonic acid are cautiously added and mixed by careful shaking with the substance, the flask being kept cool. After some time 1 gm. of mercury, and 3 grms. of zinc-dust are added; the flask is allowed to stand for half an hour and then heated until its contents are no longer coloured. The ammonia is then distilled over as usual.

Nitrogen in nitrates may be determined by the Böttcher method:—10 grms. are dissolved in 500 cc. of water; 25 cc. of the solution are mixed in a 400 cc. distillation flask with 120 cc. of water, 5 grms. of zinc-dust, 5 grms. of iron powder and 80 cc. of soda solution (32° B.). After the mixture has remained (connected with the condenser) at the ordinary temperature for 1-2 hours, during which time it has been frequently shaken, the ammonia is distilled over in the usual manner.—A. G. B.

## ORGANIC CHEMISTRY.—QUALITATIVE.

*Comparison of the Analytical Methods for the Detection of Margarine in Butter.* Seyda and Woy. Chem. Zeit. 18, 1894, 906.

**I. Koettstorfer's Method.**—By this method the saponification equivalent of the fatty acids in the butter is estimated. Using 2 grms. of butter and  $\frac{1}{2}$ -normal hydrochloric acid the maximum error of the titration is 0.05 cc. The analysis of 185 samples of butter gave the following results:—

From 221.5 to 223.....	4 per cent.	} 11 per cent.
" 223 " 225.....	7 " "	
" 225 " 226.....	7 " "	} 64 per cent.
" 226 " 228.....	15 " "	
" 228 " 230.....	21 " "	
" 230 " 233.....	28 " "	
Above 233.....	18 " "	

A very small proportion of these results falls below 225°, and a lower result than 221.5 has never been obtained from any pure butter. The saponification number of margarine is about 195.5; generally rather lower. Therefore a Koettstorfer number below 221 is sufficient proof of adulteration. Samples showing numbers from 221 to 225 may be considered doubtful, and 225 is the lowest number which should be accepted for pure butter.

**II. Reichert-Meißl's Method** is one by which the volatile acids contained in butter fat may be estimated. No other method has undergone so many alterations and additions, owing to the numerous sources of error inherent in it. There is a great uncertainty as to the minimum value, which by his method may be held to represent unadulterated butter, indeed the question as to whether a given sample is adulterated is always doubtful, unless the so-called Reichert-Meißl number be below 18. But in this case the Koettstorfer number will have been found so low as of itself to indicate the adulteration of the sample with margarine.

**III. Hehner's Method**, by which the percentage of fatty acids insoluble in water is estimated, is, in the opinion of the author, the most tedious of all and contains, the author considers, some considerable sources of error. The method, it is stated, will be most useful as confirmatory of the results of the two before-named tests; it should not be employed alone as a final and decisive test.—C. O. W.

## Detection of Castor-Oil in Olive-Oil. V. di Vetere.

Selmi, 1894, 4, 48.

OLIVE-OIL is sometimes adulterated with castor oil in spite of the apparent improbability of such a sophistication. 15-20 per cent. of castor oil is not recognisable by taste, and strong-smelling rancid olive oils appear to be sensibly improved by the addition. The methods for detection of the adulteration which are used by the author, are as follows:—10 cc. of the oil to be tested and 5 cc. of hydrochloric acid (sp. gr. 1.186) are shaken together in a test glass graduated to 0.1 cc. When castor-oil is present the liquid separates into three layers. The lowest is hydrochloric acid, the top layer consists of olive oil, and the middle stratum contains the castor oil. The colour of this middle layer varies according to the character of the oil with which it is mixed. The separation is not complete, but the size of the middle layer is nevertheless a fair index of the quantity of castor-oil present. The author has proved the utility of the method for sesame, cotton-seed, colza, earthenut, and linseed-oil as well as for olive-oil. Other methods applicable for confirming the results thus obtained, are the use of the oleo-refractometer of Amagat and Jean, and the determination of the turbidity temperature with acetic acid (Valenta's test). The results obtained with known mixtures of olive and castor oil are given in the following table:—

Olive-oil.	Castor-oil.	Per Cent.		Volume of the Middle Layer.	Degree of Oleo-refractometer.	Turbidity Temperature.
		Found.	Calculated.			
100	..	..	..	1		95° C.
..	100	..	..	44		{ Sol. at ordinary temp.
90	10	0.5	1.0	5		
85	15	1.2	1.5	7		85° C.
80	20	1.8	2.0	8.5		35° C.
70	30	2.9	3.0	12.5		25° C.
60	40	3.9	4.0	16		{ Sol. at ordinary temp.

—B. B.

## Detection of Castor-Oil in Balsam of Copaiba. L. Maupy. J. Pharm. Chim. 1894, 29, 362.

ADVANTAGE is taken of the fact that castor-oil yields sebacic acid and caprylic alcohol on dry distillation with excess of alkali. Sebacic acid is insoluble in cold, but soluble in hot water. The test is made by heating 10 grms. of the sample in a silver dish with dry caustic potash. The mass is stirred until volatile oily matters have been driven off, during which process the presence of castor-oil can be detected by the smell of caprylic alcohol. The mass separates into an upper resinous layer and a lower white liquid portion. After thorough separation the resin is removed and the lower layer treated with 50 cc. of water, boiled and filtered. On cooling this solution, sebacic acid separates, if the original sample contained castor oil. Further identification of the acid can be effected by converting it into its lead salt.—B. B.

## Behaviour of Strychnine towards Vitali's Atropine Reaction. G. P. Menyazzi. Boll. Chim. farm. 1894, 33, 103.

THE colour reaction discovered by Vitali, according to which the alkaloid (atropine) is oxidised by gently warming it with nitric acid, the yellow residue being moistened with alcoholic potash, a violet coloration being produced,—is also exhibited by strychnine and its salts.



The colorations produced by the two alkaloids can scarcely be distinguished. The alcoholic potash employed must be freshly prepared, colourless, and not too concentrated.

—A. R. L.

*Logwood Extract.* E. Donath. Chem. Zeit. 18, 277—278.

THE waste liquors from dyeing with logwood extract contain a certain proportion of nitrogen. As logwood extract is entirely soluble in water, the nitrogen compounds contained in it must be so also; hence these compounds are not fixed on the fibre and appear in the waste liquors. The analysis of two samples of the unadulterated commercial extract and of an extract prepared by the author, from rasped logwood which had been exposed to air for some time, gave the following results:—

	I. Extract of German Make.	II. Belgian Extract, Superior Quality.	III. Author's Extract, Dry.
	Per Cent.	Per Cent.	Per Cent.
Water.....	8.26	11.34	..
Nitrogen (calculated on ash.....) dry substance.	0.78	0.58	0.81
	6.39	4.99	1.33

If the nitrogen be expressed as protein substances, by multiplication by the protein factor 6.25, we have 4.87 and 3.62 per cent. respectively in the commercial extracts.

The large quantity of ash in the commercial samples consists chiefly of alkaline chlorides and phosphates; the extracts being made from "aged" wood, these salts result from the use of stale urine for sprinkling the wood during the "ageing" process.

The chief adulterants added to commercial logwood extracts are chestnut extract, sugar by-products (molasses), and gum. They may be detected as follows:—

*Chestnut Extract.*—An analysis of a sample of chestnut extract gave—

	Per Cent.
Water.....	14.48
Nitrogen (on the dry substance).....	1.28
Ash (on the dry substance).....	1.00
Reducing substances (calculated as glucose).....	31.40

Chestnut extract is much less readily decolorised by animal charcoal than logwood extract, the former also contains tannin, and gives a precipitate with gum solutions, which logwood does not. In testing the logwood extract its aqueous solution is decolorised, the presence of chestnut extract being indicated by a difficulty in decolorising, compared with a solution of an unadulterated extract under similar conditions. If the colourless solution gives a considerable precipitate with Fehling's solution the adulteration is almost certain. A further test, with gum solution, is only decisive when at least 7 per cent. of chestnut extract is present.

*Molasses.*—If the decolorised extract solution gives no precipitate, or a very slight one, with Fehling's solution, but after inversion with hydrochloric acid a copious precipitate of  $\text{Cu}_2\text{O}$  is thrown down, the presence of sugar by-products is evident.

*Glue.*—If the nitrogen present be over 1 per cent. adulteration with glue is probable. If glue be present to the amount of 8 per cent. or more, it is detected by extracting the dried and pulverised extract with strong alcohol, dissolving the residue in water, and precipitating with excess of lead acetate solution. The precipitate is filtered off and excess of lead removed by sodium carbonate. Filter, to remove lead carbonate, and to the filtrate add a tannin solution. The presence of glue is indicated by a precipitate or by considerable cloudiness.—R. B. B.

## ORGANIC CHEMISTRY.—QUANTITATIVE.

*Sources of Error in Roesse's Method of Estimating Fusel Oil in Spirit.* M. Glasenapp. Zeits. für Spiritusind. 1894, 17, 169.

THIS method consists in shaking spirit, brandy, &c. of 30 per cent. volume strength with a measured volume of chloroform, which takes up the fusel oil, and the increased volume of the chloroform is then observed. With careful attention to details, very exact results can be obtained.

To clean the burette used for the shaking, it should be treated with concentrated sulphuric acid, and allowed to stand at least one hour. If this precaution be neglected, drops of the alcoholic liquid stick to the tube, and get into the chloroform layer, thus increasing its volume.

Many spirits, especially raw spirits, contain  $\text{CO}_2$ , since, in the distillation of a mash, this gas passes off with the volatile portion, and is absorbed by the alcohol.

Now, when the fatty acids are to be estimated by titration with  $\frac{1}{100}$  normal alkali, the amount indicated is too great because of the alkali used in titrating  $\text{CO}_2$ .

The most important influence of the  $\text{CO}_2$  however is to increase the apparent volume of fusel oil present in the spirit by increasing the specific gravity of the alcohol and by decreasing that of the chloroform.

A sample of "automatically-refined" spirit, of sp. gr. 0.81263, contained 0.844 gm. of  $\text{CO}_2$ , estimated with lime water, followed by back titration with oxalic acid. On diluting to 30 per cent. with water, it apparently contained 0.0696 per cent. by volume of fusel oil, equivalent to 0.228 per cent. by volume in the original spirit. A second portion was boiled for one hour with inverted condenser in order to drive off the  $\text{CO}_2$ , after which the specific gravity had decreased to 0.81232, and the fusel oil, estimated in a 30 per cent. solution, was equivalent to 0.049 per cent. in the original spirit. This is, then, only  $\frac{1}{3}$  to  $\frac{1}{4}$  of that found before removing the  $\text{CO}_2$ ; since, however, during distillation any acetaldehyde contained in the fusel oil is lost, the  $\text{CO}_2$  was removed from a third portion of spirit by exact precipitation with lime-water and the fusel oil then estimated. It was found to represent 0.079 per cent. or only about  $\frac{1}{3}$  of that in the spirit containing  $\text{CO}_2$ .

Sell states that an increase of 1 per cent. by vol. of alcohol causes an increased volume of 0.03 cc. in the chloroform. To investigate this point further, dry  $\text{CO}_2$  was passed through alcohol of 30 per cent. vol. until the gravity was increased from 0.96564 to 0.96588 equal to 0.950 gm. of gas or 478 cc. at N.T.P. In order to reduce the gravity again to 0.96564 the author calculates that 0.14 cc. of absolute alcohol per 100 cc. is requisite, and the solution then contains 30.14 per cent. by vol., which according to Sell indicates an increase in the chloroform of 0.042 cc., equivalent to 0.028 per cent. by vol. of fusel oil in the 30 per cent. solution.

The influence of  $\text{CO}_2$  on chloroform is, on the other hand, to increase its volume and decrease its specific gravity. Dry  $\text{CO}_2$  was passed into chloroform for  $\frac{1}{4}$  hour, when the specific gravity became reduced from 1.48207 to 1.48055. The above alcohol, specific gravity 0.96588, containing  $\text{CO}_2$ , when shaken up with chloroform, increased 0.04 cc., and if the alcohol is first reduced to its original gravity of 0.96564 before shaking with chloroform an increase of 0.082 cc. results. Of this, 0.042 cc. is due to the action of  $\text{CO}_2$  on the mixture of alcohol and water, and the remaining 0.04 cc. is due to the increased vol. of the chloroform. The increase 0.082 cc. is equivalent to 0.54 per cent. of 40 per cent. spirit.

It is necessary, then, to remove  $\text{CO}_2$  before estimating fusel-oil by this method, and this can be done by exactly precipitating with lime-water and estimating fusel-oil in a 30 per cent. solution, or by gently boiling for at least  $\frac{1}{2}$  hour in a flask provided with a condenser 1 metre long, with or without the addition of caustic alkali. The latter method is quicker, but gives rather too low results, due to loss of aldehydes through volatilisation or neutralisation with alkali.

The author invariably finds fusel-oil, even in the purest spirit. The shaking burette of Dr. Karl Windisch is recom-

mended in preference to all others, since readings can be made to 0.005 cc. representing 0.0033 per cent. fusel-oil in 30 per cent. or 0.011 per cent. in 35 per cent. alcohol. For specific gravity estimation a pycnometer is used.—T. A. G.

*Gravimetric and Volumetric Methods for the Determination of the Alkaloids in Alkaloidal Tinctures.* E. H. Farr and R. Wright. *Proc. of the Pharm. Conference, 1894* (Oxford). *Chem. and Druggist*, **45**, 1894, 184—185.

THE authors in this note referred to the work done by Lyons, Lloyd, Allen, and Cripps in the direction of volumetric estimations of the alkaloids in tinctures, &c., and then proceeded to the work of Caspari, who, in a note published in the *American Pharmaceutical Review*, November 1892, maintained that volumetric methods of estimation were superior to gravimetric ones, and that the alkaloids obtained by the latter invariably contained from 10 to 20 per cent. of impurity. A final reference was made to a paper read before the American Pharmaceutical Association last year by Messrs. Caspari and Dohme, entitled "The Value of Titration with Volumetric acid Solution as a Means of Assaying Drugs and Galenical Preparations," in which those gentlemen claim to show that the determination of alkaloids in galenical preparations may be effected more accurately by titration than by the usual plan of weighing.

As Messrs. Farr and Wright in their researches on tinctures had employed gravimetric processes of assay almost exclusively, they considered it necessary to test the conclusions of Messrs. Caspari and Dohme. With that object in view each of the alkaloidal tinctures previously submitted to examination was prepared in sufficient quantity and estimated as follows:—

1. Two gravimetric estimations by the methods published in their notes on tincture menstrua.

2. The crude alkaloid was extracted from the evaporated tincture by shaking with chloroform after addition of alkali; it was then purified by extraction from the chloroform with several portions of acidulated water, and finally recovered by addition of alkali and shaking with chloroform. The chloroformic solution thus obtained was washed with distilled water to remove any traces of adhering alkali, and then used for the direct titration of the alkaloids with  $\frac{N}{20}$  HCl, methyl-orange, iodosin, and phloxin being used as indicators in the separate experiments. In using methyl-orange a little distilled water is added along with two drops of the indicator, but with iodosin and phloxin a single drop of a  $\frac{1}{1000}$  solution is shaken up with the chloroformic solution until the latter has become distinctly coloured. The indication of the end-reaction is in the case of methyl-orange the appearance of a pink colour in the upper layer, whilst in the case where iodosin and phloxin are used the reaction is complete when the colour disappears from the chloroform.

3. The alkaloid obtained by the usual gravimetric method was dissolved in a known excess of standard acid and titrated back with standard alkali in a white porcelain dish, using methyl-orange and Brazil wood as indicators, supplemented by iodosin and phloxin where the volume of tincture permitted. In this case the indication of the end of the reaction when methyl-orange is used is the disappearance of the pink tint; and with Brazil wood the production of a purple colour. In the employment of iodosin and phloxin as indicators, except in direct titration, we have followed the plan recommended by Cripps of adding to the acid alkaloidal solution sufficient neutral ether to form a distinct supernatant layer after being shaken with the solution. At the end of the reaction the aqueous layer becomes pink.

The following were the test-solutions used in the work:—Standard  $\frac{N}{20}$  hydrochloric acid and standard  $\frac{N}{100}$  barium hydrate, which is recommended as giving a quicker end-reaction than potassium or sodium hydrate. The strength of the latter must be taken at the commencement of each experiment. The indicators used were as follows:—

*Methyl-orange.*—A solution of the strength of 1 gr. per fl. oz. in equal parts of S.V.R. and water.

*Brazil Wood.*—The U.S.P. test solution, made by boiling 50 grms. of finely-cut Brazil wood with 100 cc. of water for half an hour, replacing the water evaporated from time to time; the liquor is then cooled, strained, and made up to 100 cc., and 25 cc. alcohol added, then the whole filtered.

*Iodosin and Phloxin.*—An aqueous solution containing 1 part in 1,000 fluid parts.

The equivalents used were those of the principal alkaloid in each case, except cinchona, nux vomica, and veratrum, where the equivalents are calculated by taking the mean weights of the chief alkaloid.

The results obtained are embodied in the table given on next page.

*Notes on the Results.*—The authors conclude from their work that volumetric methods are useless in the case of the alkaloids of acenite, the large proportion of acenine (equivalent 0.02715) making the readings much too high, also in the case of preparations of colchicum, on account of the absence of basic properties in the alkaloids colchicine and colchicine.

For gelsemium they have used the formula  $C_{21}H_{27}N_3O_6$ , proposed by L. Spiegel, instead of  $C_{21}H_{27}N_3O_8$ , as proposed by Gerrard, their results being more in accord with the former.

In the titration of the cinchona alkaloids the results obtained were not satisfactory, the end-reactions being extremely difficult to observe, and the authors condemn the use of volumetric estimations in the case of these alkaloids.

On the other hand, they consider that in the case of the alkaloids of belladonna, henbane, stramonium, conium, jaborandi, nux vomica, lobelia, and opium, the application of the volumetric methods may be made without disadvantage, the results obtained being almost equally reliable, and somewhat more expeditious than by gravimetric processes.

The authors give a general process for estimating the alkaloids in tinctures by titration with standard acid. From 25 to 50 cc. of the tincture (or in the case of hyoscyamine 100 cc.) to be estimated, is evaporated over the water-bath to expel spirit, a little water being added if necessary. The residual extract is filtered into a separator, and the dish and filter washed. The acid liquid is next shaken with two successive small quantities of chloroform, and the latter drawn off. The separated chloroform is washed with a little acidulated water, and the latter, after separation, added to the contents of the separator, which are then made alkaline, and the alkaloids taken out by shaking with three successive small quantities of chloroform. The chloroformic solutions are drawn off into a cylinder provided with a good cork and washed with distilled water until free from adhering alkali. A drop of  $\frac{1}{1000}$  iodosin or phloxin is then added, and the whole shaken until the chloroform is distinctly tinted; after this  $\frac{HCl}{20}$  is gradually run in from a burette graduated into tenths of a cc., the mixture being shaken after each addition of acid, until the colour is discharged from the chloroform. The reading is then taken, and the proportion of alkaloid calculated from the proper equivalent.

The authors, in conclusion, whilst admitting that the application of volumetric methods of estimation to the alkaloids obtained from tinctures is useful as affording a check on the results obtained by gravimetric methods, maintain that the results yielded by the latter are, on the whole, the more reliable of the two, and that gravimetric processes are more suitable for use by the average practical pharmacist.

*Estimation of the Insoluble, Non-Volatile Fatty Acids.* Meillère. *J. Pharm. Chim.* **29**, 60.

FOR the preparation and storing of the fatty bodies preparatory to analysis, they are melted and poured into test tubes, and after cooling are each protected by a layer of mercury from the action of the air, and kept in the dark. Oils are poured into test tubes drawn out at one end, the narrowed portion being closed with gypsum and a layer of mercury. Butter is melted on the water-bath, mixed with burnt gypsum, and extracted with ether;

TABLE showing COMPARATIVE RESULTS obtained in ESTIMATING TINCTURES (a) GRAVIMETRICALLY, (b) VOLUMETRICALLY.

Tincture.		Alkaloid by Weight. Mean of Two Estimations.	Alkaloid Indicated by Direct Titration of Chloroformic Solution with HCl 20			Alkaloid Indicated by Dissolving Crude Alkaloid in Excess Ba2HO 100 and Titrating back with			HCl 20
			Methyl- orange.	Iodeosin.	Phloxin.	Methyl- orange.	Brazil Wood.	Iodeosin.	Phloxin.
Aconite.....	1	0.013	..	0.018	0.019	0.020	..	0.018	0.018
	2	0.014	0.019	0.019	0.022	0.022	0.022	0.022	0.025
Belladonna.....	1	0.022	..	0.022	0.022	0.022	0.021	0.020	0.020
	2	0.031	0.033	0.032	0.034	0.031	0.032	0.032	0.030
Cinchona.....	1	0.074	..	0.144	0.145	0.078	..	0.072	0.071
	2	0.087	0.093	0.087	0.086	..	0.052	..	..
Conium.....	1	0.047	..	0.042	0.042	0.042	0.042	0.042	0.042
	2	0.019 (Hydrochloride).	0.017	..	..	..	..	0.016	0.015
Colchicum.....	1	0.024	..	..	..	0.005	..	..	0.005
	2	0.028	..	..	..	0.006	..	0.005	..
Gelsemium.....	1	0.019	..	0.018	0.019	0.019	0.017	0.018	0.018
	2	0.024	0.024	0.020	0.024	0.027	0.024	0.024	0.024
Hyoscyamus.....	1	0.005	..	0.005	0.005	0.005	0.005	0.005	0.005
	2	0.009	0.010	..	..	0.0085	0.008	0.008	0.0086
Jaborandi.....	1	0.020	..	0.018	0.018	0.017	0.017	0.017	0.017
	2	0.018	0.018	0.017	0.017	0.018	0.018	0.017	0.018
Lobelia.....	1	0.009	..	0.009	0.008	0.008	0.008	0.008	0.008
	2	0.009	0.008	0.009	0.009	0.007	0.008	..	..
Nux vomica.....	1	0.043	..	0.044	0.043	0.043	0.042	0.041	..
	2	0.066	0.066	..	..	..	0.066	0.066	0.065
Opium *.....	1	0.100	..	..	..	0.095	0.094	0.095	0.094
	2	0.100	..	..	..	0.099	0.099	0.097	0.099
Stramonium.....	1	0.011	..	0.011	0.011	0.011	0.011	0.010	0.010
	2	0.017	0.017	..	..	0.017	0.017	0.016	..
Veratrum viride.....	1	0.023	..	0.023	0.022	0.019	0.020	0.019	..
	2	0.027	0.027	0.020	0.023	0.023	0.021	0.024	0.019

\* For the opium estimations, a solution of anhydrous morphia, obtained in estimating the tincture by the B.P. process, was employed. This was dissolved in excess of  $\frac{N}{20}$  HCl.

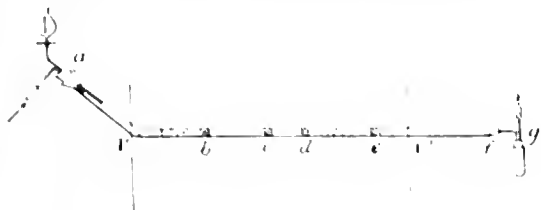
the ether is then partially driven off by evaporation. Milk is similarly extracted, or treated with Adams' solution. For saponification the fat is brought into a beaker, heated to 70° C. for one hour, and weighed. Five cc. of alcoholic potash (0.5 gram. in 5 cc.) are added, and the mixture again heated for an hour at 70° C. The saponification product is rinsed with hot water into a 2-litre flask, filled to the mark with cold water, and treated with 5 cc. of syrupy phosphoric acid. On vigorous agitation the fatty acids adhere together: they are then brought on to a weighed filter and dried at 70° C. until a constant weight is obtained. To estimate the volatile acids, the filtered acid liquid, after neutralisation, is concentrated to 110 cc., acidified with a non-volatile acid, 100 cc. distilled, and the acid titrated in the distillate. Butter gives on the average 87.7 per cent. of solid acids at 70° C., and butters from various sources show very slight variation in constitution.—R. B. B.

*Process for the Estimation of Small Quantities of Chlorine in Fats.* R. Benedikt and H. Zikes. Chem. Zeit. 18, 640—641.

THE authors were led to devise the method to be described, in view of the recent controversy as to the occurrence of chlorine in one kind of wool fat.

A hard glass tube, open at both ends, 100—110 cm. in length and 11—12 mm. internal diameter, is widened out at one end in the form of a funnel, and thereupon bent upwards at an angle of 45° at a distance of about 15 cm. from this end. An asbestos wad *b* is then inserted, and lime introduced in a layer of about 25 cm. in length, after which another asbestos wad *c* is inserted. A second layer of lime, 15—20 cm. long, is held in position by the wads *d* and *e*. The tube is now shaken in a horizontal position so as to form a canal, the spaces *v* and *v'* filled with pieces of porcelain, and the tube placed in a combustion furnace reaching from *v* to *v'*. The end *a* of the tube is fitted with a doubly-bored caoutchouc stopper. Through these borings pass the tube of a tap funnel bent at an angle of 45°, and a glass tube. A slow current of air is passed through the

latter, or, in order to avoid the formation of a trace of cyanogen, carbonic anhydride may be passed through it. At the end *f* of the tube, a test-tube is placed, in which



tar collects; the gas issuing at *g* is burned. The materials used for charging the tube must, of course, be free from chlorine. The asbestos employed is moistened with concentrated nitric acid, and, after volatilising the latter on the water-bath, it is washed with water by decantation and ignited. When lime free from chlorine is not obtainable, the amount of chlorine in that which is obtainable (not less than 100 grms.) is determined. The weight of the lime used, is then ascertained, and the results corrected accordingly.

The dropping funnel may conveniently be calibrated so that the quantity of fat introduced into the tube can be approximately determined. The tap funnel is charged with about 100 grms. of the fat, and its weight ascertained. After inserting the funnel, the tube is heated at a red heat from *b* to *c'*, but not between *c* and *b*: a gentle current of carbonic anhydride is passed through the tube, and the fat allowed to drop from the funnel at a rate of 8–10 drops per minute, or 7–10 grms. per hour. In the case of wool-fat, it is necessary to keep a small flame near the dropping funnel in order to maintain the fat liquid. The fat in the space between *v* and *b* is gradually warmed; the height of the flame at *g* is the indication of the speed of the distillation.

After the introduction of 25–50 grms. during 3–5 hours, the supply of fat is cut off until the flame *g* is extinguished. The gas flames of the furnace are now turned out, the lime transferred to a beaker, moistened with alcohol and water, and dissolved in dilute nitric acid. In the filtrate, the chlorine is estimated gravimetrically as silver chloride. When the operation has been properly conducted, the layer of lime between *d* and *e* should contain no chlorine.

Crude oleic chloride containing 21.39 per cent. of chlorine was mixed with technical oleic acid, and in this way mixtures containing known amounts of chlorine were prepared. The following values were obtained:—

	Chlorine found.		Chlorine calculated.
	I.	II.	
Mixture I. ....	0.0220	0.0235	0.0225
Mixture II. ....	0.0877	0.0869	0.0894
Mixture III. ...	0.235	..	0.232

—A. R. L.

*Technical Analysis of Ethereal Oils.* J. Klimont. Chem. Zeit. **18**, 641–642; 672–673.

THE author makes use of the property of ethereal oils of absorbing bromine as the basis of a method for their technical examination. The ethereal oil is diluted with chloroform and a solution of bromine in chloroform run in until the bromine is no longer absorbed, a point which is ascertained by the persistence of the brown colour. Misleading results are obtained by calculating the quantity of bromine absorbed by the amount of solution added or by titrating the excess, since on the one hand the solution of bromine in chloroform soon alters in titre, and on the other hand an excess of bromine is slowly absorbed by oil of turpen-

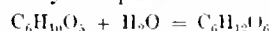
tine. As, however, in technical analyses theoretically accurate results are not required, the author makes a second test with pure terebenthene, prepared according to Ribau's directions (Bull. Soc. Chim. **5**, 6, 12) from oil of turpentine. By comparing the amounts of bromine absorbed in the two cases the terebenthene number of the ethereal oil is arrived at.—A. R. L.

*Colorimetric Methods of Estimating Carbohydrates.* Erich Reitzel. Zeits. für Spiritusind. 1894, **17**, 163.

THE known colour reactions obtained when sugar or other carbohydrate is mixed with sulphuric acid and an aromatic amine, alcohol, or phenol, are taken advantage of. A definite mixture is made, and the time which elapses until a given source of light can no longer be observed through the solution, is compared with the time required for a solution of known composition, or by reference to an empirical table. The method can be used for the examination of water from sugar factories, in beer and wine analysis, and for brewery and distillery purposes.—T. A. G.

*Quantitative Estimation of Carbohydrates.* E. Schulze. Chem. Zeit. **18**, 527–528.

ATTEMPTS are frequently made to estimate the amounts of dextrin or similar carbohydrates present in the extracts of various plants by heating these extracts with dilute sulphuric or hydrochloric acid, and determining the reducing sugar with Fehling's solution. The author points out that exact results are only obtainable when the process is carried out under standard conditions; very great errors are involved when the product is a mixture of carbohydrates. The causes which are generally admitted to underlie this matter are as follows:—The conversion of di- and polysaccharides into "glucoses" by the action of hot dilute acids proceeds with different speeds in various cases; even carbohydrates which exhibit the closest similarity in all other properties often show considerable differences in this respect. The hydrolytic decomposition of di- and polysaccharides, which is known as inversion, is invariably attended by more or less action in the opposite direction (reversion). When the "glucoses" are heated with dilute acids for a protracted period, they are decomposed, yielding humus substances and acids (levulinic acid, formic acid, &c.). This latter is particularly true in the case of levulose. Different "glucoses" reduce Fehling's solution to a different extent. When a carbohydrate yields only dextrose on inversion, somewhere near the theoretical amount of the latter is usually obtained; thus:—Meissl obtained 98.6 per cent. of the theoretically possible amount of dextrose from maltose; Winterstein obtained 99.17 per cent. of the theoretically possible amount from trehalose; whilst Allihn (J. prakt. Chem. 1880, **22**, 84) obtained by saccharifying starch with sulphuric acid only 94.5 per cent. of the dextrose indicated by the equation—



Other chemists (Sachsse, Chem. Centralbl. 1877, **8**, 732; Salomon, Jour. prakt. Chem. 1882, **25**, 348; Schulze, *ibid.* 1883, **28**, 311) have obtained larger amounts of dextrose by employing hydrochloric acid. When a carbohydrate yields on inversion levulose, either alone or in conjunction with another "glucose," an amount in any way approaching that demanded by theory is only obtained when, as in the case of saccharose, the inversion is effected by means of dilute acid and short duration of heating.—A. R. L.

*Estimation of Starch by Alcoholic Fermentation.* A. Munsche. Zeits. für Spiritusind. 1894, **17**, 202–203; **17**, 209–210.

ANALYSIS by physiological methods was suggested by Pasteur and used by Delbrück and Petzold (Zeits. für Spiritusind. 1882, 126). Jodlbaur (Zeits. f. d. ges. Brauw.), Hansen (*ibid.* 422), and others using pure cultivated yeasts. It has been shown by Delbrück that starch can be completely fermented in presence of diastase, whilst

Jodlbaur has established the fact that under proper conditions the last traces of fermentable sugars are removed by fermentation, and that the carbon dioxide and alcohol produced are constant quantities.

**The Carbon Dioxide.**—Air-dried potato starch was saccharified with malt extract, heated to destroy any organisms present, and fermented, after cooling, with pure cultivated top yeast. Species II.; Hayduck's solution (50 grms. potassium acid phosphate + 17 grms. magnesium sulphate per litre) and asparagin 0.4 gm. per 100 cc. being added. Fermentation was always completed in about 72 hours, as shown by Fehling's solution, the estimation being made in a control experiment carried on concurrently. The fermentation flasks were connected with a drying vessel containing concentrated sulphuric acid, followed by a calcium chloride tube, to retain any moisture or alcoholic vapour given off. The  $\text{CO}_2$  was calculated from loss of weight, the gas in solution being separately estimated. Tabulated results show that 100 parts of starch yield 51.70 to 51.29 parts of  $\text{CO}_2$ , which agrees well with Pasteur's  $\text{CO}_2$  value for dextrose (51.55). The amount of dissolved gas averaged 0.64 gm. Under equal conditions the same volume of liquid will dissolve the same amount of  $\text{CO}_2$ . In all cases correction was made for the malt extract used.

**The Alcohol.**—This was estimated in the distillate from the fermented liquid by means of a pycnometer, any alcohol retained by the sulphuric acid being separately determined. 100 parts of starch yield 53.43 parts of alcohol, as against Pasteur's figure 53.67 for dextrose, and Jodlbaur's figure 54.08.

The unfermentable extract, after inversion with hydrochloric acid, reduced Fehling's solution strongly. This is due to the gummy matters present which occur in cereals, and have been attributed by Lintner and Döll to galactoxylao, which with acid yields the reducing sugars galactose and xylose.

Briefly stated the results obtained are:—

(1.) The chief products of alcoholic fermentation are, under the foregoing conditions constant, as already shown by Pasteur and Jodlbaur.

(2.) In presence of malt diastase a vigorous fermentation with pure yeast will remove all traces of fermentable, reducing, starch products in about 60 hours.

(3.) 100 parts of dry, pure starch yield on fermentation 51.29 parts of carbon dioxide and 53.43 parts of alcohol.

It is hence possible to estimate starch quantitatively by fermentation in presence of diastase.

The "inversion" method gives too high results with vegetable substances because of the presence of half-soluble gummy bodies which yield reducing substances on inversion. These, being unfermentable, produce no error in the "fermentation" method.—T. A. G.

#### *The Estimation of the Acidity of Malt.* E. Prior, Bayer. Brauerjourn. 1894, 4, 74.

100 GRMS. of the malt are placed in 200 cc. of water or alcohol, and after 12 hours' standing the acid is determined in the filtrate. To prevent acid fermentation during this period through bacteria, chloroform water may be used and 14 hours allowed for extraction at ordinary temperatures. The analysis of a malt gave the following results:—100 grms. of the air-dry malt showed a total acidity requiring for neutralisation 37.0 cc. of  $\frac{1}{10}$  normal caustic soda, of which 3.0 cc. were neutralised by volatile organic acids, 5.9 cc. by non-volatile organic acid, 28.0 cc. by acid phosphates. Calculating the volatile acids as acetic acid, the non-volatile acids as lactic acids, and the acid phosphates as free phosphoric acid, the malt would contain 0.018 per cent. of acetic acid, 0.053 per cent. of lactic acid, and 0.199 per cent. of phosphoric acid.—C. O. W.

#### *Analytical Methods for the Valuation of India-Rubber Goods.* C. A. Lobry de Bruyn. Chem. Zeit. 18, 1894, 329.

1. **Extract Test** (Henriques' method).—3 grms. of the finely cut sample on being boiled for six hours with 50 cc. of a 6 per cent. alcoholic solution of caustic soda should

not lose more than 8 per cent., the loss to be calculated upon the organic substance of the sample only. The extract should contain sulphur and india-rubber resins only.

2. **Dry Heat Test.**—2 grms. of the finely-cut sample are heated to 135° C. for two hours. When cold the sample should not have suffered any alteration and should show a loss of weight not exceeding 1.5 per cent.

3. **Moist Heat Test.**—A small piece of the sample is sealed up in a glass tube filled to about one-half with water, and this tube is then heated to 170° C. during four hours. The sample should not have suffered by this treatment.

4. **Ash.**—From 0.5 to 1.0 gm. of the sample is first fused, decomposed, and partially converted to ash over a small flame. The lamp flame is then increased and the residue cautiously ignited in a porcelain crucible. See this Journal, 1893, 468.

Considering the deficiency of our present knowledge on the influence of the percentage of sulphur upon the durability of an india-rubber article, the maximum quantity of sulphur to be allowed cannot be fixed. The extractive matters allowed in india-rubber are only considered as regards the presence of rubber substitutes, the use of which should be altogether prohibited. The ash should be required to consist of zinc-white only, except in articles which come in contact with acids, where barytes or lead sulphate should be used. Red articles should be vulcanised with antimony sulphide and should not contain any ferric oxide.—C. O. W.

#### *Contributions to the Analysis of India-Rubber Articles, III.*

R. Henriques. Chem. Zeit. 18, 1894, 411.

In extracting india-rubber with a solution of caustic soda the rubber substance retains not inconsiderable quantities of caustic alkali, the weight of which must be determined in order to obtain correct figures for the percentage of extracted matters. The bulk of this alkali can, however, be removed from the extracted rubber by boiling the well-washed residue on extraction repeatedly with dilute hydrochloric acid. The analysis of a sample of india-rubber consisting of a mixture of rubber substitute or oil, india-rubber, and sulphur requires now the determination of the following data:—(a.) Total sulphur. (b.) Total ash. (c.) Weight of substance after extraction with alcoholic soda. (d.) Sulphur in c. (e.) Ash in c. (f.) Sulphur in the fatty acids extracted.

For the estimation of c, from 1.5 to 2 grms. of substance are used and the extraction is twice repeated, boiling each time for from two to three hours. The quantity of india-rubber dissolved by the alcoholic soda is deducted from the weight of the total extract. This correction amounts on the average to 2.5 per cent. From the above figures the percentage of india-rubber (x) and fatty acids (y) in the sample may be calculated by means of the following equations:—

$$x = \frac{100}{97.5} (c - d - e)$$

$$y = 100 - (a + b + x).$$

The quantity of sulphur contained in the rubber substitute is represented by f, assuming that quantity to be about equal to that of the fatty acids in the white substitutes and about 1.5 per cent. larger than the quantity of fatty acids contained in the brown substitutes. The sulphur of vulcanisation is found as the difference between total sulphur and sulphur in the substitute.

The following results were obtained in this manner:—

	I. India-Rubber with Brown Substitute.	II. India-Rubber with White Substitute.	III. India-Rubber with Oil.
	Per Cent. 19.46	Per Cent. 11.38	Per Cent. 21.55
a	0.72	0.40	0.40
b	68.37	67.68	63.40
d	2.64	1.51	3.06
e	2.70	2.81	3.17
f	16.90	9.27	3.20
Cl	None	Strong reaction	None

From this follows:—

	I.		II.		III.	
	Found.	Manufactured from.	Found.	Manufactured from.	Found.	Manufactured from.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Sulphur.....	16.13	16.4	9.19	9.6	21.55	23.5
Brown substitute.....	18.50	19.3	..	..	..	..
White substitute.....	..	..	25.43	25.8	..	..
Oil.....	..	..	..	..	29.42	17.7
India-rubber.....	64.65	64.3	61.98	64.6	57.63	58.8
Ash.....	0.72	..	0.40	..	0.40	..

Holde's method for the separation and estimation of oil and india-rubber by extraction with ether-alcohol is impracticable whenever the sample in question contains at the same time india-rubber substitute. This is owing to the considerable quantities of fatty matters which ether alcohol extracts from rubber substitutes. In the analysis of "recovered rubber," which frequently contains asphaltum, the separation of the latter from the india-rubber becomes desirable. This is effected by treating 1 gram. of the finely subdivided sample at ordinary temperatures, with 30 cc. of nitrobenzene. After one hour's standing the mass is filtered, the residue on the filter squeezed with a small pestle and washed with 30 cc. more of nitrobenzene. The washed residue is then boiled with water until all the adhering nitrobenzene is expelled, again filtered, dried, and weighed. The loss of weight thus found, however, is not entirely due to the presence of asphaltum in the sample, but is to about 1.5 per cent. accounted for by the slight solubility of india-rubber in nitrobenzene.—C. O. W.

#### Contributions to the Analysis of India-Rubber Articles.

R. Henriques. Chem. Zeit. 18, 1894, 412.

In estimating the percentage of asphaltum in vulcanised india-rubber articles by the nitrobenzene process, it is necessary to first remove the free sulphur from the sample. This is accomplished by the same means which serves for the estimation of the rubber substitute, *i.e.*, extraction with alcoholic caustic soda. No asphaltum is dissolved by this reagent, although besides the free sulphur all the rubber-substitute and about 3 per cent. of the rubber substance enter into solution. While thus the presence of fatty oils and rubber substitute does not prejudice the estimation of the asphaltum, it is practically rendered impossible by the presence of mineral oils, paraffin wax, and resins. The estimation of lampblack in india-rubber samples containing as further admixtures only oils, rubber substitute, and asphaltum (which are extracted by use of the already-described methods), can be accomplished by a combustion analysis. All reliable analyses of india-rubber indicate the relationship C:H=10:16, so that an admixture of carbon with the rubber can be estimated with satisfactory accuracy from the altered relation of C:H. In applying these new methods to the analysis of some samples of "recovered rubber," the following figures were obtained:—

	A.		B.		C.	
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Rubber substitute and oils.....	56.78	52.24	56.24			
Asphaltum.....	9.74	10.81	8.98			
Lampblack.....	0.61	0.65	1.38			
India-rubber.....	32.87	36.30	33.40			

The analysis of goloshes (St. Petersburg product) was carried out in the following manner:—

1. One gram. of the finely-ground sample was decomposed in a Geissler apparatus and the total gases ( $\text{CO}_2 + \text{H}_2\text{S}$ )

evolved, were weighed. The contents of the apparatus were then filtered and the undissolved residue repeatedly boiled with dilute acid. The bases (lime, lead, and a little iron) were almost completely dissolved. No sulphuric acid was found in the solution which was used for the estimation of the metals.

2. In the insoluble residue, the sulphur was determined in the manner already described (this Journal, 1893, 467). A small quantity of silica, alumina, and lead sulphate, was obtained at the same time.

3. The total sulphur was estimated in 1 gram. of the original substance. The difference between the percentage of sulphur thus found and that found in 2, represents the sulphur evolved as sulphuretted hydrogen, and by subtraction of the latter from the total gases evolved the quantity of carbonic acid evolved, was obtained; it was found to be equivalent to the quantity of lime observed.

4. Five grms. of the original substance were boiled with acid, filtered, washed, dried, and weighed. Of the substance thus obtained 2 grms. were extracted with alcoholic soda. The extract contained 2½ per cent. of india-rubber; the rest consisted of fatty oils. In the residue from this extraction the asphaltum was estimated by extraction with nitrobenzene, and in the residue thereby remaining the percentages of india-rubber and lamp-black were determined by combustion. The results of the analysis were:—Lead oxide, 8.45; calcium carbonate, 47.81; ferric oxide and alumina, 1.70; silicic acid, 0.44; sulphur, 1.50; fatty matter, 0.95; asphaltum, 8.46; lampblack, 0.39; india-rubber, 30.30 per cent.—C. O. W.

#### Analytical Control of the Vulcanisation of India-Rubber.

R. Henriques. Chem. Zeit. 18, 1894, 701.

The question as to the quantity of sulphur chemically combining with the india-rubber in the process of vulcanisation is still undecided, chiefly owing to the fact of no reliable method being known for the separation of the free sulphur from the combined sulphur in india-rubber articles. Alcoholic soda extracts free sulphur from vulcanised india-rubber, and it appeared, therefore, desirable to test the suitability of this method for the purpose in view. Three samples of best vulcanised Para rubber were experimented upon. Before and after treatment with alcoholic soda they showed the following percentages of sulphur:—

	A.		B.		C.	
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Total sulphur.....	6.44	7.75	8.24			
Combined sulphur.....	3.30	1.86	3.39			

It is evident that a sample of vulcanised india-rubber consists of a really vulcanised compound and possibly an unvulcanised proportion of india-rubber and free sulphur. The two last-named components being readily soluble in various solvents, the isolation of the vulcanised rubber might be effected by treating it with a suitable solvent until

the percentage of sulphur in the undissolved residue remained constant, or until the percentage of sulphur in the latest fraction of the extract is the same as that in the residue. Carbon bisulphide was used for the extraction, the sample being repeatedly treated with the solvent in the cold. Eight subsequent extractions gave the following results:—

Extract No.	Per Cent. of Previous Operation.	Per Cent. of Original India-Rubber.	Sulphur in undissolved Residue.
1	4.39	4.59	4.66
2	3.91	3.72	3.91
3	3.40	3.95	3.77
4	7.79	6.76	3.85
5	6.71	5.37	3.81
6	2.90	2.16	3.70
7	3.92	2.87	..
8	3.71	2.58	3.72

This shows that three extractions are sufficient to remove all uncombined sulphur.

The first two of the above extracts contain chiefly sulphur, with very little organic matter.

The following fractions consist of a grey spongy mass easily soluble in petroleum ether, but invariably leaving a slight residue of sulphur. This indicates a partial dissociation of the vulcanised india-rubber during the process of extraction. On heating the above-mentioned solutions in petroleum-ether for a short time to 100° C. revulcanisation takes place. The composition of that part of the three samples of vulcanised india-rubber, which is insoluble in carbon bisulphide, was found to be:—

—	A.		B.		C.	
	Before	After	Before	After	Before	After
	Extraction.		Extraction.		Extraction.	
Ash	1.16	1.89	2.74	2.81	1.52	1.77
C	76.49	77.70	76.22	80.23	77.50	80.31
H	9.79	10.31	16.73	11.82	11.26	11.47
S	6.44	3.72	7.75	1.82	8.24	3.59
O	6.12	6.47	2.56	3.82	1.48	2.86

This shows the following percentages of free sulphur:—

	A.	B.	C.
	Per Cent.	Per Cent.	Per Cent.
Extr. with alcoholic soda .....	3.39	1.86	3.39
Extr. with carbon bisulphide .....	3.72	1.72	3.59

These figures leave no doubt that, by treatment with alcoholic soda, the quantity of free sulphur in vulcanised india-rubber may be estimated.—C. O. W.

*Contributions to the Analysis of India-Rubber Articles.*  
R. Henriques. Chem. Zeit. 18, 1894, 905.

TERRY and Lobry de Bruyn expressed doubt as to whether the accuracy of the estimation of oils and rubber substitute in india-rubber articles was not affected if no distinction was made between those compounds and the acid resinous bodies which occur naturally in every india-rubber. This

question was tested by submitting various samples of vulcanised india-rubber to the alcoholic soda test. The results were:—

	Sulphur.	Ash.	Residue from Extraction with Soda.	Ash in Residue.	Resins Dissolved.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Para.....	6.43	1.16	91.90	2.92	2.28
Columbian .....	9.17	1.68	91.08	5.04	1.87
Ceara .....	11.69	2.75	91.61	6.16	1.78
Mozambique ..	11.86	2.69	91.27	5.93	2.05
Sierra Leone..	7.44	2.20	97.42	8.38	3.13
Madagascar...	11.77	1.80	90.39	5.82	3.58
Borneo.....	12.21	2.60	92.78	7.90	2.63
Accra.....	11.37	3.30	92.90	9.72	3.86

Particularly striking in these figures is the considerable increase of the ash found in the lower qualities of india-rubber after the extraction. The cause of this is to be found in the formation, during the extraction, of sodium salts of the resinous constituents of india-rubber. These sodium salts are soluble in the alcoholic extract, but after evaporating the alcohol and dissolving the residue in water, these particular sodium salts separate out as insoluble masses, which of course are filtered off and added to the extracted sample with which they are weighed together. This shows that the resinous constituents of india-rubber in no way prevent the correct estimation of the fatty oils and rubber substitute.—C. O. W.

*The Analysis of India-Rubber Articles.* C. A. Lobry de Bruyn and F. H. van Leent. Chem. Zeit. 18, 1894, 309.

See under XIII. C., page 963.

## New Books.

*RASA-RANGA-RAHASYA.* The First and only Monthly Magazine of the Chemical and Tinctorial Trades. Editors: M. G. DESHMUKH, B.Sc., B.A. M.D., and T. K. GAJJAR, B.Sc., M.A. Baroda. Bombay: Printed at the Education Society's Steam Press. 1894. Price Rs. 9 inland, 12s. overland, post free, per annum.

THIS is a new journal, in Hindustani, treating of Chemical Industry; more especially devoted to the tinctorial trades, however. It is issued from Baroda, but printed in Bombay. This the first issue (April 25, 1894) is of quarto size, and the subjects treated of in it are the following:—"A new System of Chemical Notation and Nomenclature." "Elements of Chemistry." "The Manufacture and Industrial Value of Aluminium Alloys." "Manufactures wanted in India." "Removal of mechanically-absorbed Water." "The Causes and Prevention of Spontaneous Combustion." "The most recent Development in Artificial Indigo." "Review of Colours." "New Colouring Matters." "Monthly List of Indian Patents." "Imports, Bombay Presidency; Exports, ditto." "Monthly Price List of Chemicals and Drugs."



**HANDWÖRTERBUCH DER PHARMACIE.** Herausgegeben von A. BRESLAWSKI. Wien und Leipzig: Wilhelm Braumüller, K. u. K. Hof- und Universitäts-Buchhändler, 1894. London: H. Grevel and Co., 33, King Street, Covent Garden.

Is continuation of Vol. II. of this Dictionary of Pharmacy, Parts 17 and 18 now appear. Part 17 commences with "*Pastellfarben*," and ends with "*Polykohleensäure*," which subject is completed in this part. Part 18 commences with "*Polymer*" and ends with "*Rhodium*," continued into and completed in Part 19, yet to appear. Parts 17 and 18 contain 79 pages of subject-matter each.

**DESCRIPTIVE CATALOGUE OF ESSENTIAL OILS AND ORGANIC CHEMICAL PREPARATIONS.** Compiled by FREDERICK B. POWER, Ph.G., Ph.D. Published by Fittsche Brothers, New York, U.S. America. H. Grevel and Co., 33, King Street, Covent Garden, London. 5s.

This little book is strongly bound, of 8vo size, and contains preface and 96 pages of subject-matter. An alphabetical index is unnecessary as the substances described are arranged in the text in alphabetical order.

In PART I. appear the OFFICIAL ESSENTIAL OILS, recognised by the Pharmacopœia of the United States of America, 7th Decennial Revision, 1890. PART II. contains the NON-OFFICIAL ESSENTIAL OILS, not recognised by the U.S. Pharmacopœia, 7th Decennial Revision, 1890. Many of these, however, are stated to be of commercial importance, whilst others are to be regarded at present as possessing chiefly scientific interest. PART III. contains the ORGANIC CHEMICAL PREPARATIONS. An interesting and, for many, a useful feature in the work is the setting in small type and in brackets, under the English names of oils or drugs, &c., the corresponding German names, thus combining the character of a glossary with the other features of the work.

**WATTS' DICTIONARY OF CHEMISTRY.** Revised and entirely rewritten by M. M. PATTISON MUIR, M.A., Fellow and Praelector in Chemistry, of Gonville and Caius College, Cambridge, and H. FORSTER MORLEY, M.A., D.Sc., Fellow of University College, London, and Lecturer on Physics and Chemistry at Charing Cross Hospital Medical School. Assisted by Eminent Contributors. In Four Volumes. Vol. IV., with Addenda. London: Longmans, Green, and Co.; and New York: 15, East 16th Street. 1894. 3l. 3s.

This is the final volume of this Dictionary of Chemistry. It commences with an *Introduction to the Articles on Inorganic Chemistry*, by Mr. Muir, followed by an *Introduction to the Articles relating to Organic Chemistry*, by Dr. H. Forster Morley; these introductions are followed by a List of Special Contributors, and a List of Abbreviations.

The work of the Dictionary itself commences with *Phenyl-Azazole*, and concludes with *Thallium tri-iodide*, the matter within the limits thus indicated, filling 922 pages. The article on "Physical Methods used in Chemistry," alone fills 101 pages. It is the joint work of Messrs. Capstick, W. N. Hartley, and W. Ostwald. Twelve and a half pages are devoted to "Solutions," by Messrs. Arrhenius and Spencer Pickering, and six to "Specific Volumes" by T. E. Thorpe. An important article on "Sugar" (36 pages), appears from the pen of C. O'Sullivan. The Terpenes are treated of by W. A. Tilden in an article occupying twelve pages. M. M. P. Muir has written a full account of Vanadium and its compounds, in a report filling sixteen pages. At the close of the volume, and of the work consequently, is a List of Addenda, referring to descriptive Inorganic Chemistry only. (See this Journal, 1892, 552.)

## Trade Report.

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

(From the Board of Trade Journal.)

#### RUSSIA.

##### Customs Decisions.

The following decisions affecting the classification of various pharmaceutical products in the Russian Customs tariff have recently been given by the Russian Customs authorities:—

Disinfectants known as Britannia and Ichtyolum are exempt from import duty, as provided by Article 112.

The importation of the following pharmaceutical products is, however, prohibited:—

Vaucher anti-choleric pastilles.

P. Florent coen.

Bombelon and Dengel ergotinum.

Sanitas preparations.

P. Florent glycine.

#### SWITZERLAND.

##### Customs Decisions.

The following decisions affecting the classification of articles in the Swiss Customs tariff have recently been given by the Swiss Customs authorities in the period extending from January to August 1894:—

Metallic cement is removed from Category 15 and placed in Category 352, paying a duty of 80 cents per quintal.

Sulphate of barytes, ground, washed, in paste.—Category 45. Duty, 30 cents per quintal.

Antimonic acid (acidum stibiosum, oxide of antimony).—Category 48. Duty, 1 franc per quintal.

Mannocitine (preservative against rust, composed of vaseline and essence of turpentine).—Category 66. Duty, 1 franc per quintal.

Chloride of ethyl (Dutch oil).—Category 72. Duty, 2 francs per quintal.

Polysulphine (substance for bleaching and for lyes).—Categories 76 and 77. Duties, 1.25 francs and 2.50 francs per quintal.

Manganese in a crude state is exempt from duty, under Category 328.

Sulphate of barytes in a crude state is exempt from duty, under Category 332.

#### FRANCE.

##### Customs Decisions.

The following decisions affecting the classification of articles in the French Customs tariff have recently been given by the French Customs authorities:—

Phosphate of lime obtained in the manufacture of gelatine, and commercially known under the name of precipitated phosphate, and used as a manure, is exempt from duty, under Category 281 bis.

#### GUATEMALA.

##### Tariff Changes.

Mr. Audley Gosling, Her Majesty's Minister at Guatemala, in a despatch to the Foreign Office, dated the 18th August, forwards copy and translation of a notice issued by the Guatemalan Government, according to which the import duties on the following descriptions of acids have been reduced from 15 to 5 cents per kilogramme:—

Sulphuric acid, sulphurous acid, carbonic acid, sulphydric acid, phosphoric acid, hypophosphoric acid, fluorhydric acid, hydrofluosilicic acid, nitric acid, muriatic acid, nitromuriatic acid, and any other mineral acids not classified as anhydrous, or in solution at whatever degree of concentration, in any kind of common receptacle, gross weight per kilogramme.



## THE JAPANESE DRUG TRADE AND THE NEW TREATY.

A treaty of commerce and navigation between Japan and the United Kingdom, signed in London on July 16, has only just been made public in this country through being published in full in a British newspaper published in Yokohama. The principal feature of the treaty, from the point of view of our general relations with Japan, is that it removes, so far as British subjects are concerned, the restrictions upon foreigners trading and settling in Japan, and places our countrymen upon a footing of absolute equality with the Japanese with respect to these pursuits. On the other hand, the right of foreign subjects in Japan to be tried by a court composed of officials of their own nationality is abolished, and foreigners will in future be amenable to the native courts of justice, on the same footing with the natives of the country. By virtue of the provisions of the "Protocol" which accompanies this treaty the Japanese Customs tariff undergoes considerable modifications. These changes, unlike the treaty itself, come into operation at once, and are applicable to all goods now imported into Japan. The two countries agree to give to each other the benefit of any advantages which they may hereafter grant

to other countries in the matter of trade, and Japan undertakes that her new provisional tariff, which provides for *ad valorem* duties of from 5 to 10 per cent. upon most of the staple articles of import, is to be replaced as soon as possible by another, with a specific-duty basis. The provisional new tariff makes such articles as mercury, paraffin-wax, saltpetre, and condensed or desiccated milk dutiable at the rate of 5 per cent. *ad valorem*; mercury, paraffin oil, paint in oil, and dry indigo at the rate of 10 per cent. *ad valorem*. There is no mention in the official protocol of any alteration in the duties on drugs and medicines, but it is not unreasonable to assume that it is the intention of the Japanese Government, in the final tariff which is in project, to raise the duties upon several of these articles, on especially such as it is possible to manufacture in the country. Last year Japan imported 446,000*l.* worth of "drugs and chemicals," of which 219,000*l.* worth came from the United Kingdom, 150,000*l.* from Germany, and 30,000*l.* from China; other countries counting for very little in the direct supply of articles of this class.

The following official Japanese statistics, give an interesting detailed view of the respective shares of the principal foreign nations in the Japanese drug market:—

Article.	Total Import into Japan.			Imported in 1893.	
	1893.	1892.	1891.	From Britain.	From Germany.
Acid (carbolic)..... C.	..	..	..	73,781	11,250
" (salicylic)..... C.	..	..	..	6,804	48,502
" (tartaric)..... C.	..	..	..	17,638	5,040
Alcohol..... C.	..	..	..	31,843	4,630,709
Bleaching powder..... C.	..	..	..	5,000	..
Cinchona..... C.	..	..	..	6,140	..
Cinchonia..... C.	..	..	..	225	2,512
Cod-liver oil..... Doz.	..	..	..	325	130
Glycerin..... C.	..	..	..	9,610	112,212
Morphia..... Oz.	9,610	10,424	12,730	..	..
Opium (medicinal by Government)..... C.	1,323	1,157	1,860	..	..
Phosphorus (amorph.)..... C.	130,475	89,385	100,200	106,850	23,625
Potassium bichromate..... C.	20,735	42,776	42,322	7,644	19,571*
" chlorate..... C.	2,490,596	1,543,350	1,224,748	2,321,268	84,154†
" iodide..... C.	7,711	14,844	22,160	1,050	6,651
Quinine..... Oz.	193,309	147,907	109,800	68,309	64,000
Rhubarb..... C.	111,061	121,543	129,008	..	..
Santonin..... C.	6,307	5,009	4,987	300	6,007
Sarsaparilla..... C.	806	5,140	3,612	..	..
Soda (bicarb.)..... C.	2,577,520	2,375,476	1,760,649	2,577,420	99
" (anhydric)..... C.	5,005,580	4,767,150	7,404,579	4,755,374	210,216
" crystal..... C.	11,947	5,777	778	5,301	.. ‡
Sandal-wood..... C.	13,022	63,823	35,492	..	..
Wormseed..... C.	16,777	80,275	38,957	..	16,677
Drugs unenumerated..... Yen	130,922	103,714	94,218	27,478	9,818
Chemicals unenumerated..... Yen	738,680	532,918	399,142	310,086	329,003
Total..... Yen	3,300,451	2,493,556	2,225,766	..	..

The sign C. means cattie, of 1½ lb. English.

The sign Yen means silver yen, nominally 4s. 2d.

\* 2,520 cattie came from the United States.

† 25,174 cattie came from France.

‡ 6,616 cattie came from France.

It will be seen that the great increase in the value of the drug imports into Japan is principally due to her growing requirements of a few heavy chemicals used in manufacturing industries, such as phosphorus and chlorate of potash.

These articles are mainly supplied by this country, but in many varieties of medicinal chemicals Germany has taken the lead in the supply. This is partly due to the fact that increased familiarity with European sources of supply has

enabled the Japanese to go directly to the fountain head for their requirements, instead of, as formerly, giving the whole of their orders to firms in England to place where those houses thought fit. Another reason may lie in the discrimination in favour of non-British goods with which the Japanese Customs officers are sometimes charged. The Japanese Pharmacopœia is framed upon the model of the German one, but in respect to certain articles it is far more stringent than any European work of the kind, and even the best qualities of certain drugs made in Europe fail to reach its extravagant standard. Now, the Japanese Customs law gives to the authorities of the country the right to "restrict or prohibit the importation of adulterated drugs, medicines, food, or beverages," and it is freely said that the Anglo-phobe officials who are charged with the enforcement of this clause have systematically exceeded it in spirit by refusing admittance to British goods which would pass muster in any European country. The prohibition clause, we may add, has expressly been maintained in the new treaty. It is to be hoped that it will be applied in future with more partiality and less excess of zeal.—*Chemist and Druggist*.

#### THE NEW JAPANESE TARIFF.

The following articles of the tariff deal with the metal and allied trades:—

Articles.	<i>Ad valorem</i> Rates of Duty.
	Per Cent.
Cement, Portland.....	5
Glass, window, ordinary:—	
(a.) Uncoloured and unstained .....	8
(b.) Coloured, stained, or ground.....	10
Iron and steel:—	
Pig and ingot.....	5
Rails .....	5
Bar, rod, plate, and sheet .....	7½
Tinned plates.....	10
Galvanised sheet.....	10
Pipes and tubes.....	10
Lead, pig, ingot, and slab.....	5
Mercury or quicksilver .....	5
Nails, iron .....	10
Oil, paraffin.....	10
Paint in oil.....	10
Saltpetre .....	5
Screws, bolts, and nuts, iron .....	10
Tin:—	
Block, pig, and slab.....	5
Plates .....	10
Wax, paraffin .....	5
Wire:—	
Telegraph .....	5
Iron and steel, and small rod iron and steel, not exceeding ½ inch in diameter.	10
Zinc:—	
Block, pig, and slab.....	5
Sheet .....	7½

#### Rule for Calculating *ad valorem* Duties.

Import duties payable *ad valorem* under this tariff shall be calculated on the actual cost of the articles at the place of purchase, production, or fabrication, with the addition of the cost of insurance and transportation from the place of purchase, production, or fabrication to the port of discharge, as well as commission, if any exists.—*Ironmonger*.

#### NEW UNITED STATES TARIFF.

##### *Free List.*

Acids, n.s.p. in A. T.	Iron, sulphate of.
(Including hydrochloric, nitric, carbolic, acetic, arsenious, oxalic, picric, tartaric, &c.)	Kelp.
Albumen.	Kieselguhr.
Alizarine.	Kaimo.
Alizarine dyes, natural and artificial.	Lime citrate.
Aniline salts.	Lithons.
Antimony ore and metal.	Magnesium sulphate.
Antimony sulphide.	Magnesium carbonate native.
Apatite.	Magnesium.
Argols.	Manganese oxide and ore.
Arsenic.	Manure and all substances expressly used for manure.
Arsenic sulphide.	Myrabolans.
Arsenate of aniline.	Ochres, n.s.p. in A. T.
Asbestos, unmanufactured.	Oil-cake.
Baryta carbonate.	Oil, aniline.
Baryta.	„ cotton-seed.
Barytes, unmanufactured.	„ croton.
Bauxite.	„ palm.
Bismuth.	„ cocoa-nut.
Bleaching powder.	Paper stock, crude, all kinds for paper-making only.
Blood, dried.	Paraffin.
Bones, crude, calcined, ground, or steamed.	Petroleum (subject to R.T.).
Bone-dust for fertilizing only.	Phosphates, crude.
Bone-ash for fertilizing only.	Pitch—coal-tar.
Bromine.	Platinum, unmanufactured.
Burgundy pitch.	„ manufactured only for chemical uses.
Cadmium.	Plumbago.
Calamine.	Potash, crude.
Camphor, crude.	„ carbonate.
Chalk, unmanufactured.	„ caustic, including refined.
Chromate of iron.	„ nitrate, crude.
Cinchona barks.	„ sulphate, refined and crude.
Coal tar, crude.	„ chlorate.
Coal-tar preparations (except medicinal preparations and products), not colours or dyes, n.s.p. for in A. T.	„ chloride.
Cobalt and cobalt ore.	Pumice.
Cod oil for leather dressing, &c., only.	Pyrites (exceeding 20 per cent. sulphur).
Copper sulphate.	Salt, in bulk (subject to R.T.).
Cryolith.	Salt, packed (packages pay duty), subject to R.T.
Cudbear.	Sodium.
Cutch.	„ nitrate.
Divi divi.	„ chlorate.
Emery ore.	„ sulphate.
Farina.	Sulphate of lime, unground.
Feldspar.	Sulphur, all kinds.
Flints.	Sulphuric acid (subject to R.T.).
Grease and oils (n.s.p. in A. T.) for soap-making and wire-drawing only.	Tallow and degrass.
Guano.	Tar and pitch—wood.
Hoofs, unmanufactured.	Terra alba.
Horns and horn tips, un- manufactured.	Turmeric.
Indigo, and extracts of.	Turpentine, spirits of.
Iodine, crude, and re- sublimed.	Uranium, oxide and salts.
	Valonia.
	Verdigris.

—*Chemical Trade Journal*.

#### *Government Report.*

REPORT FROM THE SELECT COMMITTEE ON PETROLEUM,  
TOGETHER WITH THE PROCEEDINGS OF THE COMMITTEE,  
MINUTES OF EVIDENCE, AND APPENDIX. 214. Price  
10½d.

This is the report of the Select Committee of the House of  
Commons appointed to inquire into and report upon the

law relating to the keeping, selling, and conveyance of petroleum and other inflammable liquids, including the precautions to be adopted to prevent the sale of dangerous lamps for use with inflammable liquids.

The Committee state in their report that they are of opinion that considering the late period of the Session it is not in their power to conclude their investigation; they have therefore agreed to report the evidence already taken to the House, and to recommend that a Committee on the same subject should be appointed in the next Session of Parliament.

The Select Committee was appointed on the 28th of June 1894, and the present report is dated the 27th of July of the present year.

### EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

#### COTTON-OIL INDUSTRY IN SOUTH CAROLINA.

In a report to the Foreign Office, dated the 10th September, Mr. A. Harkness, Acting British Consul at Charleston, transmits the following information on the subject of the cotton-oil industry in the State of South Carolina:—

The cotton-seed oil business in South Carolina is an industry of comparatively recent growth, the first oil mill established in this State having been a small one built near Columbia in 1868, which was not successful owing to the unsettled business conditions generally prevailing at that time. Later on, however, other mills were established, and they have, with few exceptions, proved to be profitable investments. In the year 1867 there were only four cotton-seed oil mills in the United States, but at the present time there are 253 throughout the country, of which number 27 are situated in South Carolina, 21 being owned by local capital, many of the stock-holders being farmers. The above-mentioned 27 mills now in this State have a capital of over 1,000,000 dollars (200,000*l.* sterling), and last year the Carolina mills crushed about 75,000 tons of cotton-seed, valued at 800,000 dollars (160,000*l.*), producing 60,000 barrels of oil, 26,000 tons of cotton-seed meal, 5,000 bales of linters, and 25,000 tons of hulls. The oil is most sold in northern and western markets, and is much used in the manufacture of compound lard, soaps, and for other purposes.

Part of the shipment to northern markets is exported to foreign countries, and later on some of it comes back to this country under fancy labels as olive oil.

The refined cotton-seed oil appears to be rapidly coming into favour for cooking purposes, either in its natural condition or compounded with other preparations now on the market. It is a sweet and wholesome product—a pure vegetable oil—and is regarded by many persons as being far superior to hog's fat for culinary purposes. The South Carolina farmers also use the cotton-seed meal largely for fertilizer uses, applying it directly to the soil or composting it with acid phosphate. The meal and hulls are also now used by stockmen and dairymen as an article of food for fattening cattle, considerable quantities having been consumed in this manner during the last two or three years.

The cotton-oil industry now gives employment to a large number of business men, mechanics, and labourers, most of the latter being negroes.—*Board of Trade Journal.*

#### THE MEXICAN LEATHER INDUSTRY.

A report on the subject of the leather trade and industry of Mexico has recently been prepared by M. André Beaufils, who is attached to the French Legation in that country.

The raw hides of Mexico are of average quality, and might successfully compete on the European markets with the best South American raw hides, by reason of their solidity, although they are very thin.

An estimate of the cost price of 1,000 Mexican hides delivered at a European port gives approximately the following results:—Cost price in Mexico of 1,000 raw hides at 1.20 *dols.* each, 1,200 *dols.*; carriage to the coast, 50 *dols.*; sea freight, 614 *dols.*; 10 per cent. for other

expenses (loading, unloading, &c.), 25 *dols.*; total, 1,889 *dols.*, which is equivalent to about 20*l.* per 1,000 hides. As the South American hides cost from 20*l.* to 22*l.* there would appear to be great advantage in using the Mexican hides, especially when their superiority to the products of Brazil and La Plata is taken into account. It may be observed that the United States already purchases from Mexico the greater portion of its raw hides.

Speaking of the tanning industry in Mexico, it is stated that it is still in its infancy, both as regards the small number of tanneries met with and the simple processes employed. The largest Mexican tanneries are to be found chiefly in the district of Mexico and in a few of the northern and eastern States.

The tanning of the raw hides may be said to consist of four distinct operations: 1. The washing of the raw hides; 2. Liming; 3. Placing in the bran pit; 4. Tanning properly so-called.

The raw hide arriving from the interior of the country, whether it is salted or dried, is first washed in fresh water, in the opposite direction to the fall of the hair, where it is left for a few days in order to soften and to reject all the substances preserving it from putrefaction. This operation is called the washing.

The washing being completed, the hide is placed in a pit filled with lime-water, water known in Mexico as *pelambre*. It remains in these pits from a week to a fortnight, during which period the hides are stirred several times a day. This operation is for the purpose of removing the hair from the hide. When the hair commences to fall off, the hides are taken out and are scraped on the upper side with a square-bladed knife, so that no hair remains attached to the hide.

The scraped hides are then placed in other pits containing bran-water and called by the Mexicans *salvaderas*. The hide is withdrawn at the end of a few days and is fleshed (*décharné*), that is to say, all waste or fatty matter is removed from the surface of the hide.

All these preparatory operations being finished, the tanning properly so-called commences. The tanning material in Mexico is not represented by tan resulting from oak-bark. It comes from *dividivi*, known by the Mexicans as *cascalote*, a very common tree on the borders of the Gulf of Mexico, belonging to the *cuscalpinia coriaria*, and the pods of which contain an astringent and bitter matter. It is of these pods reduced to powder that the raw material for tanning consists.

The hide thoroughly cleaned and all foreign matter being removed is placed in layers in pits, the hides being separated from each other by a light layer of *dividivi* powder. The pit being piled up, it is filled with water and then closed. Twenty days after, the hide is taken out, cut in two, and placed in other pits, richer in tanning materials. This operation is repeated as many times as may be deemed necessary, the quantity of *dividivi* being increased each time. The raw hide has then become leather, suitable for domestic and industrial uses. Owing to the warmth and the special climatic conditions of Mexico, the process of tanning only lasts two months. Unfortunately, as is always the case with leathers tanned in such a short time, Mexican leathers are of inferior quality; they are spongy, wanting in suppleness and in resisting power. Their use is therefore a very limited one, and is confined to the manufacture of common shoes and cheap saddles.

For the reasons given above, Mexican leather tanned in the country is not of sufficiently good quality to be used for shoes of a superior kind, and leather imported from Europe is obliged to be used. The leathers mostly consumed in native manufacture are calf, kid, and *gamuza*, a kind of badly prepared morocco.

Foreign leathers used chiefly for the uppers of shoes represent about 15 per cent. of the general consumption. The superior qualities come principally from France (about 85 per cent. of the total import), and from England and the United States (about 10 per cent.) for good medium leathers. Germany sends leathers of a very inferior quality, but they meet with little approval on the Mexican

markets. The competition of Germany with France is therefore very insignificant.—*Ibid.*

#### GERMAN MINING INDUSTRY.

Sir C. Oppenheimer, Her Majesty's Consul-General at Frankfurt-on-the-Main, in his general commercial report for 1893, writes that according to official statements the results of the mining industry in Germany for the year 1893 are as follows:—

The total amount of coal produced in 1893 was 73,908,999 tons (1 ton equal 1,000 kilos.), 3.6 per cent. more than in the year 1892, in which 71,372,193 tons were obtained. Notwithstanding this increase in the output, the total value of the coal produced in 1892, viz., nearly 527,000,000 marks, decreased in the year 1893 to 498.5 million marks, as on the average in 1893 only 6.74 marks per ton was received, as against 7.38 marks in 1892. In the coal output of 1893 the share of the district of Dortmund amounted to 52.2 per cent., that of Breslau to 28 per cent., and Bonn to 16.6 per cent., and that of the kingdom of Saxony to 5.9 per cent. The brown coal output was in 1893, 21,567,218 tons, against 21,171,857 tons in 1892; as the total value of this production for 1893 was estimated at 55,000,000 marks, and that for 1892 at 58.5 million marks, an average value of 2.55 per ton in 1893, and of 2.76 marks in the year 1892 was obtained. About three-fourths of the brown coal fell to the district of Halle. Also the production of rock-salt (669,942 tons), kainite (664,986 tons), and other potassic salts (861,162 tons) was greater in 1893 than in 1892, which was 662,577 tons, 548,445 tons, and 842,630 tons respectively; the average price of these products was in the year under report a little higher than in 1892.

The production of common salt has also risen (505,023 tons, against 504,687 tons in 1892), as also of chlorate of potash (137,216 tons, against 123,962 tons in 1892), and for the former product somewhat higher prices, whereas for the latter rather lower prices were obtained in 1893 than in the previous year. As regards the products of the foundries, the production of raw iron (4,986,003 tons, against 4,937,461 tons in 1892) and of zinc (142,956 tons, against 139,938 tons) has increased in comparison with the preceding year; on the other hand, that of lead (94,639 tons, against 97,742 tons in 1892), of copper (24,011 tons, against 24,781 tons), of silver (449 tons, against 489 tons), and of gold (3 tons, against 3 tons 8 cwt.), has not come up to the figures of 1892. The prices of all metals have decreased, in some cases even very considerably.—(No. 340, *Foreign Office Miscellaneous Series.*)

#### THE PRESERVED FRUIT INDUSTRY IN PORTUGAL.

Mr. Honorius Grant, Acting British Consul at Oporto, in a report to the Foreign Office, dated the 17th September, encloses a memorandum on the subject of the fruit-preserving industry in Portugal, in which he says:—

"A certain trade is done from Portugal in the exportation to England of dried preserved fruit, an article the manufacture of which was a kind of speciality of the nuns, but since the gradual extinction of the convents the trade has passed into other hands. The fruit exported consists of plums, peaches, figs, and apricots, and is packed up in round boxes of various sizes and decked with paper lace, silver wire, tinsel, and the like, making up a somewhat showy *tout ensemble* and not unsuitable for a dinner-table ornament.

"Now, considering that better fruit and cheaper sugar are procurable at home, there would seem to be no reason why this article should go from here to England instead of being made by the growers at home.

"The process used here is, I understand, simple in the extreme, and consists, roughly speaking, in boiling the fruit (before complete maturity) with sugar six times in the same water, after which the sugar is allowed to run off, and the fruit is put out on trays to dry in the open air."—*Board of Trade Journal.*

#### PRIZES FOR CHEMICAL RESEARCH.

In addition to the prizes to be offered next year by the Society for the Encouragement of National Industry, an

award of 2,000 fr. is to be given in 1896 for the best paper on the discovery of processes for the synthetic manufacture of useful organic bodies, such as quinine, cane sugar, &c. The prizes will only be awarded in the event of the papers proving of sufficient interest.—*Chemist and Druggist.*

#### GENERAL TRADE NOTES.

##### SOUTH AFRICAN NITRATE FIELDS.

Dalziel's correspondent at Cape Town sends the following account, given by a correspondent of the *Pretoria* (Cape Colony) nitrate fields. The nitrate beds at Prieska present the most valuable and richest deposit of nitrate of potash ever found. It is a most valuable substance, not to be confounded with sodium nitrate or Chilean saltpetre, so largely exported from South America, and sold on the London market at 19s. per ton, whereas the average price of unrefined potassium nitrate is 10s. 10d. per ton. The yield of the farms prospected is virtually unlimited, and, while in many of the large kloofs enormous masses of practically pure salt are found, the average in the soil for the whole area may be calculated at 25 per cent. Attention need only be drawn to the working of nitrates in other countries to prove the richness of the most recent South African discovery. Soils containing nitrate of potash are worked in India and Ceylon when containing only 2½ to 5 per cent., and the richest deposits in those countries rarely contain more than 8 per cent., while in Hungary nitroferous soils containing ½ to 2½ per cent. are worked at a profit. The deposit here is most easily extracted, merely by lixiviation, or hot or cold water decantation of the clear liquor from the soil into shallow tanks, and evaporation by the rays of the sun, when practically pure nitrate crystallises out. By this process, nitrates on a large scale could be produced at less than 2s. per ton, and the cost of transport over 140 miles of country to De Aar, and thence by rail and sea to Europe, is 5s. per ton.—*Engineering and Mining Journal.*

##### THE PRESENT STATUS OF THE CANADIAN ASBESTOS INDUSTRY.

The following particulars of the present condition of the Canadian asbestos industry have been obtained by the writer in a recent visit to the mining district. There is at present no prospecting for asbestos, and with one exception, to be noted hereafter, no business in buying and selling mining properties. After a somewhat prolonged period of dullness in the crude asbestos market, there are now decided indications of a legitimate improvement. Evidently stocks in the hands of manufacturers are running very low, and there is a moderate demand from all parts of the world.

With the asbestos miner it is not now so much a question of finding buyers as of finding a margin on the prices offered. Sales have recently been made at the following prices f. o. b. at the mines: No. 3, a round lot at 40 dols. per ton; No. 2, 65 dols. to 67.50 dols. per ton; No. 1, 115 dols. to 140 dols. per ton.

Nos. 2 and 3 are the grades mostly called for, and these are more largely produced by the mines. No. 1 quality is apparently not as much sought after as in the past, and as a consequence there is a notable stock of this grade in producers' hands. This is doubtless due to the very high price asked for the first quality during the speculative period of three years ago, which caused manufacturers to devise improvements in plant, and methods which enabled them to replace the costly fibre by lower grades. At the same time there is a limited demand for fibre of the very finest quality, as to length and colour, and freedom from impurities; for example, a leading dealer recently was offering 150 dols. per ton for 30 tons that would answer to certain specifications by no means beyond the choice product of our mines.

As a consequence of the improved inquiry for crude asbestos, the large and well-equipped mines, with two or three exceptions, are being worked, not to their full

capacity, but yet to an extent that will make the output of 1894 a decided advance on that of 1893. Fully 3,300 tons have been exported during the first six months of this year.

The old Jeffrey mine near Danville, on the Quebec branch of the Grand Trunk Railway, has long been of geological interest, from the fact that it occupies a knoll of asbestos-bearing serpentine, distant from 40 to 50 miles from any other known deposit of asbestos. This mine, which has also possessed a local interest, inasmuch as it has been owned and actively operated by the oldest miner in the province, a veteran of some four score and seven years, has recently changed hands. It is now being vigorously worked by two of our most enterprising Canadians, who are at present employing 150 hands. These new owners have the great advantage of being in close touch with manufacturers of asbestos goods, and it is probable that this well known mine will become even more prominent than it has been in the past.

The old problem of separating the lower grades of fibre from the enclosing rock is still to the fore. A special feature in this connection has been the installation by Messrs. W. G. Costigan and Co., of Montreal, of their cyclone fiberising machinery in three of the most important mines, viz., the Jeffrey mine, mentioned above, the Anglo-Canadian mine at Black Lake, and the Bell mine at Thetford. In this cyclone machinery the low grade asbestos, which is rock and fibre in intimate association, is introduced in small lumps, which meeting too rapidly revolving discs running in opposite directions have their non-fibrous particles reduced to powder by attrition, while the fibre, by reason of its nature, escapes injury, which is by no means the case when rolls or crushers are used. After leaving the cyclone the grit or gravel is separated by means of a series of screens, which, to a certain extent, also separates the fibre of different lengths.—*Engineering and Mining Journal*.

#### THE MINERAL PRODUCTION OF AUSTRIA.

The "Oesterreichische Zeitschrift fuer Berg- und Huettenwesen" of recent date gives the following statement of the mineral output of Austria for the year 1893, the figures being taken from the returns made to the Ministry of the Interior. The amounts given are in metric tons:—

	Tons.	Percentage of Change.
Gold ore.....	477	I. 190.7
Silver ore.....	18,918	I. 27.2
Quicksilver ore.....	76,215	D. 4.1
Copper ore.....	81,736	D. 0.7
Iron ore.....	1,162,112	I. 11.7
Lead ore.....	101,96	D. 19.4
Zinc ore.....	10,531	D. 10.1
Tin ore.....	26	D. 22.1
Bismuth ore.....	797	D. 6.8
Antimony ore.....	411	I. 353.6
Uranium ore.....	21	I. 19.1
Wolfram ore.....	43	I. 40.6
Chrome ore.....	"	"
Iron pyrites.....	1,959	I. 8.1
Alum and alum slate.....	13,570	D. 31.7
Manganese ore.....	5,111	D. 18.7
Graphite.....	23,807	D. 13.5
Asphalt.....	88	I. 12.7
Lignite (brown coal).....	16,815,955	I. 3.9
Coal.....	9,732,051	I. 5.3

Most of the changes, it will be seen, are not of great importance. The most notable are in zinc ore, and the gain in coal and lignite production.

The metallic output is given as follows, gold and silver in kilogrammes and the other metals and metallic products in metric tons:—

	Tons.	Percentage of Change.
Gold.....(kilos.)	35	I. 171.4
Silver.....(kilos.)	37,311	I. 1.9
Quicksilver.....	512	D. 5.6
Copper.....	944	I. 12.8
Sulphate of copper.....	177	I. 33.1
Pig iron, foundry.....	555,002	I. 4.7
Pig iron, forge.....	108,283	I. 7.5
Lead.....	7,212	D. 0.6
Litharge.....	2,011	D. 4.3
Nickel and cobalt.....(kilos.)	129	D. 21.1
Nickel sulphate.....	8	I. 79.1
Zinc.....	5,879	I. 12.1
Tin.....	66	I. 9.3
Bismuth.....(kilos.)	584	I. 6.6
Antimony.....	175	I. 53.4
Uranium salts.....	5	I. 113.7
Sulphur.....	9	D. 83.3
Sulphur ore.....	1,221	I. 0.9
Sulphuric acid.....	10,248	D. 7.2
Alum.....	837	D. 23.6
Copperas.....	1,129	I. 4.1
Mineral paint.....	3,020	I. 49.3

Most of these show no changes of importance. The increase in iron production is, however, considerable, and indicates a steady growth. The gold and silver output was almost entirely from Bohemia, as in former years. The quicksilver was chiefly from the famous mines of Idria, which supplied 87 per cent. of the total, the rest coming from St. Anna and Littai. The copper was from Bohemia, Salzburg, and the Tyrol.

Nickel, cobalt, arsenic, and chrome ore have disappeared from the list of the products reported in former years, none of these minerals having been produced last year.

#### THE INDIGO INDUSTRY IN BRITISH INDIA.

According to Dr. George Watt's recent report to the Indian Government there are in India 2,762 indigo factories and 6,032 vats, giving employment to 356,675 persons during the working season—a calculation that does not include the agricultural labour necessary to produce the plant. These are instructive figures, but not more so than those which may be shown regarding the history and growth of the industry. In 1782 the total imports of indigo into Great Britain were 495,100 lb., of which South America furnished 161,216 lb., the West Indies 64,309 lb., British Colonies 128,640 lb., and Asia (including India) 23,535 lb. Since then the Asian imports rose rapidly. In a dispatch, dated 1792, the Board of Directors congratulate the Indian Government that, as the British imports of Bengal indigo increased, those from the Spanish and French colonies declined, while at the same time a large export trade from Great Britain to the Continent had been established. It may thus be said that in less than 20 years the East India

Company had restored to India its lost industry. The average for the past ten years was fully 15,000,000 lb. Last year's exports were 126,706 cwt., valued at Rs. 41,412,000.—*Chemist and Druggist*.

#### CINCHONA CULTIVATION IN BRITISH INDIA.

In a recent report of the Indian Government, it is stated that on the Government plantation of the Darjeeling district in Bengal there were, at the end of the year 1892-93, 4,331,000 cinchona trees, or 100,000 less than in the preceding year. Of the existing trees, nearly four-fifths are of the pure quinine-yielding variety, and less than one-quarter belonging to the cinchonine-yielding variety. During the year 466,000 trees were uprooted for their bark, or died, while 181,000 were planted out; all the new plantations have for some years past consisted of quinine-yielding varieties. The harvest of dry bark was 304,000 lbs. The factory produced during the year 3,481 lbs. of cinchona febrifuge, and 5,242 lbs. of sulphate of quinine. Quinine is now sold to public institutions and Government offices at one rupee per ounce, while the febrifuge is sold at ten annas per ounce. The issues from the factory were 3,647 lbs. of febrifuge and 5,518 lbs. of quinine. The sales and issues of medicine during the year yielded a profit, after meeting all charges for maintaining the plantations, renewing plant, and working the factory. As soon as the remaining million of red bark trees are used up, it is intended to manufacture only quinine, and to cease making cinchona febrifuge at the Darjeeling factory. The capital cost of the plantation had been repaid by the sale of cinchona drugs in previous years. During the year 1892, 475 lbs. of quinine were put up in 5-grain doses; each dose is carefully packed, and bears simple instructions in one of the vernacular languages of Bengal. The packets are made up in the jail department, and they are issued to local postmasters, who, for a small commission, retail them to the public at one pie (one farthing) per packet, or dose. As yet, the arrangement has been made for Bengal only; when it is extended to the provinces, the demand for quinine thus brought in convenient shape, and in an absolutely pure state, to the doors of the people, is expected to increase indefinitely. Ample ground has been reserved for extending the Darjeeling plantations. In the Government plantations on the Nilgiri hills, in Madras, the cinchona trees are almost all of the quinine-yielding varieties. Drought, and the absence of sunshine, made the year 1893 unfavourable for cinchona cultivation on the Nilgiri hills. The stock of dry bark in hand at the beginning of the year was 416,000 lbs.; the harvest of the year was 119,000 lbs.; the consumption of the factory was 172,000 lbs.; and 2,000 lbs. were issued in the shape of bark. The produce of the factory during the year was 4,933 lbs. of quinine and 3,139 lbs. of cinchona febrifuge; 3,204 lbs. of the former and 2,600 lbs. of the latter were sold or issued to Government and municipal or local departments. The number of quinine powders issued to the public by collectors of districts rose to 170,000, from 37,000 in the previous year. The area under cinchona on private plantations outside Bengal is returned at 10,862 acres, nearly all of which are in Madras. The exportation of cinchona bark from India by sea, which was 3,074,000 lbs. in 1888-89, and 2,693,000 lbs. in 1891-92, rose to 2,814,000 lbs. in 1892-93.—*Journal of Society of Arts*.

#### CANADA AND THE AMERICAN DRUG TRADE.

Whereas not many years ago Canada imported the largest proportion of its drugs from Great Britain, its imports from the United States have of late increased rapidly, and are now larger than those from England. The total drug imports into Canada from all foreign countries in 1892-93 amounted to 3,408,166 dols. From the subjoined table it will be seen that nearly 50 per cent. was imported from the United States. The Canadian returns of imports and exports for the year ending June 30th, 1894, have not yet been published, but the following is the value of the drugs, dyes, chemicals, and medicines imported into the Dominion

of Canada from the United States during the undetermined years ending June 30th:—

	1890.	1891.	1892.	1893.
Free of duty .....	Dols. 662,504	Dols. 731,332	Dols. 741,267	Dols. 808,165
Dutiable .....	618,963	598,100	626,916	641,437
Totals .....	1,281,467	1,329,432	1,368,183	1,449,602

The value of the total exports of the above descriptions of merchandise from the United States during the year ending June 30th, 1893, was:—

	Dols.
Domestic .....	6,751,068
Foreign .....	628,122
Totals .....	7,379,190

In 1892-93 Canada purchased from the United States a little more than 20 per cent. of all its exports of these articles.—*Chemist and Druggist*.

#### PLATINUM.

The Ural platinum deposits in Russia are the only ones in the world, as this metal is worked nowhere else, and is known simply as a mineral finely disseminated in certain rocks. Platinum occurs in the Ural Government of Perm, where it is found on various private properties and State lands. In the district of Goroblagodat there are 70 allotments for the exploitation of platinum under different private individuals. The metal is found in the form of alluvial deposits or platinum-bearing sands, which frequently also contain gold. These deposits vary in thickness; they are rarely less than three, and often reach seven feet; the grains are usually small in size, but occasionally small nuggets are found weighing one or more kilogrammes. The platinum is frequently accompanied by other rare metals, such as iridium and osmium. At present all the platinum extracted in the Urals is forwarded in the crude state to St. Petersburg, whence it is sent abroad. Although there are two laboratories in the Russian capital for refining platinum ore, the greater quantity is sent abroad in the crude state. The production is subject to a tax of 3 per cent. for leasehold, and 4 per cent. for freehold works. The rapid and variable fluctuations in the price of a product having no definitely fixed exchange value, but indispensable to the arts, reflect upon the production of platinum in Russia. Thus, when the price of the metal is high, it becomes profitable to work the poorer deposits, while it is only possible to work the very richest when the price is low. Although the first platinum deposits in Russia were discovered so far back as 1819, the actual exploitation of this metal began only in 1824, when rich veins were discovered in the Nizhni-Tagilsk district. From 1828 to 1845 platinum money was coined in Russia. The denomination of these coins was three, six, and twelve roubles; the total value of platinum money put into circulation was 4,250,000 roubles. During this period the production of platinum increased considerably, but when platinum coinage ceased the exploitation of the metal was almost entirely stopped, and only revived in 1859. In 1887 the production of pure platinum was 269 poods 4 pounds, in 1890 it was 173 poods 263 pounds. The value of the yearly export of platinum, which goes chiefly to England, is about 1,560,000 roubles. The largest quantity of platinum is now extracted at the deposits of Nizhni-Tagilsk, belonging to Prince Demidoff San Donato, and at the Krestovosvzvensk deposits of Count Schouvaloff. In 1890 there were 6,000 workmen employed in the exploitation of platinum.—*Petersen's Trade Review*.

#### SULPHUR MINING IN LOUISIANA.

A New York syndicate are reported to have purchased the sulphur mines near Sulphur City, Louisiana, for 175,000 dols., and preparations are being made for their development. The sulphur is found at a depth of 180 feet,

and it is estimated that the mines contain a thickness of 100 feet solid sulphur of 98 per cent. purity.—*Chemist and Druggist*.

#### ALLEGED NITRATE DISCOVERIES IN COLOMBIA.

The *Panama Star* reports the discovery, at Magdalena, Colombia, S. America, of deposits estimated to amount to over 7,000,000 tons of nitrates, associated with iron pyrites. The quality of the deposit and its situation are said to be adapted for working in competition with the Chilean trade.

#### THE GERMAN CHEMICAL INDUSTRY SINCE 1885.

*M. Diezmann. Chem. Ind. 1894, 355.*

The progress made by German chemical industry in recent years becomes apparent from the following tabulated statement:—

	1885.	1886.	1887.	1888.	1889.
Imports—Million Marks	220'99	205'23	219'54	242'85	267'45
Exports ..	220'21	214'38	226'35	236'14	259'20

	1890.	1891.	1892.	1893.
Imports—Million Marks	261'87	261'67	266'37	273'1
Exports ..	274'69	277'97	286'04	299'1

Compared with the total trade of the German Empire, the respective percentage values amount to:—

	1885.	1886.	1887.	1888.	1889.
Imports—Million Marks	7'50	7'45	7'06	7'12	6'70
Exports ..	7'73	6'88	7'22	7'37	8'24

	1890.	1891.	1892.	1893.
Imports—Million Marks	6'32	6'38	6'63	6'89
Exports ..	8'26	8'75	9'08	9'67

Some of these changes are, no doubt, due to the incorporation, in the year 1888, of Hamburg and Bremen into the German Customs Union.

In regard to the chemical trade, the value of the raw materials imported amounts to about 55·8—62·4 per cent. of the value of the imports, this considerable variation being chiefly due to *nitrate of soda*, which occupies the foremost and most important position among the imports. Deducting the value of this commodity, there remains an almost constant figure of 92·7 million marks. Raw materials do not figure conspicuously among exports, amounting only to about 12 per cent. The *total trade in the chemical industry* and pharmacy is classified in the following way:—

	Imports.		Exports.	
	Thousand Tons.	Million Marks.	Thousand Tons.	Million Marks.
Raw materials of the chemical industry ..	324'31 up to 683'7	42'15 up to 79'18	148'59 up to 208'43	5'78 up to 8'99
Drugs for medical use .....	9'05 .. 12'68	15'59 .. 23'41	2'55 .. 3'96	5'55 .. 7'89
Resins .....	71'12 .. 110'63	19'36 .. 28'05	20'23 .. 34'84	3'83 .. 7'88
Fermenting and clarifying agents .....	13'99 .. 23'60	3'21 .. 6'12	3'71 .. 5'71	3'16 .. 1'86
Crude dyes .....	20'37 .. 108'44	10'83 .. 13'93	45'42 .. 59'33	4'83 .. 5'65
Tanning materials .....	91'39 .. 177'13	18'00 .. 25'71	1'58 .. 6'73	0'77 .. 1'70
Total .....	622'06 .. 1,000'37	118'07 .. 165'05	249'67 .. 304'24	23'92 .. 32'58

Among the raw materials of the chemical industry, the following articles may be enumerated, the individual values

exceeding 1,000,000 marks. The data refer to the period 1885—1892.

#### I. A. RAW MATERIALS OF THE CHEMICAL INDUSTRY.

	Imports.		Exports.	
	Thousand Tons.	Thousand Marks.	Thousand Tons.	Thousand Marks.
Nitre .....	153,738 up to 395,653	31,248 up to 69,239	2,430 up to 13,789	523 up to 2,387
Pyrites .....	130,502 .. 238,644	3,092 .. 4,773	8,285 .. 27,940	124 .. 447
Cream of tartar .....	1,331 .. 2,444	1,394 .. 4,346	216 .. 798	471 .. 896
Sulphur .....	11,664 .. 19,518	1,100 .. 2,245	559 .. 1,037	72 .. 150
Kryohite and bauxite .....	3,100 .. 11,782	349 .. 1,922	122 .. 424	45 .. 145
Stassfurt salts .....	125 .. 2,611	.. ..	112,123 .. 175,662	2,803 .. 4,161
Manganese ores .....	6,156 .. 9,662	516 up to 796	3,405 .. 10,620	511 .. 1,538
Not specified .....	11,376 .. 18,332	536 .. 1,411	3,694 .. 8,261	184 .. 445
Total .....	324,314 .. 683,700	42,118 .. 79,977	148,598 .. 208,439	5,784 .. 8,994

Deducting exports from imports the home consumption of *nitre* rose from 154,300 tons in 1885 to 385,000 tons in 1891, and in this respect Germany heads all other countries. The respective figures are:—

	1890.	1891.	1892.
In Germany .....	331,800	385,000	370,000
In France .....	210,500	173,800	215,000
In England.....	100,000	121,000	125,000
In United States.....	32,400	102,000	110,000

The value of nitre varied between *8l.*—*10l.* per ton.

In *pyrites* (including native alum) the chief importing centres were Spain and Portugal; the quantities improved from 123,000 tons in 1885 to 274,000 tons in 1893. The exports of sulphur are decreased from 28,000 tons in 1885 to 8—10,000 tons for the last years, Austria and Switzerland being the chief consumers. In 1893 the exports suddenly rose to 16,000 tons. The estimated average value decreased for imports from 2·3 marks for 100 kilos, in 1885 to 1·8 marks (1893, 1·9 marks), and for exports from 1·6 to 1·3 marks.

The consumption of *cream of tartar* is decidedly on the decrease. The exports consist chiefly of the refined article, hence they are given at an enhanced value (an additional 30—60 marks per 100 kilos.). The trade shows the following figures:—

	1885.	1887.	1888.	1890.	1892.	1893.
Imports ..Tons	2,173	1,646	2,452	2,005	1,391	1,552
Exports... "	216	235	265	498	798	614
Transit ... "	1,220	1,189	467	226	78	..

The importing countries are Italy, France, and Austria. The exports are taken up by England and lately by Australia. The average value decreased for imports from 200 marks per 100 kilos, in 1885 to 99 marks in 1893, and for exports from 230 marks to 145 marks.

The quantity of *Sulphur* imported increased twofold (235,000 tons in 1893), notwithstanding the increased imports of *pyrites*. The values kept fairly constant at 10—12·5 marks per 100 kilos.

The home consumption of *Cryolite* and *Bauxite* amounts for most of the years up to 1891 to 5,000 tons, increasing in 1892 to 14,800 tons, and in 1893 to 9,700 tons. The values average as follows:—

	1885.	1886.	1890.	1891.	1892 and 1893.
Imports .....	M. 9	M. 10	M. 10	M. 30·32	M. 14
Exports.....	80	50	10	42	70

The exports of *Stassfurt Salts* are subject to considerable variations. The chief consumers are:—

	1889.	1890.	1891.	1892.	1893.
United States.....Tons	72,196	119,902	77,751	130,312	162,206
England.....	14,102	38,711	37,179	27,818	58,851
Sweden.....	10,850	12,570	18,744	16,561	50,915
Other countries.....	15,481	10,070	20,526	30,470	66,187
Total.....	112,829	112,123	156,900	115,064	212,259

Since 1885 the values were taken at 2½ marks per 100 kilos.

As regards *Manganese Ores*, the export values show an apparent excess over the import values, the former averaging 7·5—8·5 marks per 100 kilos, for imports, the latter 20 marks in 1885, and since then 15—16 marks. As regards quantities the proportion is reversed. The chief importer is the Caucasus, and in the second line Austria and Belgium. The maximum of the imports was reached in 1893 with 12,000 tons, and the exports fell in the same year to their lowest level (2,700 tons).

#### I. B.—CRUDE DRUGS.

	Imports.		Exports.	
	Tons.	Thousand Marks.	Tons.	Thousand Marks.
China bark .....	4213 up to 6,065	4,122 up to 11,494	22 up to 98	55 up to 226
Sponges .....	260 " 326	3,938 " 8,108	36 " 56	615 " 1,574
Camphor .....	399 " 1,914	119 " 3,133	217 " 581	326 " 2,633
Crude manufactured products not specified.	3,090 " 6,138	2,787 " 5,218	2,025 " 2,845	5,038 " 4,268
Others.....	985 " 1,731	1,892 " 3,265	131 " 411	492 " 1,000
Total.....	9,065 " 14,765	15,594 " 23,414	2,550 " 3,955	7,702 " 7,885

Stocks of *China Bark* accumulated from 1,500 tons in 1880 to 6,000 tons in 1891 and 1892, but the available quantities decreased to 4,230 tons in 1893. The tremendous fall in its value is shown by the following table, in marks per 100 kilos.:—

	1885.	1886.	1890.	1891.	1891 and 1893.
Imports .....	30	10	10	9·32	13
Exports.....	50	50	20	42	70

The use of *Camphor* for the manufacture of smokeless powder was accompanied by a corresponding rise from 110 marks per 100 kilos, to 280 marks in 1890, and since then to 290—300 marks, for imports of the crude and refined stuff. At the same time the exports, consisting chiefly of the refined article, were valued at an increase of 40 marks in 1885, 50 marks in 1886, 55 marks in 1887, 80 marks in 1893 for 100 kilos. The chief producers are China and Japan, and the consumers France, Russia, and Austria.

The imports of *crude products not specified* rose from 3,100 tons in 1885 to 5,000 tons in 1889. They are of a manifold nature, and include laurel leaves from Austria, senna leaves, quassia wood, ipëcacuanha root from England,



vamilla and baldrian root from Belgium, &c. The exports varied between 2,000—2,800 tons, averaging 150 marks for

100 kilos. The principal customers are the United States, Austria, Russia, England, France, and Holland.

### I. C. RESINS, &c.

	Imports.		Exports.	
	Tons.	Thousand Marks.	Tons.	Thousand Marks.
Turpentine resin.....	15,990 up to 76,753	4,984 up to 7,933	4,793 up to 11,371	633 up to 1,285
Gum arabic, &c. ....	1,302 " 2,502	5,674 " 7,790	205 " 627	1,027 " 2,383
Gum lac, &c. ....	1,137 " 1,810	1,705 " 3,205	64 " 225	113 " 430
Other resins .....	2,143 " 3,514	3,840 " 7,029	236 " 1,088	328 " 2,611
Asphalt .....	15,708 " 27,059	938 " 1,488	11,948 " 17,999	584 " 1,009
Refuse for the manufacture of glue.....	4,972 " 6,393	955 " 1,391	2,256 " 5,496	519 " 1,250
Total .....	74,416 " 114,210	19,359 " 28,047	20,234 " 34,839	3,828 " 7,875

Since 1886 there is an increasing tendency in almost all these goods. As regards *turpentine resin* the chief importers are the United States. The exports go to Austria, Russia, Switzerland, and Sweden. Its value for imports is taken at 14 marks in 1880, 11·5 marks in 1885, and 9—10·4 marks since then.

The imports of *gum arabic*, Senegal and tragacanth decreased from 1,746 tons in 1885 to 1,302 tons in 1887, and rose since to 2,502 tons in 1892 and 2,392 tons in 1893. These variations are chiefly due to French imports (Senegal gum), thus : 508 tons in 1885, 98 tons in 1889, 528 tons in 1891, and about 400 tons in 1892 and 1893. The fluctuations are also due to the variations in the value of gum arabic, as evinced by English statistics, according to which 100 kilos, gum arabic are valued for imports :—

—	1885.	1886.	1887.	1888.	1889.	1890.	1891.	1892.
Marks	138	157	185	162	150	122	102	112

and according to Hamburg statistics :—

Marks	202	256	271	226	209	212	226	172
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Senegal gum :—

Marks	188	261	263	315	210	..	173	170
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and tragacanth gum :—

Marks	264	273	294	270	401	..	292	255
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According to the imperial statistics the three varieties are valued for imports at the following high rates :—

Marks	325	450	450	480	450	350	320	300
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which amounts are increased for exports, by 55 marks for 1885, 90 marks for 1886—1889, 50 marks for 1890, and since then by 80 marks per 100 kilos.

One half of the total imports originate from the British East Indies. The difference between imports and exports, which fell between the years 1885—1887 from 1,476 tons to 1,080 tons, has since then increased to 1,875 tons in 1892, but receded to 1,592 tons in 1893. The exports have considerably increased on French and Austrian account.

The home consumption of *gum lac and shellac* remains fairly constant at 1,300—1,600 tons per year, with the exception of 950 tons for 1889 and 1,840 tons for 1893; the prices varied between 120—200 marks per 100 kilos. In *other resins* the consumption varied between 1,820—2,115 tons in 1885—1890, with a minimum of 1,463 tons in 1885. Since then a marked increase of imports over exports has taken place, to 1,933 tons in 1890, 2,371 tons in 1891, 3,025 tons in 1892, and 3,787 tons in 1893, chiefly owing to supplies from England and from the States. No explanation is to be found for this increase, except it be due to incorrect declaration of, say, turpentine resin.

The imports of *asphalt*, including resin and wood-cement, rose from 15,708 tons in 1885 to 29,615 tons in 1893. One half of this quantity comes either direct or via England from Trinidad, and the remainder from Switzerland. The exports amounted to 12,615 tons in 1885, 17,999 tons in 1891, and 12,622 tons in 1893. The chief consumers were, for a long time, the States, but the quantities consumed there decreased in 1893 from 7,350 tons to 2,590 tons. Other consumers are England, Russia, Sweden, &c. The average price was taken in 1885 at 8 marks, and since then between 4·5 and 5·5 marks for both imports and exports.

As regards the *residues for the production of glue* the chief consumer is Austria. The average price for imports was taken at 20 marks for 100 kilos. in 1885, and since then 19 marks; for exports it is taken at 23 marks.

The considerable decrease in the imports of chiefly Austrian and Russian *bone charcoal* from 19,000 tons in 1885 to 10,000 tons in 1892, is no doubt due to the diminished demand for this article in sugar refineries. There is also a corresponding decrease in the exports. The average value of the imports is taken at 20 marks per 100 kilos. in 1885, and at 12 marks in 1893, of the exports at 18—22 marks.

*Yeast*, other than wine yeast, shows a similar decrease in both imports and exports. The imports of *wine yeast*, on the other hand, especially of Italian origin, increased considerably from 710 tons in 1880, to 5,453 tons in 1889; since then it amounted to :—

—	1890.	1891.	1892.	1893.
Tons	3,832	3,812	4,703	5,537

In the same time, the values increased temporarily, from 40 marks per 100 kilos. in 1885 to 50 marks in 1887, and then receded gradually to 25 marks in 1893. The supplies are derived from Italy, France, and Spain.

## I. D. FERMENTING AND CLARIFYING AGENTS.

	Imports.		Exports.	
	Tons.	Thousand Marks.	Tons.	Thousand Marks.
Bone charcoal .....	9,992 up to 19,967	1,204 up to 3,833	2,018 up to 3,538	419 up to 778
Wine yeast .....	2,843 " 5,455	1,137 " 2,245	2 " 33	0 " 13
Other yeast .....	42 " 694	97 " 694	1,327 " 2,128	1,394 " 2,128
Other materials .....	20 " 36	895 " 643	9 " 18	189 " 376
Total .....	13,898 " 23,597	3,207 " 6,120	3,704 " 5,710	1,800 " 3,161

## I. E. CRUDE DYE-STUFFS.

	Imports.		Exports.	
	Tons.	Thousand Marks.	Tons.	Thousand Marks.
Dye-woods .....	48,650 up to 63,326	7,271 up to 10,656	39,960 up to 14,154	1,473 up to 2,281
Graphite .....	7,681 " 13,967	922 " 1,875	1,287 " 3,178	131 " 579
Barytes .....	3,193 " 5,661	369 " 453	21,917 " 31,226	1,563 " 1,561
Dye-earths .....	4,129 " 7,507	495 " 731	8,316 " 10,997	1,175 " 1,510
Others .....	12,314 " 21,937	711 " 2,260	2,382 " 5,622	120 " 669

The German consumption of *dye-woods*, as arrived at by deducting exports from imports, has been decreasing since 1885, with the exception of the year 1893, thus:—

	1887.	1888.	1889.	1890.	1891.	1892.	1893.
Logwood..Tons	38,847	43,085	41,314	42,180	37,446	31,458	35,371
Fustic.... "	6,876	6,129	5,033	4,157	2,836	1,402	4,545
Brazil wood "	4,673	5,518	6,827	1,535	1,826	2,676	1,756

Logwood is imported chiefly from Mexico, Jamaica, and Hayti. Its average value per 100 kilos. amounted to 14 marks in 1885, 17 marks in 1889, then 15 marks, and finally 17 marks in 1893. The English quotations vary between 10·5 and 14 marks. Fustic is valued at between 9 and 12 marks, Brazil wood at 16—18 marks. The trade in dye-woods amounted to:—

	Imports.	Exports.
Logwood .... 1,000 Marks	6,347 up to 8,638	1,697 up to 1,819
Fustic .....	840 " 835	81 " 207
Brazil wood.. "	500 " 1,329	208 " 301

The *graphite* trade resolves itself into transit from Austria to England, and back from England (Ceylon graphite) to Austria. The home consumption is:—

	1885.	1886.	1887.	1888.	1889.	1890.	1891.	1892.	1893.
Tons...	9,519	6,201	6,844	7,602	8,776	11,783	8,086	9,429	10,133

Graphite is supplied chiefly from Austria, and in the second place, and in increasing quantities, from Ceylon:—

	1889.	1890.	1891.	1892.	1893.
East Indies (resp. Tons)	1,669	3,414	3,138	2,864	3,765
England..	1,347	2,119	731	150	?
Holland.....	8694	7,820	7,822	8,450	8,257
Austria.....	324	614	553	639	789
Other States .....					

Whilst dye-woods and graphite are the main imports, *heavy spar* and *dye-earths* are the principal exports. The former is exported to England, Austria, the United States, and Russia. On the other hand, barium carbonate (Witherite) is imported from England and barium sulphate from Belgium. The imports are valued at 8—10 marks per 100 kilos., the exports at 5—6 marks. The exports of *dye-earths* varies between 8,500—9,700 tons, with the exception of the year 1892, when they reached a total of 10,997 tons. The exports are valued at 13—14 marks per 100 kilos., against 10—12 marks for the imports. The latter are increasing, and the main sources are France, England, Italy, Austria, and Belgium.

Among "*others*" crude chalk is the most important as regards quantity. The variations in the values of these articles is due to the circumstance that up to 1887 there were included therein "unspecified dyeing and tanning materials," and which have since been classified among manufactured goods.

The imports of *bark* and *tanning liquid* reached a maximum in 1890, falling off in 1892 to 95,000 tons and in 1893 to 96,400 tons. They are derived chiefly from Austria, France, and Belgium, and their value varies between 9·5—11·5 marks per 100 kilos. The reason of the recent decrease lies chiefly in the competition of uncut Quebracho wood; this Argentine product was imported to the extent of 16,600—21,800 tons a year between 1881 and 1890,

## I. F. TANNING MATERIALS.

	Imports.		Exports.	
	Tons.	Thousand Marks.	Tons.	Thousand Marks.
Bark .....	61,813 up to 103,441	4,778 up to 12,653	2,810 up to 5,207	267 up to 373
Galls, knopperrn, &c. ....	6,132 .. 10,507	4,172 .. 6,578	89 .. 124	84 .. 277
Tanning extracts .....	5,077 .. 8,531	2,285 .. 4,049	171 .. 1,685	85 .. 644
Sumach .....	4,802 .. 8,135	1,123 .. 1,626	222 .. 886	45 .. 195
Quebracho .....	4,339 .. 50,226	284 .. 2,511	7 .. 1,044	1 .. 63
Dividivi .....	1,964 .. 4,708	215 .. 1,224	19 .. 859	5 .. 227
Others .....	2,719 .. 4,205	456 .. 710	205 .. 391	39 .. 83
	91,296 .. 177,130	18,994 .. 25,714	4,582 .. 6,072	684 .. 1,698

50,200 tons in 1891, 27,100 tons in 1892, and 39,000 tons in 1893. The values were:—1886–90, 6·5–9 marks per 100 kilos.; 1891–93, 5 marks. In *galls* distinction is made since 1889 between Knopperrn, Eckerdopperrn, and Valonia. The former are of Chinese and Turkish origin, and their home consumption amounts to 2,000–2,400 tons, valued at 2½–3 million marks. The use of *sumach* is on a steady increase, with the exception of 1893, when a drop to 6,590 tons has taken place. The average value decreased from 25 marks for 100 kilos. in 1885 to 18–20 marks. There was considerable and rapidly increasing demand for *Dividivi*, the values varying between 20–26 marks per 100 kilos. The importing countries were Venezuela, Curacao, Columbia, Hayti, &c. There is a distinct increase in the exports of *tanning extracts*, chiefly for Holland, Belgium, Scandinavia, Russia, and the States. The importing countries are Austria, France, and Belgium.

The average values for 1885–90 were from 45–50 marks per 100 kilos., for 1891 40 marks, and since then 30 marks. An addition of 5 marks may be made for exports.

In summarising the trade in the raw materials of the chemical industry and pharmacy we find that the import values suffered a *decrease* in cream of tartar, china bark, sponges, bone charcoal, beer yeast and pressed yeast, krapp, and cochineal. An *increase* has taken place in sulphur, camphor, drugs not specially enumerated, turpentine-resin, gum arabic, resins not enumerated, graphite, dividivi, and quebracho. The exports of the more important commodities show a considerable decrease in the case of bone charcoal and yeast; an increase in camphor, turpentine-resin, tanning extracts (since 1888), gum arabic (since 1889), gum lac, resins, and residues for the manufacture of glue.

The *manufactured products* show a different picture:—

	Imports.		Exports.	
	Thousand Tons.	Million Marks.	Thousand Tons.	Million Marks.
Chemically simple materials, &c. ....	75·11 up to 88·56	23·18 up to 32·18	204·26 up to 294·96	47·73 up to 77·23
Ether, medicines, &c. ....	6·29 .. 6·93	10·27 .. 12·35	28·78 .. 40·40	26·58 .. 33·11
Resin oils, lacquers, &c. ....	12·88 .. 19·14	8·65 .. 11·04	11·61 .. 16·90	8·31 .. 10·12
Explosives and matches .....	0·66 .. 2·83	0·72 .. 4·72	7·69 .. 11·04	12·85 .. 18·75
Pitch, tar, and distillates .....	47·69 .. 74·43	9·36 .. 21·36	25·32 .. 40·58	7·24 .. 15·61
Writing and drawing materials, colours.	27·65 .. 34·42	20·18 .. 42·46	55·27 .. 72·83	81·73 .. 108·20
Total .....	178·30 .. 219·92	87·16 .. 111·94	341·16 .. 433·97	190·45 .. 254·92

The import values are irregular, but the export values have an upward tendency, with the exception of one year

(1886). As regards the individual items, the following details ought to be mentioned:—

## II. A. CHEMICALLY SIMPLE SUBSTANCES, ALKALIS, ACIDS, AND SALTS.

	Imports.		Exports.	
	Tons.	Thousand Marks.	Tons.	Thousand Marks.
Ammonium sulphate .....	31,110 up to 36,558	7,466 up to 8,959	61 up to 957	15 up to 230
Iodine .....	438 .. 169	3,463 .. 4,479	9 .. 14	237 .. 315
Phosphorus .....	136 .. 521	481 .. 1,067	10 .. 40	48 .. 410
Borax and boric acid .....	1,337 .. 4,297	862 .. 2,504	801 .. 1,758	497 .. 1,171
Chlorate of potash and of soda .....	550 .. 917	495 .. 1,100	94 .. 682	127 .. 920
Bromate of potash .....	796 .. 1,739	319 .. 1,161	107 .. 276	82 .. 188

II. A. CHEMICALLY SIMPLE SUBSTANCES, ALKALIS, ACIDS, AND SALTS—*cont.*

	Imports.		Exports.	
	Tons.	Thousand Marks.	Tons.	Thousand Marks.
Lime salts .....	813 up to 7,422	315 up to 1,602	186 up to 1,517	204 up to 394
Chloride of potash .....	294 .. 1,804	41 .. 253	57,908 .. 88,958	9,012 .. 12,154
Sulphate of potash .....	265 .. 1,548	21 .. 170	16,104 .. 32,558	1,577 .. 2,767
Saltpetre .....	14 .. 1,783	36 .. 749	3,124 .. 10,135	1,187 .. 1,558
Potash .....	1,324 .. 2,163	431 .. 717	9,203 .. 12,233	3,221 .. 5,015
Prussiates .....	6 .. 68	10 .. 113	260 .. 586	430 .. 1,061
Iodide of potash .....	1 .. 6	41 .. 205	98 .. 109	2,397 .. 2,951
Soda ash .....	118 .. 2,023	18 .. 213	11,981 .. 35,350	1,318 .. 1,240
Caustic soda .....	350 .. 3,262	84 .. 718	1,071 .. 5,819	246 .. 1,571
Saltlake .....	309 .. 3,227	31 .. 129	15,003 .. 57,103	740 .. 1,175
Ammonium carbonate and chloride ....	615 .. 1,587	461 .. 194	927 .. 3,094	630 .. 1,048
Alum .....	157 .. 462	19 .. 60	7,812 .. 15,895	1,172 .. 2,046
Copper sulphate .....	374 .. 1,105	125 .. 381	1,398 .. 4,323	438 .. 1,729
Sulphuric acid .....	6,350 .. 8,708	429 .. 701	15,159 .. 20,313	1,094 .. 1,324
Tartaric acid .....	15 .. 47	56 .. 154	777 .. 1,598	1,942 .. 4,188
Salicylic acid .....	1 .. 4	13 .. 48	129 .. 303	1,672 .. 3,329
Tannic acid .....	16 .. 95	8 .. 71	274 .. 605	824 .. 1,572
Products not enumerated .....	1,321 .. 5,598	1,982 .. 8,907	3,541 .. 10,525	8,853 .. 19,643
Others (not estimated) .....	.. ..	.. ..	.. ..	.. ..
	75,105 .. 288,555	23,982 .. 32,176	204,262 .. 294,963	47,727 .. 77,235

—II. A.

## THE NEW SODIUM NITRATE DEPOSITS OF COLUMBIA.

Through the courtesy of the *Panama Star and Herald*, we have been able to obtain the following additional information concerning the discovery of deposits of sodium nitrate in the Columbian States. The deposits were found in the department of Magdalena, extending over a very wide area, and as regards quality and appearance the Columbian nitrate is said to closely resemble the Chilean. It is asserted, indeed, that the deposits possess certain advantages that will allow of their competing very successfully with the Chilean nitrates.

The discovery was made by M. C. F. Z. Caraeristi, in the neighbourhood of the Sierra Nevada de Santa Marta, and his investigations, of which the results have lately been published, seem to place the value of these new deposits beyond question. So far, it is known that the nitrates extend over a district of seventy-five square kilometres, but it is believed that they will be found to be quite as extensive as the deposits of Chili. The layer of mineral, which has a thickness of from thirty centimetres to three metres, has been cut through at depths varying from 2·40 metres to 6 metres. It is covered by a stratum composed of sulphate of calcium, oxide of iron, and other minerals, while underneath is a layer of sandstone, which has prevented the nitrates from sinking through the lower strata. Being so thoroughly protected, the mineral is found occasionally in a pure state, in the form of crystals, in which the percentage of nitrate is about 24 per cent., the other elements being chloride of sodium and insoluble silicates. These crystals, however, compose a small part of the deposits, and the mineral available for working contains from 11 to 12 per cent. of nitrates. The other constituents are carbonate of calcium, sulphate of calcium, and silicates. It will be seen, therefore, that the mineral is a very valuable agent for fertilising

purposes, and if worked upon an extensive scale is sure to be in large demand.

Of course it is impossible at the present moment to give an accurate estimate of the amount of nitrates available for working, but an engineer, who lately arrived in the country with a view of ascertaining the richness of the deposits, is of opinion that at least 7,380,000 tons of mineral can be produced. Besides nitrates, any country undertaking to develop this territory could make a considerable profit out of the enormous quantities of iron pyrites that are to be found in the adjoining district. These pyrites are said to contain more than 42 per cent. of sulphur. In view of the splendid returns that are likely to result from the investment of money in the nitrate industry of Columbia, these new fields, from which the most satisfactory returns are to be expected, should not fail to attract the notice of American and European capitalists.

One of the principal advantages of the nitrate field of Columbia is its favourable situation for transport. The mineral lies at a distance of about 65 miles from the town of San Juan de la Ciénaga, and some of it is to be found in the basin of the San Sebastian River, which, by means of the Ciénaga Grande Lake, is in direct communication with the sea. Certainly, some work would be necessary to make the waterway navigable the whole length of its course, but it is estimated that this could be done for something like 1,000/. As regards railroad transport also, exceptional facilities will be afforded for the conveyance of the mineral to the port of shipment. When the line from Santa Marta is completed, it will pass within half a mile of the nitrate fields, and, as the railroad is already laid down as far as Rio Frio, it only remains for another 62 miles to be constructed.—*Chemical Trade Journal*.

## BOARD OF TRADE RETURNS.

## SUMMARY OF IMPORTS.

Articles.	Month ending 29th September.	
	1893.	1894.
	£	£
Metals.....	1,687,076	1,605,524
Chemicals and dyestuffs.....	401,822	381,200
Oils.....	637,235	549,473
Raw materials for non-textile industries.	4,034,494	4,271,530
Total value of all imports ....	31,378,830	30,219,436

## SUMMARY OF EXPORTS.

Articles.	Month ending 29th September.	
	1893.	1894.
	£	£
Metals (other than machinery) ....	2,300,494	2,263,814
Chemicals and medicines .....	637,277	654,719
Miscellaneous articles.....	2,379,534	2,352,216
Total value of all exports.....	18,114,129	17,590,820

## IMPORTS OF METALS FOR MONTH ENDING 29TH SEPTEMBER.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Copper:—				
Ore..... Tons	5,302	12,722	22,885	64,514
Regulus .....	11,800	9,006	292,600	176,319
Unwrought ....	4,826	3,954	218,076	160,050
Iron:—				
Ore.....	330,416	292,500	23,652	193,120
Bolt, bar, &c....	6,030	7,228	55,028	68,416
Steel, unwrought..	711	439	6,137	4,288
Lead, pig and sheet ..	15,480	15,089	152,204	141,532
Pyrites .....	40,888	50,149	70,289	86,820
Quicksilver..... Lb.	45,000	118,200	3,833	9,695
Silver ore..... Value £	..	..	218,085	209,275
Tin .....	18,134	76,687	194,585	271,315
Zinc .....	4,277	3,661	71,305	55,727
Other articles ... Value £	..	..	119,007	164,453
Total value of metals ..	..	..	1,687,076	1,605,524

## IMPORTS OF OILS FOR MONTH ENDING 29TH SEPTEMBER.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Cocoa-nut..... Cwt.	18,221	9,157	23,786	10,274
Olive .....	607	1,329	25,338	41,072
Palm .....	196,479	88,146	130,110	96,445
Petroleum .....	14,374,163	14,126,888	233,710	203,238
Seed .....	2,706	1,407	62,985	43,315
Train, &c..... Tons	1,539	2,538	31,763	40,528
Turpentine .....	46,706	41,844	56,495	42,182
Other articles .. Value £	..	..	79,349	66,389
Total value of oils...	..	..	637,235	549,473

## IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDING 29TH SEPTEMBER.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Bark, Peruvian .. Cwt.	2,449	4,338	5,648	5,160
Bristles..... Lb.	395,555	486,880	54,841	70,536
Caoutchouc..... Cwt.	23,762	18,815	292,004	187,831
Gum:—				
Arabic.....	2,939	2,875	8,857	6,983
Lac, &c.....	2,470	5,642	12,521	31,112
Gutta-percha ....	1,264	2,652	10,726	23,505
Hides, raw:—				
Dry.....	19,857	27,121	55,954	66,826
Wet.....	67,444	51,050	138,083	99,573
Ivory .....	544	1,245	24,643	45,756
Manure:—				
Guano..... Tons	500	1,645	4,096	11,850
Bones.....	1,679	4,758	7,413	22,179
Nitrate of soda... ..	4,051	7,965	36,441	70,581
Phosphate of lime ..	30,326	38,059	52,220	70,056
Paraffin..... Cwt.	55,692	58,186	58,264	39,063
Linon rags..... Tons	1,776	1,773	18,057	13,050
Esparto.....	11,766	15,680	53,615	67,613
Pulp of wood ....	19,054	21,028	105,423	110,853
Rosin..... Cwt.	141,113	216,617	47,412	61,210
Tallow and stearin ..	149,968	163,830	214,550	208,400
Tar .....	22,753	32,399	11,281	23,314
Wood:—				
Hewn .....	215,948	255,633	457,469	449,751
Sawn .....	648,384	729,333	1,444,366	1,569,988
Staves .....	19,884	17,327	53,040	54,466
Mahogany .....	3,960	8,059	34,692	60,955
Other articles.... Value £	..	..	831,648	901,306
Total value .....	..	..	4,034,494	4,271,530

Besides the above, drugs to the value of 66,956*l.* were imported, as against 61,852*l.* in September 1893.

IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH  
ENDING 29TH SEPTEMBER.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Alkali..... Cwt.	6,011	13,541	£ 5,494	£ 10,718
Bark (tanners', &c.) ..	36,323	34,122	12,718	13,216
Brimstone..... "	67,127	34,155	18,349	7,206
Chemicals..... Value £	..	..	98,668	89,893
Cochineal ..... Cwt.	438	414	2,982	2,402
Cutch and gambier Tons	1,920	1,589	42,929	35,178
Dyes:—				
Aniline..... Value £	..	..	18,023	32,143
Alizarin ..... "	..	..	31,474	17,605
Other ..... "	..	..	1,633	442
Indigo ..... Cwt.	729	797	13,938	10,538
Nitrate of potash . "	15,892	25,337	14,767	21,577
Valonia ..... Tons	3,672	2,007	47,745	21,740
Other articles... Value £	..	..	92,602	115,641
Total value of chemicals	..	..	401,322	381,209

EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING  
29TH SEPTEMBER.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Alkali..... Cwt.	350,743	499,663	£ 114,663	£ 136,794
Bleaching materials ..	73,247	114,585	26,912	45,412
Chemical manures. Tons	56,765	34,452	201,394	171,920
Medicines..... Value £	..	..	76,645	75,592
Other articles ... "	..	..	214,723	214,222
Total value .....	..	..	637,277	654,719

EXPORTS OF METALS (OTHER THAN MACHINERY) FOR  
MONTH ENDING 29TH SEPTEMBER.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Brass..... Cwt.	10,154	8,495	£ 38,984	£ 30,190
Copper:—				
Unwrought..... "	42,551	50,969	96,900	67,200
Wrought..... "	30,291	28,706	85,845	73,986
Mixed metal .... "	25,698	28,320	58,705	59,238
Hardware..... Value £	..	..	175,562	155,264
Implements..... "	..	..	195,846	91,897
Iron and steel..... Tons	234,024	258,978	1,601,168	1,572,959
Lead ..... "	3,554	4,026	40,467	44,384
Plated wares... Value £	..	..	26,010	26,426
Telegraph wires ..	..	..	32,865	49,820
Tin ..... Cwt.	13,418	7,772	57,765	28,480
Zinc ..... "	14,074	16,755	10,673	11,101
Other articles .. Value £	..	..	59,714	52,878
Total value .....	..	..	2,360,494	2,263,814

EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH  
ENDING 29TH SEPTEMBER.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Gunpowder..... Lb.	557,300	587,100	£ 14,936	£ 12,412
Military stores.. Value £	..	..	98,432	100,809
Candles..... Lb.	1,493,300	1,702,900	28,728	28,125
Caoutchouc.... Value £	..	..	108,182	92,272
Cement..... Tons	32,983	42,668	54,586	70,969
Products of coal Value £	..	..	94,592	86,674
Earthenware ... "	..	..	119,725	151,146
Stoneware..... "	..	..	12,156	14,174
Glass:—				
Plate..... Sq. Ft.	140,514	83,624	7,114	5,000
Flint..... Cwt.	7,623	7,790	21,035	18,063
Bottles..... "	64,871	55,271	31,593	26,756
Other kinds.... "	18,277	16,800	14,154	13,192
Leather:—				
Unwrought .... "	12,288	9,940	110,268	92,591
Wrought ..... Value £	..	..	35,035	31,309
Seed oil..... Tons	4,826	3,753	106,365	79,551
Floorcloth ..... Sq. Yds.	1,389,390	1,557,000	56,141	60,249
Painters' materials Val. £	..	..	109,640	110,129
Paper ..... Cwt.	76,611	68,257	122,772	108,960
Sacks..... Tons	2,718	4,250	16,574	25,698
Soap..... Cwt.	51,136	41,221	54,139	49,226
Total value .....	..	..	2,879,534	2,352,216

## Monthly Patent List.

\* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### APPLICATIONS.

17,666. S. Trivick, P. A. J. Head, and J. Elsner. Improvements in furnaces for metallurgical and other purposes. September 17.

17,836. W. W. Wardle. Improvements in drying apparatus. September 19.

17,848. C. J. Ollagnier. See Class XVII.

18,173. R. Pearson. Improvements in the method of evaporation of liquids, water, brine, &c. September 25.

18,214. H. H. Lake.—From R. Schicht, Austria. An improved method of and apparatus for drying substances which are viscous or semi-fluid in the hot state. Complete Specification. September 25.

18,462. T. W. F. Cherry. Improvements in taps for use with chemical and other fluids. September 29.

18,591. T. Hewitt. Improvements in cooling and freezing machines. October 1.

18,727. G. Krell. See Class VII.

18,828. E. Theisen. Improvements in apparatus for condensing, evaporating, or refrigerating fluids. October 4.

18,992. H. L. Doulton and A. W. Manger. Improvements in the process of and apparatus for softening waters and purifying waste liquors. Complete Specification. October 6.

19,042. J. C. S. McLay. Improvements in heat non-conducting substances. October 8.

19,085. A. Müller. Improvements in apparatus for effecting the absorption of gases by liquids and for cooling gases. Complete Specification. October 8.

19,087. R. Tervet, jun. Improved apparatus for extracting and purifying sulphur, oils, and other substances. October 8.

19,128. W. Hood. Improvements in hydrometers, salinometers, and such like instruments. October 9.

19,169. G. L. F. Edeline. An improved composition for the prevention of incrustation in boilers. Complete Specification. October 9.

19,170. H. Prince. Improvements in apparatus for rowing liquors. October 9.

19,175. A. Gray. Improvements in apparatus used in the manufacture of paper, linen, or other fabrics, coated or impregnated with celluloid, or varnish, or the like. October 9.

19,293. J. L. Mitton and T. J. L. Mitton. Improvements in retort mouthpieces and self-sealing lids for same. October 11.

19,488. T. Scott and The Patent Salt Co., Lim. Improved evaporator for obtaining common salt and for analogous purposes. October 13.

#### COMPLETE SPECIFICATIONS ACCEPTED.\*

1893.

19,618. E. J. Preston. Improvements in apparatus for intensifying fluid pressure. October 17.

22,681. G. Axdorfer and C. F. H. Sass. Improvements in apparatus for mixing gas and air for gas furnaces. September 26.

23,124. G. Dasse and C. Fettweis. Improvements relating to drying and carbonising apparatus for textile and other matters. October 10.

1894

12,880. G. A. Barnard. Improvements in apparatus for cooling or condensing fluids. October 17.

15,413. E. Fudlickar. Improvements in and relating to apparatus for heating liquids, air, and other fluids. October 10.

15,881. W. Oliphant. Improvements in filtering apparatus. October 3.

16,933. E. Herscher. An improved continuous apparatus for the sterilisation of water or other liquid by ebullition. October 10.

### II.—FUEL, GAS, AND LIGHT.

#### APPLICATIONS.

17,679. F. Bremme. Improved mode of treatment and apparatus for the carburisation of gases. Complete Specification. September 17.

17,693. J. W. Scott. Improvements in and relating to apparatus for extracting the solid and gaseous impurities from illuminating and other gases. September 18.

17,759. W. B. Hartridge. An improvement in artificial fuel. September 18.

17,760. W. B. Hartridge. Improvements in apparatus for the manufacture of artificial fuel. September 18.

17,873. H. T. Blake. Dowson gas enricher. September 20.

17,987. S. de la G. Williams. Improvements in and apparatus for the production of gaseous fuel. September 21.

18,078. P. Dvorkovitz. Improvements in and apparatus for enriching gases by liquid hydrocarbons. September 22.

18,991. R. Clough, M. L. Mulholland, and R. Clough, jun. Improvements in apparatus for purifying coal or the like material by the washing process. September 24.

18,237. G. Threlfall and R. J. Hodges. Improvements in gas generating apparatus. September 26.

18,248. C. E. Parker-Rhodes and S. F. St. J. Steadman. Improvements in or relating to the treatment of hydrocarbon oils, spirits, and other highly inflammable materials of a like nature. September 26.

18,369. G. Threlfall and R. J. Hodges. Improvements in and connected with gas-producing apparatus. September 28.

18,440. S. Hersey and Kirkham, Hulett and Chandler, Lim. Improvements in apparatus for washing or scrubbing gas. September 28.

18,441. Kirkham, Hulett, and Chandler, Lim.—From F. D. Marshall, Denmark. Improvements in apparatus for condensing and washing gas. September 28.

18,443. S. H. Terry. Improvements relating to the use of liquid fuel and apparatus for that purpose. September 28.

18,445. B. H. Thwaite. Improvements in gas-generating plant. September 29.

18,510. W. Kemp. Improvements in apparatus for enriching gas. September 29.

18,715. A. W. Summers. Fuel-saving compound. October 3.

18,889. R. Middleton. Improvements in machinery used in process of manufacturing fuel or other dust into blocks for use in furnaces. October 5.

19,003. A. W. Pickering. An improved method of and means for manufacturing gas of illuminating and combustible properties. October 6.

19,127. J. T. Donovan and H. L. Gardner. A new and improved process for the production of ozone. Complete Specification. October 9.

19,130. R. H. Courtenay. An improved method of rendering the combustion of coal-gas more perfect, with appliances for same. October 9.

19,179. F. A. Seigel and F. A. Solbrig. Improvements relating to the protection of the mantles and chimneys of incandescence gas lights. October 9.

19,457. T. Clapham and J. Laycock. Improvements in apparatus for use in the purification of coal or other illuminating gas. October 13.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

#### APPLICATIONS.

17,617. W. R. Hutton. Improvements in apparatus for the distillation of coal, lignite and dross. September 17.

### IV.—COLOURING MATTERS AND DYES.

#### APPLICATIONS.

18,272. L. Lederer. Process for obtaining pure phenols from mixtures. September 26.

18,429. A. Feer. Improvements relating to the preparation of diazo salts, and to the use of the same for dyeing and printing. September 28.

18,516. A. M. Clark.—From A. Edinger and A. Muller, Germany. The manufacture of alkylrhodonates and acid chlorides of chinolin, pyridin, antipyrin, and isochinolin, and their derivatives. September 29.

18,578. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of colouring-matters. October 1.

18,588. C. D. Abel.—From the Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of new condensation products, and colouring-matters derived therefrom. October 1.

18,589. O. Imray. From the Society of Chemical Industry in Basle, Switzerland. Manufacture of basic orange-yellow colouring-matters. October 1.

19,187. J. Y. Johnson.—From C. F. Boehringer and Soehne, Germany. Improvements in the manufacture of the alkyl-ethers of acylized *p*-amido-phenols. October 9.

19,188. J. Y. Johnson.—From C. F. Boehringer and Soehne, Germany. An improved process for the manufacture of lactyl-*p*-phenetidine. October 9.

19,420. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of a colouring-matter to dye cotton without mordant. October 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

18,769. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik. Improvements in the manufacture of colouring-matters. October 10.

22,741. O. Imray.—From The Society of Chemical Industry in Basle. Manufacture of blue acid colouring-matters of the triphenylmethane series. September 26.

23,310. C. D. Abel.—From the Actien Gesellschaft für Anilin Fabrikation. Manufacture of new bases and their sulpho-acids. October 3.

23,311. C. D. Abel.—From the Actien Gesellschaft für Anilin Fabrikation. Manufacture of colouring-matters, dyeing blue with mordants. October 10.

23,392. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of colouring-matters. October 10.

23,585. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of colouring-matters derived from anthraquinone. October 10.

### V.—TEXTILES, COTTON, WOOL, SILK, Etc.

#### APPLICATIONS.

17,625. A. Stephenson. Improvements in apparatus for scouring and washing wool and the like. Complete Specification. September 17.

17,911. L. Wilson. Improvements in apparatus for drying and otherwise treating yarn in cop or other similar compact form. September 20.

18,463. J. Ingleby. Improvements in the manufacture of linoleum and cork carpets, and apparatus or machinery therefor. September 29.

19,485. A. F. B. Gomes. An improved process for the treatment of textile vegetable fibres, more especially applicable to those of the urtica family. October 13.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

23,052. J. Dawson. Improvements in apparatus for drying wool and other fibres. October 10.

23,324. G. Dasse and C. Fettweis. See Class I.

24,003. F. Lehner. Improvements in the production of and in apparatus for producing artificial silk. October 17.

1894.

15,140. A. Gruschwitz. An improved process for the retting, cleansing, and like treatment of textile fibres. September 26.

16,137. C. H. Arnold. Improvements in the manufacture of waterproof fabrics. October 3.

17,070. E. Hornung and R. Liebl. Improvements in waterproof fabrics, garments, wrappers, and coverings, and in the process and composition for manufacturing the same. October 17.

### VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

#### APPLICATIONS.

18,077. F. Anselmi and The Clayton Aniline Co., Lim. The production of new blue-azo colours on vegetable fibres or fabrics. September 22.

18,218. J. Bolet. An improved machine for printing on textile fabrics and the like. Complete Specification. September 25.

18,249. J. Read. Improvements in machines for washing or scouring textile fabrics. September 26.

18,258. E. Potter and Co., Lim., and J. Barr. Improvements in printing upon calico and other woven fabrics. September 26.

19,226. W. H. Thorpe. Improvements in machinery for mordanting, dyeing, washing, and scouring banks of yarn. Complete Specification. October 10.

19,235. T. Ingham. Improvements in dyeing mixed woven or knitted fabrics. Complete Specification. October 10.

#### COMPLETE SPECIFICATION ACCEPTED.

1893.

22,498. A. J. Boulton.—From P. V. Rennard. Improvements in or relating to the clouding of textile materials in dyeing. October 17.



## VII.—ACIDS, ALKALIS, AND SALTS.

## APPLICATIONS.

17,978. K. J. Bayer. Improvement in the manufacture of chlorine acid potash, or chlorate of potash. September 21.

18,031. G. Millard. Improvements in the art, trade, or manufacture of bisulphide of carbon.

18,206. W. Walker, jun., and F. R. Wilkins. Improvements in utilising residual products obtained in working certain kinds of voltaic batteries. September 25.

18,519. J. Pfleger. Improvements relating to the manufacture of cyanides and to apparatus therefor. September 29.

18,526. J. Hargreaves and T. Bird. Improvements in the manufacture of chlorates of sodium and potassium. October 1.

18,727. G. Krell. Improved apparatus for concentrating and distilling sulphuric acid and other liquids of high boiling point. October 3.

18,791. W. McD. Mackey and J. F. Hutcheson. Improvements in and relating to the production of ammonia. October 4.

18,792. W. McD. Mackey and J. F. Hutcheson. Improvements in and relating to the production of cyanides and ammonia. October 4.

18,940. W. Whitehead and C. Gelstharp. Improvements relating to the separation of arsenic, antimony, bismuth, and other metals from spirit of salt (hydrochloric acid) oil of vitriol, or sulphuric acid and other acid solutions and to apparatus therefor. October 5.

18,995. J. J. Crosfield and K. E. Markel. Improvements in the process of evaporating silicate of soda solutions. October 6.

19,174. D. Levat. Improvements in the treatment of natural phosphates. October 9.

19,189. T. T. Best and J. Brock. Improvements in the manufacture of chlorate of soda. October 9.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

21,239. J. J. Hood and A. G. Salamon. Improvements in the preparation of cyanogen compounds. October 10.

21,451. J. Brock, A. E. Hetherington, F. Hurter, and J. Raschen. Improvements in the manufacture of sulphocyanides, in the recovery of by-products, and in apparatus therefor. October 17.

1894.

4916. A. J. Bonlt.—From E. Warzee, Belgium. Process for the separation and precipitation of salts of iron and zinc from solutions containing both. October 17.

16,067. J. Grossman. Improvements in the preparation and manufacture of hydrosulphurous acid and hydrosulphites. October 3.

16,068. J. Grossman. Improvements in the preparation and manufacture of hydrosulphurous acid and hydrosulphites. October 17.

17,275. T. Craney. Improvements in salt evaporators. October 17.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

## APPLICATIONS.

18,256. J. L. Wittwe. Glass which consists of spun glass. September 26.

18,397. The Worcester Royal Porcelain Co., Lim., W. M. Binns, and G. H. Hancock. Improvements in the manufacture of decorated fictile-ware. September 28.

19,515. C. Jousset and F. R. Boardman. Improvements in a composition which will adhere to glass, and is for the purpose of diffusing and colouring the light. October 13.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

16,156. J. Gratton. New design for a potter's oven bottom. October 3.

22,637. J. B. Hanquiaux. Improvements in ovens for baking or firing ceramic ware and other refractory products. September 26.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

## APPLICATIONS.

17,621. W. Smith. Improvements in the manufacture of bricks or building blocks. September 17.

17,868. C. Musker and A. Musker. Improvements in or connected with machinery for making paving or building slabs or blocks and other articles. September 20.

17,920. W. Clark and A. W. Rammage. Improvements in fireproof floorings. September 20.

17,947. J. Ryder and Sons, Lim., and J. Ryder. Improvements in the manufacture of red bricks, terra-cotta, tiles, and similar goods. September 21.

18,210. G. S. Cory. An improved composition for roads, pavements, and floors. September 25.

18,233. J. T. Pearson. Improvements in pavements and material applicable thereto. September 26.

19,080. G. Falconnier. Improvements in or relating to the construction of transparent or translucent walls, ceilings, floors, or the like. October 8.

19,180. C. A. Balph and E. P. S. Wright. Improvements in and relating to fireproof floors and ceilings. Complete Specification. October 9.

19,325. T. R. W. Mossman. An improvement in the construction of fireproof floors and roofs. October 11.

19,437. E. Tortora. Improvements relating to the treatment of terra-cotta, wood, clay, gypsum, limestone, and other materials for preserving and other purposes. Complete Specification. October 12.

19,525. J. H. Barry. Improved paving by the utilisation of waste materials. October 13.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

21,292. A. Patrick. Improvements in the manufacture of Portland cement, pigments, and other finely pulverised materials, and in apparatus therefor. September 26.

1894.

5071. G. W. A. Stein. Improved process for manufacturing hydraulic cement. September 26.

14,699. W. Whittaker. Improvements in fireproof and waterproof ceilings and flues. October 17.

15,231. C. Howard. Improved method of vulcanising and drying wood. September 26.

## X.—METALLURGY, MINING, ETC.

## APPLICATIONS.

16,003A. H. Ellis and H. G. Coleby. A new alloy applicable for umbrella fittings, parts of bicycles, and for other uses, where a non-corrodible, strong, and light metal is required. Filed September 17. Date claimed August 22, 1894.

17,666. S. Trivick, P. A. J. Head, and J. Elsner. See Class I.

17,820. W. P. Thompson.—From O. Nicolai, Germany. An improved solder for soldering aluminium, aluminium alloys, and other metals. September 19.

17,961. H. R. Angel. Improvements in the extraction of lead with silver, gold, or other minerals contained in sulphide of zinc. September 21.

17,967. J. Mactear. Improvements in the obtainment of precious metals. September 21.

17,974. A. J. Boulton.—From A. L. Graut and C. G. Richardson, United States. Improvements in the refining of nickel and copper mattes. September 21.

18,003. H. L. Sulman. Improvements in or relating to the obtaining of precious metals from their solutions. September 21.

18,070. J. Mactear. Improvements in the obtainment of precious metals. September 22.

18,146. H. L. Sulman. Improvements in or relating to the recovery of precious metals from their ores. September 24.

18,151. F. C. May. An improved process for the extraction of gold and silver from their ores. September 25.

18,155. J. Green. Improvements connected with the manufacture of iron. September 25.

18,212. G. A. Dick. Improvements in or connected with means or apparatus for treating heated metals under pressure. September 25.

18,264. W. R. Renshaw. Improvements in the treatment for re-manufacture of light scrap iron or steel; machinery and furnace therefor. September 26.

18,279. A. E. Morgans. An improved process for extracting precious metals from their ores. September 26.

18,125. J. Mactear. Improvements in the extraction of precious metals from ores. September 28.

18,529. J. Pfeiffer. Improvements relating to apparatus for use in the manufacture of alkaline metals. September 29.

18,569. J. Harris. Improvements in apparatus for treating ores. October 1.

18,592. H. L. Sulman and F. L. Teed. Improvements in or relating to the extraction of precious metals from their ores. October 1.

18,844. R. Stone. Improvements in the manufacture and treatment of iron, steel, lead, copper, tin, zinc, and similar materials. October 4.

18,919. B. Talbot. Improvements in the manufacture of steel. Complete Specification. Filed October 5. Date applied for March 23, 1894, being date of application in United States.

18,939. J. Y. Johnson.—From N. Basset and W. de Baranoff, France. A process for the treatment of either natural or artificial clays or silicates containing aluminium. October 5.

19,091. S. Trivick. Improvements in and relating to the purification of iron and steel. October 8.

19,303. W. Hammond. An improvement in various fire-bricks and blocks for heating and annealing steel, iron, or copper-wire for special manufacture and mercantile purposes. October 11.

19,394. T. Twynam. Improvements in the utilisation of purple ore and other iron oxides. October 12.

19,510. H. G. Partin. A new alloy of aluminium. October 13.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

13,696. J. B. Torres. A new or improved furnace with fixed crucible for and connected with the metallurgical and siderurgical treatment, reduction and refining of ores and metals. September 26.

21,760. S. O. Cowper-Coles and Sir B. W. Walker, Bart. Improvements relating to the deposition of metals and compounds. October 3.

22,763. G. Fisher. Combined pig-iron moulding apparatus and pig carrier for blast furnaces. October 10.

23,048. F. Watkins. A process for coating iron, steel, or other metallic plates with tin, terno, and other mixtures, by the application or use of hard paraffin or paraffin wax, ozokerite, and earth wax, or a combination of either the above substances, by themselves or in combination with palm oil or palm oil mixtures. October 10.

23,636. T. Hampton. Improvements in the manufacture of armour plates and in ingots therefor. October 10.

1894.

4199. H. W. Gabbett-Fairfax, and W. Beardmore. Improved method of and apparatus for hardening or tempering steel plates and other articles. October 3.

10,478. T. W. Walker. An improved method of preparing iron or steel blooms or billets to be used in guide and other small mills. October 10.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

## APPLICATIONS.

17,741. W. P. Thompson.—From A. Lessing, Germany. Improvements in or relating to galvanic batteries. September 18.

18,172. J. J. Maclean. Improvements in and apparatus for the extraction of gold by electrolysis. September 25.

18,202. H. V. Coad. Improvements in and connected with primary batteries. September 25.

18,288. C. Riordon. Improvements in secondary or storage batteries. Complete Specification. September 26.

18,809. F. King. Improvements in or connected with secondary or electric storage batteries. October 4.

18,814. B. A. Balasny. Improvements in electric batteries. October 4.

19,150. W. H. Beck and C. R. Fletcher, United States. Improvements in the method of and apparatus for the electro deposition of metals. October 9.

19,423. J. H. Dunn. Improvements in the construction of retorts or furnaces for the production of gases by the use of electricity. October 12.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

20,975. J. A. S. Gregg. Improvements in voltaic batteries. October 10.

23,478. A. Coehn. Improvements relating to the electrolytic preparation of oxygen and the halogens, and to the simultaneous production of electrodes. October 10.

1894.

13,377. P. Jensen.—From H. C. F. Stormer. Improvements in processes and apparatus for electrolytically treating salts in solution or in molten state. September 26.

14,634. C. E. Buell. Galvanic battery. October 3.

16,002. H. A. House, H. A. House, jun., and R. R. Symon. Improvements in apparatus for refining metal by electrolysis. September 26.

16,425. D. Young.—From The Hess Storage Battery Co. Improvements in electric accumulators or storage batteries. October 10.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

## APPLICATIONS.

17,860. J. K. Hill. Improvements in the manufacture of soap and glycerine. September 20.

18,207. A. J. Inston. Improvements in recovering grease from waste suds which have been used in washing wool, hair, or other fibre. September 25.

18,422. S. Rosenblum and F. L. Bartelt. An improved washing powder. September 28.

18,891. R. Armstrong. Improvements in detergent compounds. October 5.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

18,767. F. B. Aspinall, R. W. Hoar, and G. H. Wise. Improvements in bleaching, sweetening, and purifying oils and fats. October 10.

24,556. J. V. Ruymbeke. Process of distilling glycerine and similar liquids, and of treating the residuum obtained after distilling glycerine. October 10.

1894.

9526. A. W. Winter. See Class XVIII. A.

15,228. J. O. Klmsch. Improvements relating to the manufacture of soaps. October 17.

## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

## APPLICATIONS.

17,630. K. Ammann. Improvements in washing blue. Complete Specification. September 17.

17,662. R. Hingston and J. P. Wernegreen. An improved anti-fouling composition for the prevention of marine growth in or on wood or iron. Complete Specification. September 17.

17,740. J. Morris. Improvements in or relating to the production of crystals or formation of oxide chiefly for decorative purposes. September 18.

17,747. E. W. Oliver. Improvements in paint. September 18.

18,269. J. Schmid. Improved mass as substitute for caoutchouc and process for manufacturing same. September 26.

18,906. P. Mercier. A new composition with india-rubber. October 5.

19,363. G. Rockliffe. A new anti-corrosive and non-fouling composition. October 11.

19,469. J. Noad. Improvements in blacking and other leather-dressing compositions. October 13.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

19,115. R. G. Bennett. Improvements in varnish. October 17.

20,234. P. A. Newton.—From A. Nobel. Improvements in artificial india-rubber. October 3.

20,239. J. H. Noad and S. Z. de Ferranti. Improvements in the manufacture of white lead. October 3.

21,292. A. Patrick. See Class IX.

23,028. E. M. Desprez. A new elastic material or manufacture composed chiefly of gutta-percha, india-rubber, or other analogous substances, and the application of the same to various useful purposes. October 17.

24,356. H. H. Lake.—From A. C. Girard and E. A. G. Street. An improved manufacture of writing pencils. October 17.

1894.

9823. D. Coventry. An improved composition or colour for shading glass on conservatories, or for similar purposes. October 3.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

## APPLICATIONS.

18,262. J. Baird. A novel process or means for improving the colour of raw or tanned skins, hides, and leathers, and in the employment of an ingredient therewith. September 26.

19,427. R. Ergang. Improvements in machinery or apparatus for dyeing skins. October 12.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

22,849. W. L. Jackson. An improvement in softening and cleansing hides. September 36.

1894.

11,426. P. C. Hewitt. Improvements in processes and apparatus for cooling glue and forming it into sheets. September 26.

13,369. P. C. Hewitt. Improvements in clarifying or removing impurities from glue and other substances. September 26.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

## APPLICATIONS.

17,744. L. I. N. Norman. Improvements in the manufacture of brewing syrup. September 18.

17,976. W. P. Thompson.—From H. A. J. Manoury, France. An improved method of purifying "masses cuites" and raw sugars. September 21.

18,358. H. C. E. Wilnot. Improvements in the treatment of starch or starchy matter, or crystallisable sugar, for the obtainment therefrom of products more especially suitable for use in brewing. September 27.

## XVII.—BREWING, WINES, SPIRITS, Etc.

## APPLICATIONS.

17,712. E. G. Francis. Improvements in the method of and in means for conditioning beer and other similar liquids. September 18.

17,744. L. I. N. Norman. Improvements in the manufacture of brewing syrup. September 18.

17,848. C. J. Ollagnier. Improvements in apple wine and other presses. September 19.

17,964. A. Kinder. Improvements in malt kilns. September 21.

17,965. F. W. Golby.—From J. Puybeyroux, France. A process and apparatus for distilling and rectifying alcohol. September 21.

18,014. F. W. Forbes-Ross. Improvements in or relating to brandy, whiskey, and other spirits or liquors. September 22.

18,435. A. J. Boulton.—From A. Delhaise, Belgium. Improvements in or relating to the purification of beer. September 28.

18,638. T. R. Todd. Improved apparatus for use in brewing. October 2.

19,199. A. D. Currie. Improvements in fermenting apparatus. October 9.

19,338. W. G. Johnston and J. F. Warwick. Improvements in or relating to the treatment or preparation of yeast. October 11.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

19,843. W. S. Squire. Improvements in the manufacture of yeast. September 26.

21,332. J. Heron. Improvements in the manufacture of compounds suitable for the production of ginger beer, herb, or botanic beers, or other fermented beverages. September 26.

23,846. F. Faulkner and J. E. J. Johnson. Improved manufacture or production of extracts from malted grain. October 17.

1894.

2889. G. G. Cave. Improvements in the treatment of yeast. October 10.

### XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

#### APPLICATIONS.

##### A.—Chemistry of Foods.

17,676. J. C. Benit. Improvements in preserving milk and cream. September 17.

18,133. E. Edwards.—From R. Luders, Germany. Improvements in the process of and appliances for sterilising and keeping sterilised milk in infant's feeding bottles. September 24.

18,143. W. Paasch. Improvements in pasteurising apparatus for milk, cream, or the like. Complete Specification. September 24.

18,285. G. Gaertner. Process for reducing the proportion of caseine contained in milk, and regulating simultaneously the proportion of fat. September 26.

18,535. J. Gill. Improvements in furnaces for destroying towns' refuse and the like. October 1.

19,037. J. Pumphrey. Improvements in means for preventing the destruction of food and similar substances by excessive heat. October 8.

19,064. E. Leslie. Improvements in milk sterilizers. October 8.

19,309. T. T. Chard. A new and improved phosphate food for horses. Complete Specification.

19,340. R. G. Nash. Improvements in the preservation of milk. October 11.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1894.

9526. A. W. Winter. Improvements in compound edible fats. September 26.

##### B.—Sanitary Chemistry.

18,765. W. E. Adeney and W. K. Parry. Improvements relating to the employment of aluminium, sulphate, and like clarifying agents in the purification of sewage. October 3.

19,482. H. L. Hinnell. Improvements in the construction of filter beds for the filtration of liquids from sewage sludge, mud, bleachers' dyers', tanners', and manufacturers' refuse, and in apparatus or arrangements for use therein and covering the same. October 13.

#### COMPLETE SPECIFICATION ACCEPTED.

1894.

15,000. R. A. Chesebrough. Improvements in the method of cremating garbage and other refuse. September 26.

### XIX.—PAPER, PASTEBOARD, Etc.

#### APPLICATION.

17,905. J. Baier. Improvement of imitation whalebone. September 20.

#### COMPLETE SPECIFICATION ACCEPTED.

1894.

14,384. H. O. Band. Improvements in the manufacture of parchment. October 3.

### XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

#### APPLICATIONS.

18,509. M. Otto and A. Verley. Manufacture of ethers of the alcohols contained in natural essences and of their corresponding alcohols. Complete Specification. September 29.

19,263. G. Braun. Improved method of recovering solvents used in the manufacture of pyroxylin products. October 10.

19,342. L. Lederer. Production of anilides of phenox-acetic acid and their homologues. October 11.

#### COMPLETE SPECIFICATION ACCEPTED.

1894.

15,171. P. P. Monnet. A process for the manufacture of a scent essence termed "Rhodinol." September 26.

### XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.

#### APPLICATIONS.

17,758. V. Mathieu. A process for photographing in colours. September 18.

18,282. C. Beadle. A process for sensitising photographic paper. September 26.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

21,686. W. Fairweather.—From J. Kost. A reflecting and ventilating device or apparatus for photographic exposure by the magnesium light. October 17.

1894.

14,161. J. Joly. Improvements in or relating to photographing in colours. October 3.

## XXII.—EXPLOSIVES, MATCHES, Etc.

## APPLICATIONS.

17,949. H. J. Koster. Improvements in the manufacture of matches. September 20.

17,960. A. Brock. An improved pyrotechnic compound. September 21.

17,994. H. S. Maxim. Improvements relating to smokeless explosives. September 21.

18,156. G. H. Hosie. Improvements in and relating to the manufacture of nitro-cellulose, oxy-nitro-cellulose, and hydro-nitro-cellulose. September 25.

18,496. F. W. Bawden. An improved explosive. September 29.

18,682. H. Maxim. Improvements in fulminating compounds. Complete Specification. October 2.

18,683. R. C. Schupphaus. Improvements in the process of nitrating cellulose. Complete Specification. October 2.

18,921. O. Imray.—From F. C. Glaser and Co., Germany. Manufacture of a new explosive. October 5.

19,098. A. G. Brookes.—From W. J. Smith, United States. Improvements in exploders or fuses for projectiles. Complete Specification. October 9.

19,299. G. Hübner. Improvements in smokeless explosives. Complete Specification. October 11.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

20,467. P. A. Newton.—From A. Nobel. Improvements in fuses for mining and similar purposes. October 17.

23,416. K. C. Edmunds. Improvements in explosives. October 3.

23,579. H. R. von Dahmen. An improved manufacture of explosives. October 10.

1894.

15,887. G. J. Buechert. Explosive compounds. September 26.

## XXIII.—ANALYTICAL CHEMISTRY.

## COMPLETE SPECIFICATION ACCEPTED.

1893.

23,039. R. W. Woosnam and the Dairy Supply Co., Lim. Improvements in apparatus specially suitable for use in estimating the amount of fat in milk and other substances. September 26.

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### NOTICES.

Notice is hereby given that, in accordance with the alterations in Rule 27 sanctioned at the Annual General Meeting in Edinburgh, all new members elected after January 1st next, and also those elected since the last Annual General Meeting, who do not pay subscription for 1894, will be required to pay an entrance fee of one guinea. In accordance with the same rule the Life Composition Fee will be 20*l.*, instead of 15*l.* as heretofore, after the 1st January next.

## BANKERS' ORDERS.

For the convenience of Members, the Treasurer has arranged with the Bankers of the Society that they shall collect subscriptions from Bankers in town and country; and Members, who have not already done so, are invited to fill up and sign the Banker's Order enclosed with the December number of the Journal, which should then be sent to the Honorary Treasurer, Mr. E. Rider Cook, East London Soap Works, Bow, E.

Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 41 of the bye-laws, the Society has the right of priority of publication for three months of all such papers. Infringement of this bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. EYRE and SPOTTISWOODE, the Society's printers and publishers, to whom all communications respecting them should be addressed.

Notice is hereby given that the next List of Members will contain the professions or business occupations of the Members of the Society. Any Member who desires to confirm or alter his description as given before election should communicate with the General Secretary.

Foreign and Colonial Members are reminded that the subscription of 25s. for 1895, payable on January 1st next, should be sent in good time to the Treasurer, in order to ensure continuity in the receipt of the Society's Journal. Any changes of address to appear in the new List of Members now in course of preparation, should reach the General Secretary not later than January 15th, 1895.

## LIST OF MEMBERS ELECTED 23rd NOVEMBER 1894.

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Brook, G. Bernard, 27, Wostenholme Road, Sheffield, analytical chemist.

Brown, G. E., 79, Disraeli Road, Putney, S.W., chemist to L. & S. W. Railway Co.

Cameron, Alex., Agricultural Laboratory, 4, Lombard Court, E.C., analytical chemist.

Case, W. T., 7, Cedar Street, New York, U.S.A., chemist.

Chaloner, G. W., Niagara Works, Eagle Wharf Road, London, N., manager of chemical department.

Coblentz, Virgil, M.A., Ph.D., College of Pharmacy, 115, West 68th Street, New York, U.S.A., chemical lecturer.

Colquhoun, W., St. Mary's Chambers, Cardiff, engineer.

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Court, Haywood, 67, Surrey Street, Sheffield, analytical chemist.

Diestel, Wm., 77, William Street, New York, U.S.A., dyestuff importer.

Evans, Jno. H., 71, Lambton Road, Cottenham Park, Wimbledon, analytical chemist.

Ewan, Thos., Ph.D., B.Sc., The Yorkshire College, Leeds, teacher of chemistry.

Fairechild, Sam. W., College of Pharmacy, 115, West 68th Street, New York, U.S.A., manufacturing chemist.

Fischer, Albert, c/o Messrs. Grant, 23, Billiter Street, London, E.C., journalist.

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Gruencklee, Carl, c/o Messrs. Chas. Pfizer and Co., 11, Bartlett, Brooklyn, N.Y., U.S.A., chemist.

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Kebler, Lyman F., B.Sc., 305, Cherry Street, Philadelphia, Pa., U.S.A., manufacturing chemist.

Marten, T. Tremills, 45, Stapleton Hall Road, Stroud Green, N., analytical chemist.

Moszczenski, J. von, 133, South First Street, Brooklyn, N.Y., U.S.A., analytical and consulting chemist.

Oakes, Geo. A., Bloomfield, N.J., U.S.A., woollen mill owner.

Parker, Chas. E., c/o Seabury and Johnson, East Orange, N.J., U.S.A., chemist.

Parker, J. Gordon, Leather Department, Yorkshire College, Leeds, chemist, Ph.D.

Reid, Walter C., 20, Percy Park Road, Tynemouth, manufacturer.

Rettie, Theodore, B.Sc., Cassel Gold Extraction Co., 13, W. Scotland Street, Kinning Park, Glasgow, metallurgical chemist.

Scott-Smith, G. E., 67, Surrey Street, Sheffield, analytical chemist.

Smith, Bernard E., 73, Westmoreland Road, Newcastle-on-Tyne, alkali works inspector.

Taylor, Albert, Technical School, Stockport, lecturer in chemistry and dyeing.

Taylor, G. Midgley, 27, Great George Street, Westminster, S.W., analytical chemist.

Tipler, Fred. C., 7, Richard Street, Crewe, analytical chemist.

Traphagen, Frank W., Ph.D., Montana Agric. Expt. Station, Bozeman, Mont., U.S.A., chief chemist.

Weston, Robt. S., B.Sc., Cinclare Central Factory, Brusley Landing P.O., La., U.S.A., chemical director of sugar factory.

Woltereck, Dr. H. C., 35, Wall Street, New York, U.S.A., consulting chemist.

## CHANGES OF ADDRESS.

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Barrie, D. McL., 1/o Johannesburg; retain Journals until further notice.

Chadwick, L. N., 1/o Elsham Road; 30, Avonmore Gardens, Kensington, W.

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Elworthy, H. S., 1/o 1; 19, Hill Road, Bandra, Bombay.

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Galt, Hugh A.; Journals to Michigan Alkali Co., Wyandotte, Mich., U.S.A.

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Hall, Jno. A., 1/o Manchester; Victoria Chemical Works, Victoria, British Columbia.

Jones, W. Norris, 1/o Weston; The Lancashire Metal Works, Widnes.

Krause, O. H.; Journals to c/o American Sugar Refining Co., Jersey City, N.J., U.S.A.

Lawson, Dr. T. A., 1/o 15; 111, Alexandra Road, N.W.

Loewenthal, Dr. R., 1/o Steglitzerstrasse; Magazinstrasse 12a, Berlin, O.

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Steel, R. Elliot, 1/o Plymouth; Northampton and County School, Northampton.

Taylor, Ambrose; Journals to Madron, Penzance, Cornwall.

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Winstanley, H., 1/o Caldercruix; Cale Green Villa, Stockport.

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#### SESSION 1894-95.

Monday, December 3rd.—

Mr. E. W. Kuhn (of Paris). "The Rational Sterilisation of Alimentary Liquids."  
Dr. B. H. Atfield, M.A., D.P.H. "An Investigation of the Natural Sodium Sulphate Lakes of Wyoming, U.S.A."  
"Specimens of India-rubber, and Petroleum Oil, Varnish, and Soap," will be exhibited by Mr. Thos. Christy, F.R.S.

Meeting held Monday, November 5th, 1894.

MR. B. E. R. NEWLANDS IN THE CHAIR.

The Session for 1894-95 commenced on Monday, November 5th.

After some brief opening remarks from the new Chairman of the Section, Mr. B. E. R. Newlands, and the reading of the Minutes of the last meeting by the Secretary, Mr. Watson Smith was called upon to read the following posthumous paper by Dr. C. R. Alder Wright. He said:—

"With regard to the paper I am about to read, when our Secretary, Mr. Jno. Heron, wrote and told me Dr. Alder Wright had promised to prepare one for the autumn session of the London Section, I found that Mrs. Alder Wright was unaware that such a paper existed.

"On looking among Dr. Wright's papers however, relating to his work on the alloys, we found last of all a bundle of papers forming a rough draft, with empty spaces left for references and for two illustrations.

"Mrs. Wright then remembered how that the week before his death our friend had laboured to complete this paper, and so fulfil his promise to our Secretary and to the Section. She recalls that on finishing the paper, he seemed much exhausted, and exclaimed, 'There, I have done all I shall ever do on that subject, and now I shall take some rest.'

"Somewhat remarkable, too, are the words he wrote on the cover of the paper, 'Final Paper for the Society of Chemical Industry.'

"I have filled in the references required, and have discovered one of the figures referred to, in the Proc. Roy. Soc., but I cannot find the second, and must omit it.

"I may perhaps add that it is at the request of his widow that I have undertaken to revise and to read this, Dr. Wright's, last scientific contribution.

"Probably no more fitting memorial of an eminent man of science could be imagined than that now left behind him by Dr. Alder Wright himself, in the presentation of the series of experimental essays to the Royal Society on the scientific aspects of this question of the alloys, now completed in the presentation of the paper before us on the technological and other bearings of the subject."



## THE COMPOSITION AND CONSTITUTION OF CERTAIN ALLOYS.

BY DR. C. R. ALDER WRIGHT, F.R.S.

For some years past the author has devoted a large amount of time to a laborious experimental inquiry into the subject of the relations of metals to one another as regards their mutual miscibility or solubility when rendered fluid, by heat; many of the results obtained have from time to time been communicated to the Royal Society, and abstracted from the proceedings of that Society to the pages of this Journal.\* The present paper has for its object partly to collect together and epitomise the general conclusions that may be drawn from these particular researches, so far as at present completed, and partly to discuss the subject in general; more especially since a good deal of confusion appears to exist in the minds of various writers between certain phenomena which, though allied, are really sharply distinguishable from one another; whilst a number of the statements on the subject, repeated from text-book to text-book till fossilised, are in point of fact considerably inexact.

When two metals are melted separately and the one fluid poured into the other and the whole vigorously stirred, it sometimes happens that more or less complete solidification is brought about on account of chemical action taking place between the two, resulting in the formation of a definite atomic compound which is solid at the temperature of the crucible, and is not wholly dissolved in whatever excess of either constituent may happen to be present. This solidification, however, is comparatively of rare occurrence; in any case it can always be avoided by employing a temperature sufficiently high to keep the mass perfectly liquid, in which case one of two things happens: either the two metals intermix perfectly, mutually dissolving each other so as to form a single permanent homogeneous fluid that remains uniform in character so long as the temperature is maintained high enough to prevent incipient solidification; or, they form two different liquids not miscible with one another, and separating from one another by gravitation on standing molten for a sufficiently long period; these fluids consisting respectively of the first metal containing in solution a saturating amount of the second, and *vice versa*. The two cases are precisely correlative with the behaviour of alcohol and water when intermixed at the ordinary temperature, and that of ether and water employed in proportions not outside of certain limits, *e.g.*, used in equal volumes.

In some cases the separation by gravitation only takes place slowly, notwithstanding that a notable difference in density may subsist between the two binary alloys formed; many hours or even days standing molten at an equable temperature may be requisite before approximately complete separation is effected, the two fluids forming a sort of emulsion, comparable with the minute oil globules suspended in milk, or with the persistent froth formed on shaking up ether with soap-water, or aqueous fluids containing various forms of organic matter. This circumstance has in some instances led to the erroneous belief that a given class of metals could be permanently mixed together in all proportions when molten, forming in all cases a homogeneous liquid, when in point of fact only metallic froths or milks are formed, consisting of two different intimately mixed alloys. In particular, aluminium and bismuth and aluminium and cadmium have been long described as being freely miscible, whereas the contrary is the actual fact.

On the other hand, if a homogeneous fluid mixture of two metals be gradually cooled down, a point is sooner or later arrived at when solid matter begins to form. In some cases, and with certain proportions of the constituents of the mixture, the temperature at which solidification begins, remains constant until the whole has solidified; this only happens when a definite compound of metals in atomic proportions is produced, no excess of either constituent being present. In general, the temperature at which solidification commences is higher than that observed later on, when more solid matter has formed, especially towards the end of the operation, the portions last solidifying often requiring a considerable reduction in temperature before they become solid. The "incipient solidification point,"\* or temperature at which solid matter first begins to form, is sometimes referred to as the "freezing point" of the mixture, but the term is an undesirable one in this connection, it being preferable to restrict the term "freezing point" to that definite fixed temperature (under constant conditions as to pressure) at which a single chemical substance (*e.g.*, water, or a definite atomic compound of metals, *e.g.*, SbAl) passes from the liquid to the solid state, this temperature remaining constant until the whole mass has solidified; whereas the "incipient solidifying point" refers to a variable temperature, *viz.*, that observed at the earliest stage of the passage from the liquid to the solid condition of certain of the constituents of a fluid mixture, leaving still fluid a "mother-liquor" of composition different from that of the solidified portion; this temperature being not only more or less markedly higher than the temperature of solidification of the last portions of the mass, but being also a value which varies even for the same constituents according to their relative proportions. Thus the beautiful experiments of Neville and Heycock (C.S.J. Trans. 1889, 666; 1890, 376; 1892, 888) have shown that when comparatively minute amounts of one metal are dissolved in a large quantity of another, the masses exhibit a series of incipient solidification points higher or lower than the true freezing point of the predominating metal by amounts closely proportionate to the weights of the other metal added; a rise being exhibited in those cases where the two metals are capable of combining chemically together, forming compounds of higher melting point than that of the predominating metal (*e.g.*, Sb dissolved in Bi or in Sn, or Ag dissolved in Cd); whilst in other cases a fall is observed, from the magnitude of which conclusions may be drawn on Raoult's principle as to the molecular aggregation of the dissolved metal. Then the effect of adding a little zinc to a large mass of bismuth or lead, is to produce a depression in the value of the incipient solidification point of the mixture below the fixed freezing point of pure lead (or bismuth) nearly proportionate to the quantity added; so that an "atomic fall" of about 1.5° in the first case and 5.0° in the second is observed; values comparable in magnitude and identical in sign with those observed when metals are employed miscible in all proportions with bismuth or lead, *e.g.*, Cu, Ag, Sn, Cd, Au. Aluminium is so slightly soluble in molten bismuth, cadmium, or lead, as to render it difficult to obtain any reliable measurements; but at any rate nothing abnormal is noticeable in its effect on these metals as compared with that in tin, with which it is miscible in all proportions when molten (Heycock and Neville, *loc. cit. supra*).

\* When a mixture of different substances in the fluid condition is slowly cooled, an immersed delicate thermometer indicates a gradually decreasing temperature until solid matter begins to form. At this point a slight rise in temperature is usually manifest on account of the mixture having become more or less superheated before the solid matter actually separated, the amount of this rise depending on the nature of the mixture, and also on the details of construction of the thermometer, its capacity for heat as compared with that of the mixture, and the amount of latent heat rendered sensible during the separation of solid matter. With a sufficiently large mass of material and proper arrangements to avoid too rapid chilling by radiation, &c., a practically constant temperature is thus indicated for some considerable period (often from 30 to 100 seconds or even more), during which the loss of heat by conduction and radiation is balanced by the evolution of heat during solidification; after this the thermometer begins to fall again. The nearly constant temperature observed during this period is usually taken as the "incipient solidification point"; the method of observation has long been well known technically in connection with the valuation of tallow, &c., as "Dallien's process" (Mon. Sci. Paris, 1868).

\* Wright and Thompson. Certain Ternary Alloys. Parts I. and II. This Journal, 1890, 211-245.

C. R. A. Wright. Certain Aluminium Alloys. This Journal, 1892, 492-493.

Wright and Thompson; Wright, Thompson, and Leon; C. R. A. Wright. Certain Ternary Alloys. Parts III., IV., V. This Journal, 1892, 245-246.

C. R. A. Wright. Certain Ternary Alloys. Part VI. This Journal, 1892, 623.

C. R. A. Wright. Certain Ternary Alloys. Part VII. This Journal, 1893, 449-450.

C. R. A. Wright. Certain Ternary Alloys. Part VIII. This Journal, 1894, 521.

The phenomenon of segregation of solid matter from a liquid mass on cooling, such solid matter being different in composition from the portion still remaining liquid, is obviously wholly different from the phenomenon of want of miscibility between two different metallic fluids where no solid matter at all is present; but in many cases the distinction has not been appreciated by writers on the subject, and notable confusion and want of exactitude have accordingly resulted. As an example of this, even in so carefully revised a work as Morley and Muir's dictionary, now just published, this statement is made (article "Copper"), that "copper and lead melted together at a red heat form alloys; but the fused mass tends to separate into two layers, the upper containing much Cu and little Pb, and the lower much Pb and little Cu." On careful examination, however, this statement is found to be incorrect; so long as the temperature is high enough to keep the whole mass fluid the fused mass does not separate into two layers any more than does a mixture of alcohol and water; but at a somewhat lowered temperature solid matter begins to separate, rendering the mass more or less pasty; this solid matter is either copper alone or copper combined with but little lead, forming a difficultly fusible alloy, just as chilled alcohol and water will allow solid crystals to form, consisting either of ice alone or possibly of a cryohydrate of alcohol. In the case of copper and lead the solid matter thus separating floats up to the top of the remaining fluid matter in which the lead is concentrated; the degree of separation depends on the rate of cooling; but the tendency towards segregation is so marked that it is almost impossible to cool a mass of 30 or 40 grms. of mixed metals (in about equal proportions) without finding a measurable excess of copper in the top layers, and of lead in the bottom ones.

The preceding remarks respecting binary metallic mixtures for the most part equally apply to ternary, and more complex alloys. These, when rendered completely fluid by heat, are divisible into two classes, *viz.*, those which form single homogeneous liquids and those which do not, but give rise to two (or possibly more) liquids not miscible together inside of certain limiting proportions. When the temperature is reduced so that solidification commences, as a general rule segregation of constituents takes place, the portion first separating in the solid form having a composition different from that of the portion still remaining liquid.

The spontaneous separation from one another of the constituents of a fluid mixture of metals is obviously a matter of considerable importance in technical operations where alloys are employed for castings, or are prepared by fusing together the constituents; whether such separation be brought about in virtue of the immiscibility of certain metallic mixtures produced on melting together the constituents, or by segregation in the solid form of certain of the constituents or definite compounds thereof in the act of cooling, in either case a want of homogeneity in the cast mass is apt to be developed which may be of serious moment, especially in the case of ingots, &c. intended to be subsequently wrought by mechanical processes. As regards segregation during the act of solidification, it is well known that the larger the mass and the more slowly the cooling is conducted, in general the more likely is the segregating action to come measurably into play; and various devices are in use in foundries to minimise the action in different cases. But as regards the formation of immiscible alloys which separate from one another whilst still fluid, very little accurate knowledge of the limits within which such separative actions occur was extant at the time of the commencement of the author's experiments on the subject; the result of these researches, however, has been to define these limits with considerable precision in the case of a very considerable number of binary and ternary mixtures of the principal metals of ordinary occurrence. In order to distinguish from one another mixtures of metals which remain homogeneous whilst molten even on long standing, and mixtures which divide into two (or possibly more) different fluid alloys on standing molten for a sufficient time, the terms "real alloys" and "ideal alloys" may be conveniently applied to the two classes respectively, this nomenclature having been

originally proposed by Sir G. G. Stokes\* to distinguish from one another certain kinds of ternary metallic mixtures.

In order to examine thoroughly into the general question of mutual miscibility or otherwise of metals, and to decide how far the statements ordinarily found in the text-books are correct or not, it was found necessary to examine in detail the behaviour of each metallic combination separately. This is a somewhat greater task than at the first blush would appear to be the case; for even when the number of metals is limited to the following nine, the total number of possible combinations is a large one. Out of the nine metals Pb, Bi, Zn, Al, Sn, Ag, Cu, Cd, Sb, no less than 502 different alloys may be formed; by taking them two at a time 36 combinations result; if three at a time, 84; if four at a time, 126; and so on, as indicated in the following table:—

No. of Combinations possible.				
No. of Constituent Metals in Mixture.	Constituent Metals miscible together in all Proportions under all Circumstances so long as Temperature high enough to maintain perfect fluidity.	Product divides into at least two immiscible Alloys when Constituents are in Proportions inside of certain Limits.	Total.	
2	31	5	36	
3	55	29	84	
4	55	71	126	
5	31	95	126	
6	9	75	84	
7	1	35	36	
8	None	9	9	
9	None	1	1	
182		320	502	

In addition to the binary and ternary combinations of these nine metals (and some few more complex mixtures), various alloys have also been experimented with to some extent containing other metals, although their peculiar physical qualities more or less interfere with the systematic determination of limits of miscibility where fusion for lengthened periods is requisite; thus the volatility of mercury and arsenic prevents their convenient use at moderately elevated temperatures, whilst the high oxidisability of sodium and potassium renders special appliances necessary in their case. Again, the high fusing points of iron, nickel, platinum, and others, and the costliness of gold and of the rarer metals, prevent their being freely employed in the simple way practicable with the nine above mentioned. In several instances indications have been obtained that interesting results would follow the extension of the systematic experiments made so far, to include a larger number of metals.

#### Binary Alloys.

On systematically examining the 36 pairs of metals obtainable from the above list of nine, from the point of view of forming "real" alloys only under all conditions, or of giving rise to "ideal" alloys when mixed in proportions inside of certain limits, and allowed to stand molten for a considerable length of time, it at once becomes manifest that in several instances the statements current in the text-books are erroneous. For instance, it is ordinarily stated (apparently on the authority of Tissier) that fused antimony and aluminium will not intermix. This, however, is not the case; when fused aluminium and antimony are stirred together in certain proportions a mixture of solid and fluid matter results: the former has been shown

\* Proc. Roy Soc. 49, 1890-91, 179.

by the author to consist essentially of a difficultly fusible\* compound of antimony and aluminium indicated by the formula  $SbAl$ ; whilst the liquid part consists of a solution of this compound in whichever of the two constituents may be present in excess. If, however, the temperature be raised sufficiently high to render the mass fluid, a perfectly homogeneous liquid results which shows no tendency to separate into two different fluids, even on standing at rest molten upwards of eight hours. Again, as above stated, copper and lead are described in various text-books as not mixing together perfectly; but the fact is that so long as the mixture is kept hot enough to be perfectly fluid a single homogeneous liquid alloy is formed. Similar remarks apply to copper and bismuth. On the other hand, according to the text-books, aluminium and bismuth unite together, forming useful alloys.

The following experiments may be quoted as examples illustrating the kind of figures obtained by operating in one or other of these ways, the alloys examined being accordingly regarded as not exhibiting the phenomenon of separation into two different binary mixtures of metals, whilst molten.†

Mixture of about one part ZINC and two parts TIN, kept Fused for Seven Hours and cooled quickly.

—	Zinc.	Tin.
Top layer of compound ingot .....	31.37	68.6
Bottom " " .....	31.13	68.87
Difference $\pm 0.24$		

Mixture of about equal Weights of TIN and CADMIUM, Fused for Six Hours and quickly cooled.

—	Tin.	Cadmium.
Top layer.....	49.37	50.63
Bottom layer .....	49.69	50.31
Difference $\pm 0.32$		

Mixture of about two parts CADMIUM and one of COPPER, Fused Six Hours and quickly cooled.

—	Cadmium.	Copper.
Top layer.....	66.20	33.80
Bottom layer .....	66.35	33.65
Difference $\pm 0.15$		

Mixture of about equal Weights of BISMUTH and COPPER, Fused Four Hours and Sampled by "SPOON and PIPETTE."

—	Bismuth.	Copper.
Top layer.....	49.51	50.49
Bottom layer.....	49.76	50.24
Difference $\pm 0.25$		

\* This Journal, 1892, 349. Prof. Roberts-Austen has subsequently determined the "solidifying point" of this compound by means of the Le Chatelier pyrometer, and found it to be practically identical with that of pure gold,  $1045^{\circ} C$ , or upwards of  $350^{\circ} C$  higher than the melting point of the highest fusing constituent. So large a rise in fusing point is a phenomenon *anterioris* amongst compounds of metals with one another, although not unknown to a lesser extent: thus Roberts-Austen (See also this Journal, 1893, 810) has shown that aluminium and gold combine to form the compound  $Al_2Au$ , which solidifies at  $1,065^{\circ}$ — $1,070^{\circ}$  or upwards of  $30^{\circ}$  above the solidifying point of the least fusible constituent.

† The existence of the atomic compound  $SbAl$  has also been confirmed by D. A. Roche (Monit. Scient., 1893, 269; this Journal, 1-93, 840).

\* The percentages are calculated throughout on the sum of the weights of the two metals found by analysis as 100.

In these instances, as in most others, the differences in composition between the top and bottom layers do not lie outside the limits of experimental error; in some few cases, however, indications were observed of a slight tendency for the lighter alloy to float up and the heavier to descend: thus, for example:—

Mixture of about three parts TIN to one of ALUMINIUM, Fused Six Hours and quickly cooled.

—	Aluminium.	Tin.
Top layer .....	22.36	77.64
Bottom layer.....	21.39	78.61
Difference $\pm 0.97$		

Inasmuch as such differences between top and bottom were never great, and also varied from experiment to experiment, they were considered to be more probably due to incipient segregation during solidification, than to the formation of true "ideal" alloys, incapable of permanent existence, and dividing into two entirely different real alloys.

Operating in one or other of the ways above described, the following 31 pairs of metals were ultimately found to produce "real" alloys, no matter what their relative proportions: *i.e.*, no natural difference was discernible between the uppermost and lowest layers of the mass obtained by melting together, thoroughly intermixing by stirring, and allowing to remain at rest molten for some time (often several hours):—

Aluminium antimony.	Bismuth tin.
" copper.	Cadmium copper.
" silver.	" lead.
" tin.	" silver.
" zinc.	" tin.
Antimony bismuth.	" zinc.
" cadmium.	Copper lead.
" copper.	" silver.
" lead.	" tin.
" silver.	" zinc.
" tin.	Lead silver.
" zinc.	" tin.
Bismuth cadmium.	Silver tin.
" copper.	" zinc.
" lead.	Tin zinc.
" silver.	

In several cases definite atomic compounds of the constituents of the above binary alloys appear to exist, so that when a molten mass containing the constituents in suitable proportions is allowed to cool slowly, the compound tends to separate from whatever excess of either constituent may be present: if the compound fuse at a higher temperature than the constituent present in excess (as, for example, the compound  $AlSi$ ), the solid matter first separating consists of the compound, leaving as mother-liquor a solution of the compound in excess of the more fusible constituent; if, on the other hand, the constituent present in excess have a higher melting point than the compound, this constituent separates first, the mother-liquor consisting of the more fusible compound, together with more or less of the less fusible constituent dissolved therein.

On the other hand, the following five pairs\* were found to yield only ideal alloys when mixed in proportions inside of the limits under-mentioned:—

Aluminium bismuth.
" cadmium.
" lead.
Bismuth zinc.
Lead zinc.

\* The immiscibility of lead and zinc has long been known, notwithstanding that in certain books of reference they are still described as alloying "in all proportions" (*e.g.*, Morley and Muir's dictionary, article "Lead Alloys," Vol. III., 125). More than 30 years ago their limits of mutual solubility were approximately determined by Matthiessen and v. Bose (Proc. Roy. Soc. XI., 430).

As the temperature rises, the limits between which ideal alloys are formed with these five pairs, become narrowed; precisely the same thing is noticed with analogous ternary and more complex ideal alloys (*vide infra*).

#### LIMITS FOR ALUMINIUM BISMUTH.

At near 870° C. between  $\left\{ \begin{array}{l} \text{Bi} = 99.72 \\ \text{Al} = 0.28 \end{array} \right\}$  and  $\left\{ \begin{array}{l} \text{Bi} = 2.02 \\ \text{Al} = 97.98 \end{array} \right\}$ .

#### LIMITS FOR ALUMINIUM CADMIUM.

At near 750° C. between  $\left\{ \begin{array}{l} \text{Cd} = 99.78 \\ \text{Al} = 0.22 \end{array} \right\}$  and  $\left\{ \begin{array}{l} \text{Cd} = 3.39 \\ \text{Al} = 96.61 \end{array} \right\}$ .

#### Ternary Alloys.

Out of the 84 combinations obtainable by taking the nine metals chiefly experimented with, three at a time, 55 are "real" under all circumstances, but the remaining 29 form "ideal" alloys when mixed in proportions lying outside of certain limits which are conveniently expressible as the boundary of a particular area inside a triangle (*vide infra*).

Theoretically, three different classes of ideal ternary alloys may be distinguished; but as regards the particular metals examined, one of these classes is unrepresented amongst the various combinations thence derivable. The three classes may be distinguished thus: let the three constituents be respectively A, B, and C; then, by taking them two at a time, three pairs are obtainable, A B, A C, and B C. In the first class only one pair, A B, is incapable of mixing in all proportions. In the second class two of the pairs, A B, A C, are incapable of mixing in all proportions. In the third class all three pairs are similarly incapable. The three classes may accordingly be thus tabulated:—

	Immiscible Pairs.	Miscible Pairs.
Class 1	A B	A C, B C
Class 2	A B, A C	B C
Class 3	A B, A C, B C	Nil

The class of which no representative is found amongst the 29 combinations as above, is the third; 23 combinations of the first class, however, and six of the second class, can be formed from the nine original metals, *viz.*:—

#### CLASS I.

A.	B.	C.
Pb	Zn	Either Sn, Ag, Sb, Cu, or Cd (5)
Bi	Zn	Do. do. (5)
Pb	Al	Either Sn, Ag, Sb, or Cu (4)
Bi	Al	Do. do. (4)
Cd	Al	Either Sn, Ag, Sb, Cu, or Zn (5)
		Total 23

#### CLASS II.

A.	B.	C.
Either Zn or Al	Bi	Pb (2)
Either Bi or Pb	Al	Zn (2)
Al	Either Bi or Pb	Cd (2)
		Total 6

[1861]; at the same time the corresponding limits for bismuth and zinc were also approximately deduced. Deville showed many years ago that aluminium and lead were immiscible. As previously stated, the immiscibility of aluminium and bismuth and of aluminium and cadmium was apparently unknown until recently pointed out by the author (Proc. Roy. Soc. 52, 19; this Journal, 1892, 492).

Ternary ideal alloys of the first and second classes, in practice, always divide into pairs of real alloys. A ternary alloy of the third class, however, might, under certain conditions, divide into three real alloys; thus if A could dissolve but little of B or C; B only small quantities of A or C; and C only a little of either A or B, a mixture of A, B, and C, in approximately equal quantities would necessarily divide into three different ternary alloys; *viz.*, A containing a little B and C; B with a little A and C; and C containing a little A and B; so that the fused mass would divide into three layers according to the relative densities of the alloys formed.

Although mixtures of three metals possessing this peculiar feature have not been obtained so far, still it is possible to prepare analogous mixtures of non-metallic fluids. Thus, a mixture of approximately equal volumes of castor oil, petroleum (kerosene), and water, when well shaken up, forms a sort of emulsion, which, on standing for some time, separates into three distinct layers.

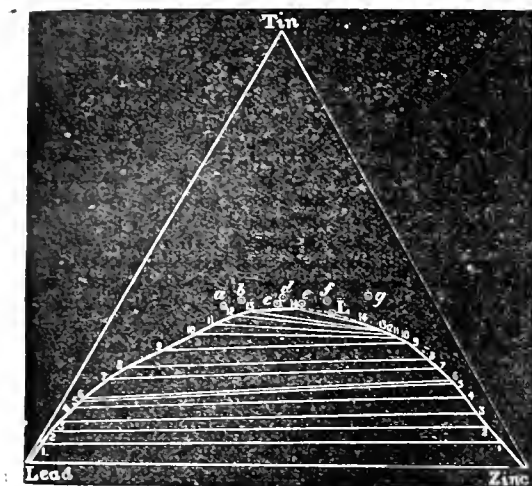
#### Determination of Limits within which Ideal Ternary Alloys of Class I. are formed.

In order to deduce accurately the limits for a given trio of metals, a somewhat lengthy series of experiments is requisite, all made at the same temperature. With alloys of the first class this is best effected by taking suitable quantities of the immiscible metals A and B, and fusing them with gradually-increasing proportions of the third metal C (termed by Sir G. G. Stokes the "solvent" metal). The relative quantities of A and B requisite for any given experiment should be approximately calculated from the results previously obtained, which is readily done at sight when the values are graphically represented as described below; the quantities should be such as to develop approximately equal volumes of the two alloys produced, since experience indicates that if one alloy be formed in very much larger quantity than the other, abnormal results are more likely to be produced. By-and-by the result is arrived at that the limit within which an ideal alloy is produced, is passed, so that a real alloy results, the two ends of the compound ingot formed being of practically identical composition; but before this is attained, pairs of "conjugate" alloys are produced with each mixture employed. To represent these results graphically, Sir G. G. Stokes has suggested the following method:—A triangle is drawn to scale (preferably equilateral), and the centre of gravity deduced (by a simple process of plotting) of three weights placed respectively at the three corners of the triangle, and in the same ratios as the percentages of the three constituents of the ternary alloy to be delineated. Conveniently the solvent metal is supposed to be at the apex of the triangle, and the other two at the respective ends of the base. This point then represents the alloy in question; so that, by finding the two points representing the two real alloys into which a given ideal alloy divides, and joining them by a line ("tie-line" or "tie"), a visible representation is given of the mode of division of the ideal alloy employed.\* A series of alloys with gradually-increasing proportions of "solvent" will then give a series of tie-lines uniting the pairs of conjugate points respectively obtained from each mixture. By drawing a line through all the points thus deduced for the heavier alloys, and another through those similarly obtained for the lighter ones, two branches or portions of a continuous curve ("critical curve") are deduced, the gap between the ends being the smaller the more nearly the series of mixtures employed approaches to a particular limiting composition, indicated on the diagram by a point towards which all the ties continually approach, and at which they vanish. The exact position of this "limiting point" is calculable with a considerable degree of approximate accuracy from the relative positions of the various pairs of conjugate points, by one or other (or both) of two methods ingeniously devised by Sir G. G. Stokes for the purpose.†

\* The ideal alloy itself is necessarily represented by some point situated in the tie-line between its ends.

† Proceedings Royal Society, 49, 1890—1891, 174—178.

The annexed figure, for example, illustrates the critical curve deduced by means of 14 tie-lines (each the result of several concordant experiments averaged) with mixtures of



lead, tin, and zinc at near  $650^{\circ}$  C. The point *L* is the "limiting point," calculated as above; *a*, *b*, *c*, *d*, *e*, *f*, and *g* represent seven different real alloys (actually prepared and found to be such) all lying slightly outside the space enclosed by the critical curve and part of the base line, forming approximately a segment of a circle or of a parabola. Any alloy represented by a point lying *inside* this space is an ideal alloy; whilst, if the point representing an alloy lie *outside* (like *a*, *b*, *c*, &c.), it is a real alloy. In this particular case the configuration of the tie-lines is remarkable: the lower ties slope downwards to the left (lead side), showing that tin predominates in the heavier alloy, as compared with the lighter one; but the upper ties slope in the opposite direction, indicating that the opposite is the case when the original mixture contains larger proportions of tin. The upper ties converge towards a sort of central point; the middle portion of that part of the right-hand side of the critical curve where this is manifest corresponds with a mixture of metals containing tin and zinc close to the ratio represented by  $\text{SnZn}_4$ , suggesting that the tendency towards the fraction of the definite atomic compound thus indicated is the cause of the convergence. The tie-line exhibiting the maximum amount of slope (No. 5) represents a point on the left-hand branch of the curve, where the tin and lead are approximately in the ratio  $\text{SnPb}_3$ , suggesting the possibility of the existence of this definite atomic compound being the cause of the direction of slope being different for the lower ties from that found for the upper ones, a notion strengthened by the facts that a precisely similar double-sloping configuration of ties is observed with lead-tin-aluminium mixtures, the maximum slope of the lower ties also corresponding with  $\text{SnPb}_3$ , whereas no double-sloping sets of ties are observed with any alloys not containing simultaneously lead and tin. In similar fashion the systems of tie-lines and the critical curves thence deduced for the other 22 ternary alloys of the first class have been more or less minutely determined and delineated.\* From these experiments, as far as at present completed, the following general conclusions may be drawn as regards alloys of Class I. :—

1. With each ternary combination of metals the position of the critical curve varies with the temperature in such fashion that the curve for the higher temperature lies *inside* of that for the lower temperature. Towards the base of the curve (i.e., for those mixtures containing smaller proportions of "solvent" metal) the effect of variation of temperature is but slightly marked; but nearer to the limiting point (i.e., for mixtures containing quantities of "solvent" approaching the maximum possible consistent

with the alloy remaining ideal) the effect is much greater; in some cases a comparatively small variation in temperature makes all the difference whether a given mixture of metals forms an ideal alloy dividing into two real alloys exhibiting a considerable amount of difference in composition, or produces a real alloy not dividing at all.\*

2. The position of the limiting point corresponds with a ratio between the two immiscible metals which is not constant, but varies widely with the nature of the solvent metal.

3. In most cases the contour of the critical curve exhibits no marked sinuosity or irregularity; but in a few instances it shows a marked bulge outwards or notch inwards, which reaches its maximum development when the ratio between the solvent metal and one of the immiscible metals approaches towards that indicating some definite atomic compound. The atomic compounds  $\text{AgZn}_5$ ,  $\text{Ag}_4\text{Zn}_5$ ,  $\text{Bi}_2\text{Sb}_3$ , and  $\text{CuZn}$  are thus pretty clearly defined by bulges and notches.

4. In some few cases, although no irregularity of outline of the critical curve is noticeable, yet the configuration of the tie-lines exhibits some peculiarity, leading to the idea that an atomic compound of some of the metals, which affects the relative distribution of the metals in the two conjugate alloys, is formed: e.g., the above-described double direction of slope with  $\text{ZnPbSn}$  and  $\text{AlPbSn}$  alloys suggesting the compound  $\text{SnPb}_3$ , and the convergence towards some central point of a number of tie-lines with  $\text{ZnPbSn}$  alloys, suggesting the compound  $\text{SnZn}_4$ . The existence of the compound  $\text{Pb}_3\text{Cd}$  is clearly indicated by a convergence of this kind. In the majority of cases, however, no change in direction of slope of tie lines is noticeable throughout the entire series, all sloping either to the right or to the left. No fixed law regulating the direction of slope has been arrived at: with silver or copper as solvent, when the results are plotted, the heavier of the two immiscible metals being towards the left, the ties invariably slope downwards to the left (i.e., the heavier alloy formed contains less solvent than the lighter), whether the immiscible pair be  $\text{PbZn}$ ,  $\text{BiZn}$ ,  $\text{PbAl}$ ,  $\text{BiAl}$ , or  $\text{CdAl}$ . With tin as solvent, on the other hand, the ties invariably slope to the right, except those obtained with smaller tin proportions in the case of  $\text{PbZnSn}$  and  $\text{PbAlSn}$  alloys, when the slope is towards the left, probably on account of the effect of the formation of a definite compound  $\text{Pb}_3\text{Sn}$ , as above described. With antimony as solvent, however, the ties all slope to the left with the certain immiscible pairs ( $\text{PbZn}$ ,  $\text{PbAl}$ ,  $\text{BiAl}$ ), but to the right with others ( $\text{BiZn}$ ); similarly, with cadmium as solvent, the ties all slope to the left with the immiscible pair  $\text{PbZn}$ , and to the right with the pair  $\text{BiZn}$ . With zinc as solvent in the alloys of  $\text{CdAlZn}$ , the ties slope to the left. It would thence seem that whilst the nature of the solvent metal obviously influences the direction of slope, and whilst the ties slope to the left in the larger number of cases out of the 23 possible ones, it cannot be deduced that one given solvent always gives a slope to the left, and another one to the right, and so on.

5. As regards the dimensions of the segment-like areas within which real alloys are not formed in different cases, it is noticeable that on comparing the critical curve obtained with a given trio of metals, of which lead is one, with that obtained with the corresponding trio, where bismuth is used instead of lead, the latter always lies *inside* the former. In similar fashion, when zinc is displaced in a given trio by aluminium, the curve obtained with the latter metal lies *outside* that obtained with the former. Similar relationships also apply (but not invariably) to certain pairs of solvent metals, the temperature and the two immiscible metals being the same; thus, whether these two metals be lead and zinc, lead and aluminium, bismuth and zinc, bismuth and aluminium, or cadmium and aluminium, the curve obtained with silver as solvent metal always lies *outside* that formed with

\* Fluids other than melted metals exhibiting the same characters as to mutual miscibility or otherwise, behave in precisely the same way: thus a mixture of water, chloroform, and acetic acid behaves in a fashion exactly parallel with that observed with a molten mixture of zinc, lead, and tin. Near the limiting point the sensitiveness to temperature variation is very great.—Proc. Roy. Soc. 50, Part V.

copper; and apparently the curve with antimony always lies inside that observed with tin. On the other hand, although the curve with tin as solvent lies inside that with Ag (and a *fortiori* inside that with Cu) in the four cases where the immiscible pairs are respectively PbZn, BiZn, PbAl, and BiAl, yet this is not the case when the immiscible pair is CdAl; the Sn curve then lying outside both the Ag curve and the Cu curve.

### *Ternary Ideal Alloys of the Second Class.*

In order to depict graphically the results arrived at by the analysis of the real alloys into which ideal alloys of the second class divide themselves, the same triangular system may be conveniently adopted, with the difference that the two miscible metals are placed at the ends of the base line, and the third metal (not miscible completely with either of the others) at the apex of the triangle. The limits within which ideal alloys only are formed, are traced out by plotting the conjugate points and connecting tie-lines as before, the result in all cases being a space of very different shape from that deduced with alloys of the first class. Instead of this space being bounded by part of the base line and a curve approximating more or less to the arc of a circle or part of a parabola, it is a quadrilateral, two of the sides of which are parts of the sides of the triangle, whilst the other two are curved lines running more or less horizontally, and consequently cutting off from the triangle a three-sided portion at the top, and a four-sided part at the bottom. Mixtures corresponding with points lying in either of the upper or lower areas are real alloys, and do not divide into two on standing molten; whilst mixtures corresponding with points lying inside the intervening quadrilateral figure are ideal alloys, and divide into two real ones, one situated on the one line and the other on the other line.

### *Quinary Alloys.*

Of the 126 quinary combinations obtainable from the nine metals employed (95 of which are capable of forming ideal alloys), obviously a yet larger number of classes and subdivisions may be *a priori* formulated, many of which have representatives amongst the combinations deducible from the nine metals used: thus the alloys—

Zn, Pb, Cd, Sn, Ag

Al, Cd, Sn, Ag, Cu

contain *one* ideal binary pair, A B; whilst the following—

Zn, Pb, Bi, Sn, Ag

Al, Pb, Cd, Ag, Cu

contain *two* such pairs, A B, A C: and so on.

### *Sexenary Alloys.*

Of the 84 sexenary alloys obtainable, only nine are real; *viz.*, the seven formed by taking each six out of the series—

Pb, Bi, Sn, Ag, Cu, Sb, Cd

(*i.e.*, by omitting each one in turn); and the two following:—

Al, Zn, Sn, Ag, Cu, Sb

Zn, Cd, Sn, Ag, Cu, Sb

### *Septenary, Octenary, and Nonary Alloys.*

Of the 36 septenary combinations deducible, only one is real; *viz.*:—

Pb, Bi, Sn, Ag, Cu, Cd, Sb.

None of the nine combinations obtained by taking the metals eight at a time, is capable of giving a real alloy, excepting when the constituents are mixed outside of certain limiting proportions; and the same remark is true of the mixture obtained by putting together all nine metals; thus a mixture, of which the great bulk consists of any one of the five ideal binary alloys PbZn, PbAl, BiZn, BiAl, CdAl, together with small admixtures of the

remaining metals, will divide into two different alloys, consisting, for example, of—

1. Chiefly Pb, with small quantities of the other eight metals.

2. Chiefly Zn, with small quantities of the other eight metals.

The CHAIRMAN said that the paper just read had an interest far exceeding the interest of the subject, it being the last work of their dear friend Dr. Alder Wright. Nearly every member of the Society knew him, and to know him was to admire him and revere his memory. The paper now before them was an illustration of the thorough manner in which Dr. Wright did all his work. It was monumental in character, covering the whole history of the subject. Probably few members would wish to discuss the matter that night, but he was happy to say that there was present with them a fellow-student of Dr. Wright, one who had known him from his boyhood, namely, their worthy President, and he would ask him to say a few words, if not on the paper itself, at any rate on the author.

The PRESIDENT: I do not propose, in the very few words that I shall say to the meeting in response, sir, to your invitation, to utter anything in the way of contribution to a discussion on the matter of Dr. Wright's last paper. Indeed, I am quite incapable of contributing anything which would be worthy of being listened to on that subject. But I should not like this opportunity to pass without saying something on the merits of a man whom I have known intimately for 30 years, and who is now no longer to come amongst us. Dr. Wright, his friend Mr. Watson Smith, who has just read to us his last paper, and I, began our chemical careers together, working shoulder to shoulder in that laboratory in the rear of Richard Cobden's old house in Quay Street, Manchester, which was the place in which the now palatially-housed Owens College had its humble beginning. As you have said, sir, to know Wright was to admire him, but I will say more: to know Wright well was to love him. For beneath the surface-aspect of the man—beneath all his eccentricities and peculiarities—there was a great substratum of genuine worth and high personal character. There was a certain large-heartedness about the man without the least trace of malice or uncharitableness. He was often misunderstood; people occasionally said harsh things of him; but no unkind word of retaliation—no word which could be misconstrued to his quondam opponents' demerit—was ever uttered by him. The very directness and straightforwardness of his speech and manner was indeed the keynote to his character. There is in the paper we have just heard the melancholy interest which springs from the circumstance that it is the last contribution which this or any other Society will ever receive from him. That of itself gives the paper a peculiar importance to us on this the first night of a new year in the existence of our Society—a night upon which we might have reasonably hoped to see Wright once more among us, and to hear his voice again: for, as the minutes just read to you show, it was actually raised among you at your very last meeting. Indeed, we should have had from him the very paper which our editor has just read, and its reading would have been accompanied by a wealth of illustration and by an amount of exposition which were characteristic of him. A man of clear ideas, with a very considerable vocabulary, and power and felicity of expression, Wright was, indeed, one of the very best expositors I have ever heard. This charm we have missed to-night—the charm which comes from the personal interest which the writer would himself have thrown into his communication. There is an aspect of the paper which struck me forcibly, as no doubt it did many here—namely, the indication it affords of the extraordinary versatility of its author. That versatility, that remarkable receptivity and power of adaptation to all sorts of fresh ideas, was characteristic of Wright from his student days. The ease and rapidity with which he accomplished his collegetasks always appeared marvellous to his fellow-students. It was so not only in chemistry, but he excelled



equally in every other department of knowledge which he took up as a student: physics, mathematics, classics—even in such subjects as Hebrew and the Greek Testament—in all he gained distinction, as the old prize lists at Owens College testify. His published papers range literally over every department of chemistry, pure and applied. Now he treats of the constitution of whole groups of the alkaloids, and was, as you know, concerned in discovering one of the most important and valuable of the artificial members of the series—apomorphine. Now he deals with a most difficult branch of organic chemistry—the nature of the essential oils, and the very minute differences to be observed in the physical characters of the isomeric terpenes. Then he passes to various sections of inorganic chemistry, and from them to industrial applications of both organic and inorganic chemistry. He was as much at home in discoursing upon the intricacies of the manufacture of alkali and sulphuric acid as in discussing the characters and constitution of the opium alkaloids. In physical chemistry, too, he finds a field for his energy. The existence of hydrogenium is established, and Wright is concerned to determine certain of its physical properties. Indeed, his first published paper was on a section of physical chemistry—the action of light on silver salts, and in re-reading that paper a few days ago I was greatly struck with its power and promise—the precision of its statements, its orderly arrangement, and literary merit. He did not think that the whole of chemistry was concerned with the study of carbon compounds—he was not merely a benzological man. In this his last paper we have one more instance of the catholicity of his tastes and the range of his acquirements. For it is to my knowledge the condensed expression of many years of thought and a great amount of experimental work. I could say much more about Wright's personal character—about his worth as a man and his staunchness as a friend. One indication of his qualities you have gleaned from the circumstances which led up to the reading of this paper. He gave his word to our Secretary that this paper should be prepared; and under the most trying conditions, while suffering from pain and great physical prostration, he fulfilled his promise. This trustworthiness was eminently characteristic of him, and was thoroughly in accord with my lifelong experience of his character and qualities. Those who were best acquainted with him, and knew the circumstances under which much of his work was done, regarded the amount and quality of it as nothing short of marvellous, and the only solution I can offer is that he found in work itself solace and comfort under the physical pain and consequent mental depression from which he had for many years suffered.

The CHAIRMAN suggested that under the circumstances it would not be fitting to discuss the paper itself, but he would suggest that the members should confine their remarks to expressing their feelings concerning the author.

Mr. THOMAS TYLER, while feeling unable to add materially to the eloquent tribute to Dr. Wright's character and work which had fallen from the lips of the President, yet wished, as one of the oldest officials of the London Section, to add his testimony to the worth, integrity, and high character of the friend they had just lost. The qualities which the President had referred to were by no means confined to important matters, but were equally noticeable in the smallest details with which Dr. Wright was concerned. He had had many proofs of this in the course of his long acquaintance with Dr. Wright, and as a consequence he held his friend's judgment in such esteem that he could confidently accept it, even if, as sometimes happened, it differed from his own.

Mr. WATSON SMITH wished also to express his high opinion of Dr. Alder Wright as a friend and as a man, apart from his conception of him as a scientist. As the President had said, they were trained together at Owens College, but his acquaintance with Wright went further back than that—almost even to his infancy. Throughout their lives Dr. Wright had been a most sincere friend, and he never could forget the help and kindness which Dr. Wright had extended to him when he came from Manchester to take up his duties

in London, which went even to the extent of placing his house at the disposal of himself and wife, until they found a house of their own. He would also like to speak of the tender way in which Dr. Wright had spared his wife the knowledge of the terrible complaint which was the cause of his death. It appeared from the wording of the paper that he never expected to be alive to read it, for it was carefully written (as it now appears in the Journal) in the third person throughout.

## NOTE ON OXIDISED LINSEED OIL.

BY WALTER F. REID, F.I.C., F.C.S.

NEARLY the whole of the linseed oil which is manufactured in this country or imported into it is finally converted into the oxidised form; in fact, the large and growing consumption of this material is mainly due to the facility with which it absorbs oxygen from the air, and, in so doing, is changed from a liquid into a solid body. The paint which embellishes or disfigures our dwellings, the floorcloth or linoleum which covers our floors, and the pictures which adorn our walls—all owe whatever stability and cohesion they may possess to this oxidised modification of linseed oil.

In some industries, such as the manufacture of linoleum, and, to a small extent, in that of india-rubber, the linseed oil is first oxidised and the solidified product is afterwards mixed with other substances. In the majority of cases, however, the oil is used in its liquid state, as in paints and varnishes, and solidifies when exposed in a thin film to the action of the air.

Where the oil is oxidised as a preliminary process, two methods are employed on the large scale. In the first and oldest the oil is heated to temperatures varying from 350° F. to 500° F., with a small quantity of litharge or red lead. The operation is sometimes accelerated by blowing air through the oil, and samples are taken from time to time to test the progress of the oxidation. If a small portion of the oil becomes "stringy" when cooled and tested the oxidation has reached a stage which is sufficient in some industries; but in others, such as the manufacture of printer's ink, the operation is pushed a little further and the surface of the oil is sometimes ignited. The product is a dark-coloured, viscid mass of disagreeable odour and sufficiently fluid to take the shape of any vessel into which it is placed.

In the more modern process of oxidising oil for the manufacture of linoleum, the linseed oil is first boiled with the usual quantity of litharge, or red lead, or a mixture of both, and the boiled oil is allowed to flow over sheets of a light cotton fabric technically known as "serim," which are suspended vertically in sheds heated to 80° F. to 100° F. Every time the "serim" is flooded by the oil, a film of oxidised oil is formed on each side, varying in thickness from  $\frac{1}{16}$ th to  $\frac{1}{8}$ th of an inch. When these accumulated films, now termed a "skin," reach a thickness of about  $\frac{1}{2}$  inch they are cut down and ground between rollers preparatory to the next process. In this transverse section through a "skin," you may be able to detect some of the daily films, and you will observe that the colour deepens considerably towards the centre. Oxidised oil made in this manner forms, as you will see by the sample shown, an amber-coloured mass of considerable elasticity, but very deficient in tensile strength. It is heavier than water, while the original linseed oil is, as you are aware, much lighter. In this process of oxidation there is an increase of about 10 per cent. in weight; but the actual absorption of oxygen is greater because considerable quantities of volatile substances, including much acrolein, are given off at the same time. The fibre of the cotton "serim" has been much weakened, for linseed oil during oxidation has a powerful action not only upon textile fabrics, but also upon wood and even iron. The preliminary oxidation of the oil in this state is by no means complete. If ground between rollers and placed in a heap the solidified

oil absorbs oxygen from the air with such avidity that ignition of the heap sometimes takes place. For making linoleum the ground oxidised oil is heated with kauri gum and resin, forming an elastic, sticky cement which, if dissolved in a suitable solvent, would form a varnish; it is, in fact, almost identical with the film left by a kauri varnish after evaporation of the solvent and oxidation of the oil contained in it. You will notice that this linoleum cement is somewhat darker in colour than the oxidised oil; but seen in section by transmitted light it appears much lighter than in bulk. It is tougher than the solidified oil which forms its main ingredient, although not quite so elastic.

The oxidised oil which you have seen in bulk is practically identical with the substance produced by the spontaneous oxidation of linseed oil when mixed with pigments to form paints. After observing the facts which I am about to bring before you, I carried out a number of experiments, extending over many years, with films of both raw and boiled linseed oil alone and also mixed with various pigments. The results showed that the product obtained was almost identical with that which you see here.

Both in the case of paints and varnishes this solid, elastic substance has hitherto been regarded as the ultimate product of the oxidation of linseed oil when exposed to the air. Mulder, whose researches have thrown much light upon drying oils, and who termed it "linoxyn," says of it:—

"It is the important basis of all linseed oil paints and the product of the drying of linseed oil."

Schaeffler repeats this, and adds that linoxyn is probably the product of most drying oils. The same opinion is generally held with regard to varnishes containing linseed oil, and is well expressed by a technical writer in the following words:—

"After an article has been varnished the process of solidification, to which we unphilosophically apply the term drying, is carried on by the absorption of oxygen gas from the surrounding atmosphere, and when the varnish is thoroughly hardened it has absorbed all the oxygen it is capable of, and the result is a beautiful, transparent glaze." Von Pettenkofer, again, who made many interesting researches on paints and varnishes and published a work on the restoration and preservation of pictures, assumes that the elastic film produced by the oxidation of linseed oil is permanent in its chemical properties.

If, however, we examine carefully the behaviour of linseed oil for a period longer than that over which the investigations of these and other observers extended, we find that the properties of the oxidised oil are profoundly modified in the course of time; so profoundly, in fact, as almost to destroy the foundation upon which our theories on this subject have been based. If a surface of pure oxidised linseed oil be exposed to the air for about two years it becomes sticky and softens somewhat. In about three years, if the surface be vertical, part of it will run down as a semi-fluid mass, and in the course of five years a layer of oxidised oil 1 centimetre thick will be completely liquefied. You will see here the product which has resulted from the gradual oxidation of a "skin" of oxidised linseed oil which was, in 1878, identical in all its properties with the fresh one which I have just shown you. It is an extremely viscid, semi-fluid mass of a much darker colour than the oxidised oil from which it is derived. Raw linseed oil produces a similar substance; but both the preliminary oxidation and subsequent liquefaction require a longer period of time than the boiled oil. The properties of this substance, which we may term super-oxidised linseed oil, differ very materially from the initial linoxyn, and possess great interest in connection with nearly every industrial application of linseed oil. By studying it carefully we may find an explanation of many phenomena which have hitherto remained obscure. Like the solid oxidised oil it is heavier than water; but it has also become to a considerable extent soluble in that liquid. In this property may be found the explanation of the necessity for the renewal, at comparatively short intervals, of all oil-paint which is exposed to the weather, while similar paint protected from the rain will last very much longer.

Linoxyn is but slightly acted upon by alcohol, while the product of its subsequent oxidation is almost completely

soluble in that solvent. Von Pettenkofer invented a process for the restoration of oil paintings by exposing them to the vapour of alcohol at ordinary temperatures; and in his work on the subject he ascribes the action of the alcohol to the restoration of the molecular contact of the pigments. He was unaware that alcohol was an almost perfect solvent for the medium by which these pigments were fixed. In this, as in other directions, there is a remarkable resemblance between linseed oil and india-rubber. The first preparation of the india-rubber by warming a thin film of the liquid from which it is derived, much resembles the oxidation of the oil. Then follows what can only be regarded as an intermediate stage of solidity and elasticity, after which comes a final, or perhaps we had better say a further, stage of oxidation. During this stage the india-rubber also becomes soluble in alcohol. There are further analogies, such as partial solubility in water, to dwell upon which would exceed the limits of this note.

In the facts which I have placed before you may be found the explanation of many phenomena which will be familiar to you. In old prints and books, for instance, you will frequently have noticed a brown stain on the paper adjoining the print, or on pages opposite dark woodcuts. From such stained paper I have extracted a substance identical with this super-oxidised linseed oil. As the oil contained in the printer's ink liquifies, it is absorbed by and spreads through the paper, producing those stains which are too familiar to lovers of old books.

Again, some of you may have been vexed at the too rapid deterioration of articles made of patent leather, especially boots. The glossy surface of such leather is composed of a mixture of oxidised linseed oil and Prussian blue, and this will explain why the surface becomes sticky or cracks long before the leather itself shows signs of wear. So well known is this defect that dealers of repute refuse to hold themselves responsible for the wear of patent-leather goods. American cloth, which is a cotton fabric dressed with a composition similar to that used on patent leather, acquires a disagreeable adhesiveness when packed away for a few months in contact with other articles.

One of the most useful articles in the chemist's laboratory is india-rubber tubing. Many of us, no doubt, have met with samples of tubing which, while new, were apparently of excellent quality, soft and supple, although, perhaps, slightly deficient in elasticity. In about a year's time such tubing, should it contain solidified oil, will be unfit for use, while pure india-rubber will show hardly any signs of decay. The india-rubber itself will, of course, eventually become unserviceable; in fact, all elastic substances with which I am acquainted, except one, are very prone to decay. That one is the curious mineral elaterite, of which I have brought a specimen with me, in the hope that it may be of some interest. This piece is from Derbyshire, and has been exposed in the open air for 13 years, without the least deterioration. You will observe that it possesses considerable elasticity under compression, although its tensile strength is not very great.

It is found in the carboniferous limestone impregnated with a dark brown mineral oil of the consistence of gas tar. The other substances which I have noticed associated with it are iron pyrites, galena, and the sulphates of iron and lead. I believe that many years ago attempts were made to distil a mineral oil from this substance, but the progress of industrial chemistry has since been such that the problem has been reversed. A process for converting the heavier mineral oils into elaterite would enrich us with a substance which might find many useful technical applications.

Those of you who take pleasure in visiting a picture gallery—and who does not?—must have remarked with regret the condition of so many of the oil paintings. The colours of the pictures of comparatively modern date are far inferior to those of the frescoes on the walls of Pompeii or in the chambers of the Egyptian tombs, and it would be an insult to the memory of the artists to suppose that they ever laid on their colours in the sombre shades which we now see. It has been supposed that some special virtue lay in the pigments used in Greece and Egypt; but analytical chemistry, together with the records which have been handed



down to us, enables us to reproduce with accuracy almost any pigment known to the ancients. Aniline colours are of very recent origin, and we must look to the medium used for an explanation of the deterioration which has undoubtedly taken place. Two main causes have co-operated in producing the alterations which are but too evident. One is the liquefaction and darkening of the medium upon which the artist has relied to cement the particles of pigment to each other and to the canvas. The substance produced being extremely viscid retains permanently any dust or dirt with which it may come into contact. The other cause of alteration in an oil painting is traceable to a more purely chemical action. Linosyn itself is an inert substance, difficult to dissolve and to bring into combination with other substances. The product of its further oxidation, however, has a strongly acid reaction and forms solid compounds with most pigments of a basic nature. With white lead, for instance, a solid compound is produced which, though very friable and brittle, is quite solid and prevents the liquefaction of any point of which that pigment forms a substantial part. Those who have attentively examined the darker portions of pictures will have noticed that cracks and flaws are there much more frequent than in the lighter portions of the same pictures. Most of the dark-coloured pigments are but slightly basic in their nature, and they require a very much larger proportion of oil than the lighter colours. While white lead gives a good paint with but one eighth of its weight of linseed oil, ivory black cannot be used with less than its own weight of oil, and lampblack requires even a greater proportion. The larger the quantity of oil present, therefore, the less basic is the substance which alone is available for maintaining it in a solid condition. When lamp-black, or any finely divided variety of carbon, is used as a pigment, the occluded oxygen in its pores has a most injurious effect on the consistence of the paint, and it is well known that such black paints have a great tendency to remain sticky or "tacky." I have known a manufacturer to prepare a special quick-drying oil for black paint with the inevitable result that the paint was worse than before. This defect of black paint has frequently been attributed to the pigment alone. John Smith, who wrote a curious treatise on the art of painting about two centuries ago, says:

"In the substance of the colour is contained a certain greasy fatness that is an enemy to drying, to remedy which, burning in the fire till it be red hot and cease to smoke, will consume that fatness, and then it will dry much sooner."

This treatment would no doubt improve the drying qualities of the paint because the oxygen contained in the pores of the carbon would be driven off at a red heat.

The surface to which oil paint is applied must necessarily exercise an important influence upon the conservation of the film. If it contain a basic substance or a carbonate the effects of the liquefaction of the linosyn may be partially neutralised. Miniatures painted in oil upon ivory no doubt owe their generally good state of preservation to the combination of some of the mineral constituents of the ivory with the organic acids as these are generated during the oxidation of the oil.

Many other similar instances might be given; but I think after what has been said you will agree with me that it is a matter for deep regret that most of the talented artists of our generation are embodying the creations of their genius in a material which is anything but permanent, however pleasing its appearance may be for the moment.

#### DISCUSSION.

The PRESIDENT said he gathered from the paper that Mr. Reid considered the process of drying or coagulating india-rubber to be similar to the process of oxidising linseed oil.

Mr. REID replied that they found no oxygen in the finished rubber, but there was a similarity in the conditions under which it solidified.

The PRESIDENT, continuing, said: of course all dried rubber contained a certain amount of oxygen. He might

say that many years ago he was in Grão Para, and had the opportunity of seeing the process of making india-rubber as carried on by the Indians and half-breeds who tapped the Seringa. That the process was not altogether one of oxidation was obvious from the fact that the finished rubber, though containing a certain amount of oxygen, was not equal in weight to the juice that had gone to make it. It was clear therefore that a considerable amount of moisture was given off in the process of coagulation. With respect to the remarkable capacity of oil for absorbing oxygen, he might mention a curious experience of his own. Some years ago he was consulted by a blanket manufacturer at Huddersfield as to the extraordinary way in which the blankets, after being drawn from the bleaching house, where they were treated by the fumes from burning sulphur, fell here and there into holes. He went carefully over the whole process, but for a time could get no clue to the mystery. Eventually, however, it was discovered that while the blankets were in the centrifugal machines after removal from the fulling mill, the engineers, in oiling the bearings, would sometimes inadvertently spatter some of the blankets with drops of oil, and when these blankets were afterwards exposed to the sulphur fumes the oil brought about the rapid oxidation of the sulphurous acid, and so effected the tendering of the fabric.

Mr. IVES observed, with reference to the extraordinary way in which the colours used in Egyptian decorations retained their brilliancy, that the vehicle employed was a wax, not an oil, and consequently did not oxidise so readily.

Mr. T. CHRISTY thought that the tendency of the paper was to convince one that the linseed oil trade was no longer one of our greatest industries, and that, in fact, it was doomed. There was another reason for coming to that conclusion, namely, that very good oil, and varnish too, could now be obtained from petroleum; so good in quality that the use of linseed oil was likely to be superseded. A case had recently come under his notice in which a city restaurant had been completely painted and varnished by means of these products, with an entire absence of the disagreeable smell which always accompanied the use of oil paints. He hoped during the session to bring these products before the Society. Curiously enough, Mr. Reid had brought to their notice that night an earth which might be utilised for their manufacture, as well as for rubber, and he had no doubt many members would turn their attention to this new earth.

The CHAIRMAN enquired whether the petroleum varnish required the admixture of a gum.

Mr. CHRISTY replied that he believed it did not.

Mr. C. G. CRESSWELL observed that a soap was usually supposed to be formed by the action of white lead on linseed oil, and asked Mr. Christy how he would produce such a soap with the aid of petroleum. It was all very well to talk of new varnishes, but the merit of the paints used by our fathers lay chiefly in the fact that they did really contain white lead, whereas the paint used by the speculative builder of to-day had so little lead in it that it could be easily rubbed off with a clothes brush, and was therefore utterly unsatisfactory as a protective covering for wood. The blue pigment referred to as having been discovered in the tombs of Egypt was described in a memoir to the Académie des Sciences, and there was an abstract of it in the Society's Journal (Feb. 1889, 142). The pigment was metallurgical in character.

Mr. CHRISTY observed that he had seen specimens of soap actually made from the petroleum products he had referred to, and works were now being erected for its manufacture. He had tried it in his greenhouse, and found it the best insecticide he had ever employed.

Mr. W. J. HORN asked whether Mr. Reid had experimented with poppy oil in the same way as with linseed oil as to its oxidising qualities.

(Continued on page 1024.)



Mr. W. F. REID, in reply, said that the non-increase in weight of india-rubber sap by oxidation, was due to the fact that the juice contained only a small portion of the active substance, just as linseed only contained a certain percentage of linseed oil; but if one could obtain the pure substance contained in juice in its liquid state, and treat it by heating, one could find out whether there was a sensible increase or not. He hardly thought there would be, seeing that the percentage of oxygen in india-rubber was very minute. In fact, it was not a process of oxidation, but it might be one of polymerisation. If linseed oil were heated in a closed vessel, it solidified, shewing that internal molecular changes took place which were not due to oxidation. Possibly, it was so with india-rubber. Mr. Ives had asked if the Egyptians did not use wax as a vehicle. He believed they did not, but the Greeks certainly did, and had a peculiar way of preparing it. They used punie wax, mixed with volatile oils as a medium. The wax was kneaded with sea water, and exposed to the light; and modern experiments proved that the punie wax so treated possessed qualities quite different from those of wax bleached in the sun without the use of salt water. He could not agree with Mr. Christy that linseed oil was doomed. It had many and increasing uses, and no doubt its defects would be neutralised by the methods of using it; as in the case of linoleum, in which a large percentage of ground cork was used, and this absorbed the liquid oxidation products of the linseed oil as they were generated. With regard to the petroleum varnish referred to by Mr. Christy, he would like to know if its basis was similar to the elaterite, of which he had shown specimens; if so, it might be a permanent varnish.

Mr. CHRISTY could only say that it was a petroleum product, and that Mr. Reid's specimen closely resembled it.

Mr. REID, continuing, said: with regard to the blue pigment which had been referred to, that he had derived his information from the article cited by Mr. Cresswell, which proved the value of the Society's Journal in gathering notes together that might otherwise be lost. As to petroleum, he hardly thought it necessary to turn it into a soap, as it was an excellent cleansing agent naturally. He had tried experiments on a small scale, not only with poppy oil, but with nut oil also. Both oxidised much more slowly than linseed, but both eventually liquefied, and Schaedler was quite right in saying that all drying oils possessed the same property, though they contained less linoxyn.

Meeting held Wednesday, 7th November 1894.

## CHAIRMAN'S ADDRESS.

### THE INVASION OF THE INVENTOR.

BY EUSTACE CAREY.

As I spoke to you last year of "the multitude of inventions, mostly bad ones, which beset us on every side, and of the trained skill and knowledge which is required to sift the good from the bad, to winnow out the few grains of wheat from the almost interminable mountain of chaff," I thought I might do worse than take the inventor, his methods, and his fair remuneration, for my subject, as it is one which is interesting to all of us.

The genus inventor may be divided into three or four well-marked species or groups.

The practical manufacturer, who, with command of capital, experience and the assistance of experts, works out an invention to a finish, ending in the adoption or definite abandonment of a new process, belongs to a class of inventors, who, when they possess capacity and perseverance, cannot fail to be of great service to the community.

But even celebrated men of this class have not been able always to restrain themselves from the weakness of inventors of a commoner type, they have, parenthetically as it were, run to the Patent Office to secure the protection of immature notions and half-fledged ideas which have occurred to them during their work on the main and particular invention which they have had in hand; in fact the very best of inventors, however practical and experienced they may be, seem to be sometimes victims of a disease which we may call inventor's mania, the spirit of invention invades them, ideas good, bad, and indifferent seize upon them, and writing specifications and paying patent fees, becomes an almost daily excitement, without which life seems to them flat and uninteresting.

But as to those inventors whom we may place in the front rank, what is their method? They begin like everybody else, by having an idea; a new method of making an old material occurs to them, or a new use for an old product. Whatever their invention may be their first step is probably to try a laboratory experiment—the laboratory experiment succeeds—instantly visions of the potentialities of the new process fill their thoughts and glorious castles in the air at once appear to charm and allure them forward. On the other hand there are many processes which work well on the large scale and could not be made to work at all on the small, at all events good working on the small scale would be a matter of great difficulty.

Those manufacturers and capitalists, even who belong to this first and best class of inventors, are not always an unmixed blessing to their partners and the firms to which they belong. They are apt to pursue will-o'-the-wisps and neglect steady attention to what I may call every-day improvements. A manager or managing partner of a works who has his eyes open to perceive the small additions or alterations which may improve or cheapen production and has the openness of mind to accept suggestions from his colleagues and his workmen as to the never ending improvements which can be made in details, is frequently a wiser man than the manufacturer who has a genius for revolutionising everything. An inventive partner often gradually becomes a perfect terror to his friends who have every cause to lament the invasion of that inventor.

Among those who are not engaged in manufacture there are also inventors, some of them men of good chemical knowledge, and some of them men of some technical experience, but who cannot afford themselves to make a large scale experiment or they cannot create a value for invention by the expenditure of sufficient capital, so what do they do? They call in an expert. Their best course

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Dr. Lewis Bailey, University College, Liverpool.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

SESSION 1894-95.

December 5th.—Mr. Eric E. Watson. "The Calculation and Composition of Blast-Furnace Charges, as applied to the Smelting of Lead and Silver Ores."

would be to procure the aid of the best expert of all, the practical manufacturer, but that in their opinion would not pay them sufficiently well. They want a large sum for the invention, and so after they have seen an expert they go to a company promoter. The company promoter's first and immediate business is to get up a prospectus, and one of the essentials to a good prospectus is to have the favourable opinion of a well-known scientific expert, who gives it as his solemn opinion that "from the interesting and successful experiments which he has been permitted to witness, and from a careful examination of the reactions which are quite feasible under proper conditions, and that if the large scale results bear out the experimental trials, which he sees no reason to doubt, then the prospects of the new company are brilliant in the extreme."

But I may say the value of an invention increases as the scale on which it is demonstrated increases. The full value can only really be estimated when the working expenses have been demonstrated for some considerable time, so as to give a correct idea of the wear and tear of the plant. It is proverbial that most inventors estimate the costs of their processes at too low a figure. To make a proper estimate of the cost of a new process is an exceedingly difficult problem.

One great difficulty which presents itself at the outset to the practical manufacturer is that a great many inventors are unwilling to take any further risk of their invention, but demand to be at once remunerated to the full value of their own estimated profit, to be made in the unexpired portion of the 14 years' life of their patent. Now, while it is difficult to fairly guess the possible saving or profit to be gained by an invention, it is still more difficult, well nigh impossible, to capitalise it, to make such a venture, except at a very short purchase, say two or three years, is almost equivalent to gambling if a company be formed to acquire new patents and large amounts are paid in cash.

If, in a manufacture, finality could be reached and a certainty thus arrived at that the patent to be acquired would not be superseded in the course of a few years, such payments to the inventors might be justified, but when we remember that a money-making invention is just the one which is sure to attract the attention of inventors all the world over the payment of a large sum is highly dangerous. Yet most inventors insist on a payment of a certain amount of cash down.

The most prudent are those which are satisfied with a small amount, though the most numerous are those who demand at once a large fortune.

One class of inventors—the dealer in secret processes—is a power in the world, too, since it is a common weakness of human beings to be charmed by the unknown almost in a direct ratio to its approach to the impossible, as witness electric sugar. Whenever a process is to be kept a secret until a purchaser has satisfied himself that it will work we might almost at once dismiss the invention, for if a patentable invention there is no secret necessary, but if not patentable there is no guarantee to the purchaser that the invention may not be worked by others. But without saying more about dishonest inventors we are attacked more than sufficiently by those who are honest and straightforward to lead us in conclusion to consider if any fair, though, perhaps, rough-and-ready method of procedure may be suggested, whereby the reasonable claims of the inventor and of the practical and established manufacturer may be harmonised. I will put out of our reckoning members of our first group of inventors who are capitalists and manufacturers, and who work out their own inventions in their own way and for their own profit, and confine myself to those cases which are more numerous and more troublesome, where the inventor is either an outsider altogether, or at the most an employé of the manufacturer.

It appears to me that when an invention has been practically worked out and fairly put upon a manufacturing basis, a royalty of from 10 per cent. to 20 per cent. of the ascertained saving might in many cases be fairly charged. But certainly it appears to me that half of this royalty is due to the manufacturer who first works out the process on a large scale, the other half may reasonably fall to the

inventor who first gets the idea, and who by preliminary laboratory or small scale experiments shows its theoretical feasibility. I have taken a simple case of an invention where it is clear who is the originator of the idea, and who is the first to work it out practically on the large scale, but if a general support may be given to the foregoing in a comparatively simple and typical case it may not be insurmountably difficult to arrange a fair *modus vivendi* in other cases not so simple, and where the rival claims may vary in one direction or another from those just now described. Let me say, then, by way of suggestion, that a royalty of from one-tenth to one-fifth of the ascertained saving may in many cases be fairly charged, and that this royalty should be divided equally between the man who has the idea and shows by preliminary laboratory experiment and scientific calculation that the idea is worth pursuing, and the man who first demonstrates on the large scale that the new process can be successfully worked. I would name only one addendum, and that is that the works at which the large scale experiments are carried out and brought to a successful issue should enjoy complete immunity from royalty, and I consider that this privilege will in the vast majority of cases be richly deserved. You may ask, "What do you mean by ascertained saving?" and "Why fix the royalty at 10 or 20 per cent., why not any other figure?" I will endeavour very briefly to answer each of these questions.

In ascertaining the saving effected by a new process it is not sufficient to calculate the actual saving in material and wages, but in the case of an established manufacture due allowance must be made for the capital required to replace the old plant by new, and for the risks to be encountered in respect of the validity of the patents connected with the new process. With regard to the amount of royalty I admit at once that 20 per cent. may be an extreme figure; I have put it as the limit beyond which my experience licenses are not often accepted. Let one instance suffice:—The Weldon Manganese recovery process was generally supposed to effect a saving of about 2*l.* per ton on the bleaching powder made by that process; the royalty was at first fixed at 10*s.* per ton, but this was found impracticable under the circumstances, and subsequently 5*s.* per ton was accepted, the pioneer manufacturers obtaining a considerable concession. Here we have a royalty of say 12½ per cent. of the generally admitted saving, but we must remember that this royalty was only obtained after the process had been thoroughly established at more than one large works. I need not multiply instances; each case as it arises will have special points of its own which require adjustment, which adjustment will be the result of bargain; but the proposition which I wish especially to submit for your consideration is not the actual royalty charged which will vary and be the result of bargain, but that the claim of the manufacturer who first works out a process is generally speaking as good as that of the inventor himself who first brings the idea to light after preliminary laboratory trial. This doctrine may not be a very welcome one to hopeful inventors, especially to young ones, but I venture to think that on consideration and in the light of experience some such rule as this will be found to be just and to work well.

With regard to the inventor who has an idea, and nothing more, and who does nothing in the laboratory or out of it to demonstrate the probable feasibility of his invention, I have nothing good to say or to suggest! I think that he may be quietly dropped out of our calculations; at all events, his contribution to sound progress is usually infinitesimal.

I should have liked to be able to allude to some of the many electrolytic processes which are fighting for supremacy, and threatening a terrible invasion of the old. That there are scores of busy, clever, and eager brains working away, endeavouring to bring these processes into successful operation, is patent to us all, and it behoves us to prepare for the invasion of this inventor as for that of all other inventors, and when he has won his spurs and proved his case, to welcome him with open mind, and by timely enterprise to show that we willingly accept and can promptly profit by the invasion of the true and real inventor.

## DISCUSSION.

Mr. E. K. MISPRAIT thought that Mr. Carey, when speaking of what might make an invention succeed or not, did not allude at all to the commercial conditions. It was perfectly possible that an invention might be really very admirable, and, from the technical point of view, be perfectly successful; at the same time, the commercial conditions might be such that it was not profitable; but that same invention, if the commercial conditions were changed, might become, and often did become, very valuable and profitable. As an illustration, he called attention to Mr. Longmaid's process for the calcination of copper ores with common salt, in order to extract the copper as soluble cupric chloride, then precipitating the copper with iron. This process, as worked by Mr. Longmaid, did not succeed; but some years later, when Spanish pyrites replaced Irish pyrites, the process, as applied by Mr. Henderson to the burnt ore, was profitable. But now the commercial conditions had again changed, and since the purple (iron) ore obtained in the process was not so valuable as previously, the commercial success of the process was naturally not so great. He was not quite sure that Mr. Carey had given the layman his full due, and he instanced Mr. Weldon as not originally a practical manufacturer. With respect to the payment of inventors, he entirely agreed with Mr. Carey, remarking that he himself had suggested a somewhat similar system, when giving evidence before the Patents Committee of the House of Commons. He thought it would be desirable that there should be a clause in any patent act, that the patentee should be bound to give his invention to the public on payment of royalties assessed by some impartial tribunal, and that, for chemical patents, at any rate, the royalty ought to take the form of Mr. Carey's suggestion.

Mr. W. P. THOMPSON agreed that patentees ought to be only too glad to arrange a free licence with the manufacturer, who was the first to give their patents a trial. But one effect of the clause for compulsory royalties was, that if a man gave a licence to anybody whatever, that was considered a precedent by which the Board of Trade adjudicated royalties. Almost every great master patent in the mechanical and chemical world had been brought out by laymen.

Professor CAMPBELL BROWN said that while he was delighted to see Mr. Carey sitting upon the false inventor with all the weight of his authority, he might say a great deal in favour of the true inventor if he needed any defence. The manufacturer and the world at large could not get on without him; the law of progress was of course a law of nature, and all they could do with wisdom was to put a brake upon the wheels, sufficient to see that each bit of progress rested upon a safe and sure foundation, and the next important thing to do was to take care that the inventor had as prolonged and thorough an education as could be obtained. The inventor would always exist, and he (Prof. Campbell Brown) did not think that his remuneration could be regulated by Act of Parliament.

Mr. CAREY, in reply, said that he was much inclined to agree with Mr. Misprait in calling Mr. Weldon a layman; but he could not quite agree with Mr. Thompson, when he stated that most of the great inventions had been made by laymen.

## Manchester Section.

ROOMS OF THE CHEMICAL CLUB, VICTORIA HOTEL.

Chairman: R. Forbes Carpenter.

Vice-Chairman: Edw. Schunck.

Committee:

G. H. Bailey.  
F. H. Bowman.  
G. E. Davis.  
C. Dreyfus.  
H. Grimshaw.  
P. Hart.

J. M. Irving.  
E. Knecht.  
I. Levinstein.  
W. H. Perkin, jun.  
Sir H. E. Roscoe, M.P.  
W. Thompson.

Hon. Local Secretary:

J. Carter Bell.

Bank House, The Cliff, Higher Broughton, Manchester.

SESSION 1894-95.

The second meeting of the Session will take place on Friday, December 7th, 1894.

Mr. Wm. Thomson, F.R.S.E., will show some new forms of chemical apparatus.

Mr. Peter S. Gilchrist (U.S.A.). "Pine Columns for saving Chamber Space in the Manufacture of Sulphuric Acid."  
Dr. Carl Otto Weber. "A new class of Lake Pigments."

Meeting held Friday, 2nd November 1894.

MR. R. FORBES CARPENTER IN THE CHAIR.

## CHAIRMAN'S ADDRESS.

SINCE our last meeting in this room Mr. Levinstein has felt it incumbent upon him to resign the chairmanship of the Section to which the Committee unanimously re-elected him in April last, and all the members of the Section will, I am sure, endorse the regret felt by the Committee that the pressure of other business engagements has been felt by Mr. Levinstein to be so great that he desired to be relieved of the duties of chairman while still remaining a member of the Committee. We shall long remember his zeal and energy in promoting the interests of our Society in general, and of our Manchester Section in particular. During our eleven years of existence as a section (since February 1883) there have been only three chairmen, Sir Henry Roscoe, Dr. Schunck, and Mr. Levinstein. The last has borne by far the larger brunt of the work, and we all know how indefatigable have been his labours in the interests of chemical industry; while the part he took in organising the chemical section of the Exhibition of 1887 is fresh in our minds and will always be gratefully appreciated. Let us hope that at no distant date Mr. Levinstein may feel able to re-assume the duties of chairman, to fulfil which for the current session your Committee have chosen my unworthy self as his successor.

I must claim your indulgence in the short address that I have prepared to open our session, for I have found some difficulty in selecting suitable material.

I have no intention or desire to obtrude my official personality upon you to night, for is not all that I have to say concerning my professional work contained in the Annual Report made to the Local Government Board by the Chief Inspector under the Alkali, &c. Works Regulation Acts. And yet there are some points that come under my notice which could claim no mention there that may be worthy of attention to those in this room, though my observations will not have the merit of novelty or originality.

Most of those present here, I daresay, had occasion to peruse and to criticise the rules issued by the Home Office under the Factory and Workshops Amendment Act of 1891 for the protection of those employed in various classes of industry, especially the chemical industries. In the case of the latter the rules embody the recommendations of a small committee, of which the chief inspector under the Alkali Acts was a member. On similar lines other committees for the pottery works and lead industry were constituted and reported to the Home Office.

I think I am not wrong in stating that the immediate stimulus to the Home Office in this matter was given by the publication of a work by Dr. J. T. Arlidge, "On the Hygiene, Diseases, and Mortality of Occupations," and it is to this book I desire to direct your attention by giving you some idea of its scope, of the careful judicial spirit in which Dr. Arlidge has approached his subject, and of the conclusions as regards some of our chemical industries at which he has arrived. I hope you have not been already prejudiced in respect to it by reading or hearing of a highly coloured review in the columns of the *Daily Chronicle* at the date of the appearance of the work, followed by a still more highly coloured series of articles entitled "Death in the Workshop"—to the writer of which any kind of physical toil seems a subject for lamentation and commiseration.

Let Dr. Arlidge speak for himself. "The problem to be solved is not merely the mortality and relative prevalence of particular diseases in the several occupations, but to discover what, if any, pathological consequences are rightly assignable to them, and what, when such are found, are their causes. It is not sufficient to analyse the returns of sickness and mortality. To arrive at a correct estimate of the effects positively attributable to an occupation it becomes necessary to eliminate all factors of disease not truly incident to it, a proceeding of great difficulty even when practicable."

Dr. Arlidge goes on to observe that physicians in France and Germany have for a long series of years directed their attention to industrial diseases and have collected a mass of observations. In France extended codes of rules and regulations have been framed to govern factories and to give security to the public against noxious trades, but they are, he says, to a serious extent, inoperative, because there exists no consolidated system of supervision by a distinct body of factory inspectors and medical men. But, as regards the comparative value of foreign statistics in the opinion of Dr. Arlidge, owing to the very dissimilar condition of life and work on the continent, and to the fact that some races of men (for instance the Celtic, Irish, and Welsh) have unfavourable racial health characteristics, the vital statistics of occupations pursued in England are only to be obtained from English sources.

In turning over the pages of the book, I find how frequently the author has to lament the deficiencies and lack of intimate knowledge caused by the absence of observations on the particular trade or occupation under notice. For instance, in speaking of the textile industries, he says: "In my search for facts relative to the changes wrought in lung tissue by the presence of dust arising from textile materials, silk, cotton, wool, and linen, I have been unsuccessful in finding any placed on record by medical men practising in the numerous and large manufacturing towns wherein those substances are in daily use, and in some of which there are medical schools with professors of medicine and pathology"; and again "Returns of the prevalent diseases of dyers and bleachers, of the ratio of mortality and of average age reached by those artisans are greatly needed, and ought to be forthcoming from the extensive works in Lancashire and Yorkshire. We are left to surmise what bodily ailments are likely to arise among bleachers, for, regrettably, no medical man has thought it worth while to determine what they actually are."

To conclude with one more example. "There is unfortunately a complete lack of statistics that might show the effects of high temperature upon workmen exposed to it, and nothing exists to help us in forming an opinion than records of individual cases of suffering; something beyond the general conviction that stokers and furnacemen on board ship are sickly and short-lived should be arrived at from opportunities for investigation possessed by the medical officers of our immense steam fleet." So much for the negative side; let us see a few of the positive conclusions. The impression I receive is that exposure to inhalation of dust, whether mineral or vegetable, is a more potent factor in reducing vitality and in producing distinct degradation of tissues than inhalation of acid vapours, speaking generally. In the latter case, personal qualities and idiosyncrasies and the principle of accommodation seem to count for more

than in the former, where inevitably, as time goes on, deteriorating change of tissues sets in. To come to more purely chemical manufacturing operations, the only table of comparative statistics Dr. Arlidge is able to give was contributed by Dr. Robertson, Assistant Medical Officer of Health at St. Helens, who had tabulated the cases of 503 labourers under two types—chemical and ordinary labourers; and under two death causes—diseases of lungs and all other diseases.

Diseases.	Mean Age at Death.	
	Ordinary Labourers.	Chemical Labourers.
Disease of lungs.....	40	35
All other diseases.....	50	44
Mean age at death, all causes..	49½	48

Unfortunately, says Dr. Robertson, these observations are made from an insufficient number of cases, 503, to do more than give a general indication of the fact that while there is a difference of 1½ years in the mean age at death from all causes, there is a difference of six years between the two classes from diseases of the lungs. "It may be added," says Dr. Arlidge, "as the mean age of chemical labourers is greater than that of ordinary labourers, as far as the mortality from diseases in general is concerned, we have an additional proof of the influence of occupation, on the one hand augmenting chest diseases, whilst on the other showing a less ratio for all other forms of sickness." Dr. Robertson, the compiler of these statistics, expresses his belief that the furnace labourers, by needless exposure to cold in a half-clothed state on their way home, are themselves in no small degree contributory to liability to bronchitis and disease of the respiratory tracts.

It is for this reason that, in my opinion, before these statistics could be accepted as conclusive that further differentiation should be made and comparison be instituted between, say, salt-cake furnacemen and workers at the glass furnaces at St. Helens (not "blowers," for here a fresh form of disease comes into play). Mr. Cooper, Medical Officer of Health at Widnes, has discovered no ill effects from the same vapours as at St. Helens, not even in the intensification of maladies. Children are remarkably healthy, contrasting favourably with those of Liverpool. The estuarine situation of Widnes is favourable for the dissipation of the vapours produced, and accounts, in Dr. Arlidge's opinion, for the difference in vital statistics noted in the two towns. Perhaps the most serious and weighty statement in regard to any chemical products is the following:—

"The comment is unavoidable that when we examine in detail the phenomena of poisoning with nitro-benzol, bisulphide of carbon, and various explosives, we cannot fail to recognise the existence of a series of maladies having certain common features, but which have no place in our accepted nosology. In other words, we are in the presence of a group of trade diseases unlike any ordinary group of every-day maladies which pathologists recognise and study, but upon which careful research may well be expended, and, in short, is in the interests of science demanded."

In describing, as he has to do, a very great variety of technical operations it is not unnatural that error has crept in, but I have come across none of any very serious moment. Dr. Arlidge wrongly attributes the dangers attendant on cleaning out sulphuric acid chambers to the sulphurous acid evolved on stirring up the bottom acid and sulphate of lead, instead of to the acid nitrogen oxides. The extremely deadly nature of exposure to these has a little escaped his notice. The only deaths with which I am acquainted directly due to being "gassed" are from this cause. Let me remind you of the lamented death of Mr. Woolley in this city, of the five (I think) deaths from the breakage of a carboy of nitric acid at Newcastle, near Messrs. Mawson and Swan's warehouse. These were of

persons not accustomed to work among acid vapour; but the danger of nitrous acid to those who are not strangers to its inhalation needs all the more to be insisted on, as exposure to it does not produce the feeling of suffocation or of spasm of the glottis to the same extent as do sulphurous, sulphuric acids, or chlorine, while the damage to lung tissue by absolute formation of chemical substitution products is not felt till perhaps the next day. I know of two deaths attributable to this cause in vitriol works.

Time does not allow me to go further in the examination of Dr. Arlidge's work, into his discrimination between the factors of town-made and trade-made diseases, or into the influence of the nature of occupation in rightly construing vital statistics, a subject in which he proceeds with the utmost caution and perspicacity. I must refer you to the first 60 pages of the work for instruction on these points. Before leaving this portion of my subject I should like to draw your attention to one of the recommendations of the Chemical Works Committee before mentioned, viz.: that at works where outbursts of dangerous gases are liable to occur, a bottle of compressed oxygen should be readily available. The first mention of the use of the "oxygen bottle" that I remember, was by an officer of the Royal Engineers in charge of some balloon experiments, when, in the discharge of the gas from the balloon at the ground level, one of the men got enveloped in the folds of the balloon and was found unconscious, on search being made for him: the poison was no doubt carbonic oxide; but the restoration of an apparently dead man on inflating the chest by artificial respiration and at the same time cautiously turning the tap of the oxygen bottle between the man's teeth was most extraordinarily rapid. In a recent number of the *Chemical Trade Journal*, I see that similar good results have followed in the case of asphyxiation by producer gas. The remedy is so simple and so easily

applied and is so instantaneously effective that I have no doubt many a valuable life will be saved by its means in the future.

And now, knowing how close is the connection of engineering with the existence and development of our chemical industries, I should like to direct your notice to a lecture delivered by the President of the Society of Mechanical Engineers, Professor A. W. Kennedy, F.R.S., at the Royal Institution as long ago as April 1893, on "Possible and Impossible Economics in the Utilisation of Energy," a subject that as treated by him may point the moral for chemists as well as engineers and the illustrations of which are so lucid and striking that he who runs may read. "Half our possibilities," says Professor Kennedy, and this I may call his text, "indeed far more than half our possibilities are of improving up to the best—it is infinitely harder to improve up from the best." For instance, plenty of room exists for raising the general efficiency of boiler work (Fig. 1), for if the average working all over the country were brought up to the standard of the best, there would probably be one-third less coal used every year than is now actually burned. But looking at Fig. 1, and knowing that of the 20 per cent. loss, we must lose something in radiation and pay a price for draught in heating the chimney gases, we cannot look for any very astounding increase of economy in boiler-work over the best that has been done. "No doubt any saving is possible," Professor Kennedy shrewdly remarks, "if one only starts with a bad enough example." In the next diagram, Fig. 2, are represented the theoretical

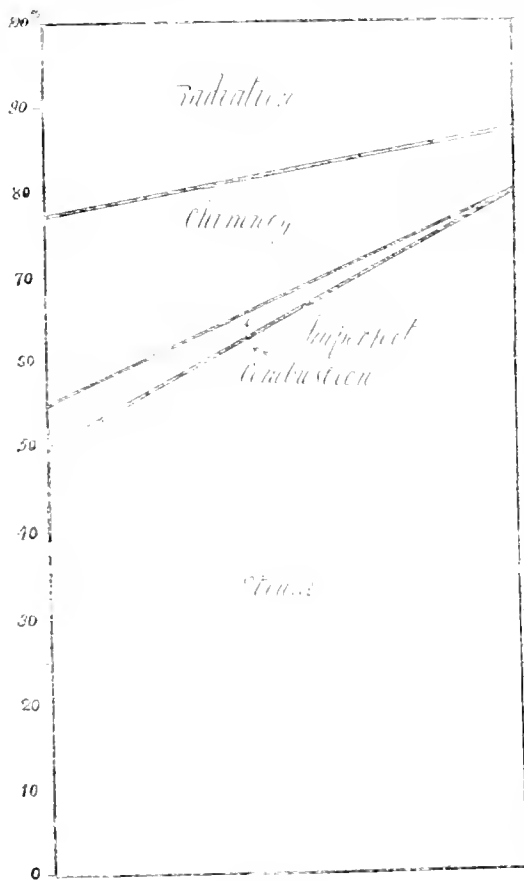


FIG. 1

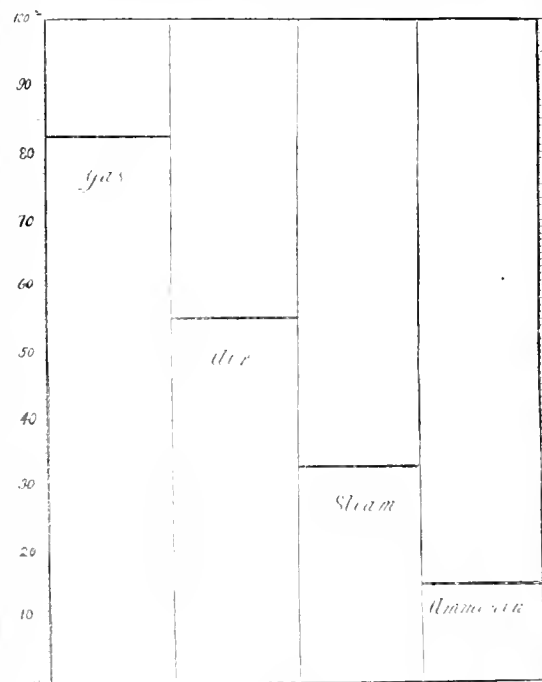


FIG. 2

possible efficiencies of the engine cycle of transformation of molecular into mechanical energy calculated from the familiar ratio  $\frac{T_1 - T_2}{T_1}$  where  $T_1$  represents the highest temperature above absolute zero at which the working fluid receives its heat, and  $T_2$  the lowest temperature above zero at which it parts with its heat—the numerator is the working difference of temperature, and the ratio, a limiting number that falls tolerably short of unity. With steam, apart from superheating the temperature  $T_1$  is dependent upon and limited by considerations of pressure and consequently of safety, with ammonia it is similarly limited, but the engine lies much further down the scale both as to



$T_1$  and  $T_2$ . But confining our attention for the moment to steam, in any actually possible steam engine, the process differs so much from the ideal that not more than 20 or 25 per cent. efficiency could be attained, even if the process were carried out perfectly, but in practice only 5 to 15 per cent. of the whole heat of the steam is ever turned into work. By further demonstration Professor Kennedy points out that the possibility of improvement is of doing *twice* as well as at present. In diagrams 3 and 4 is a graphic representation of this. In Fig. 3,

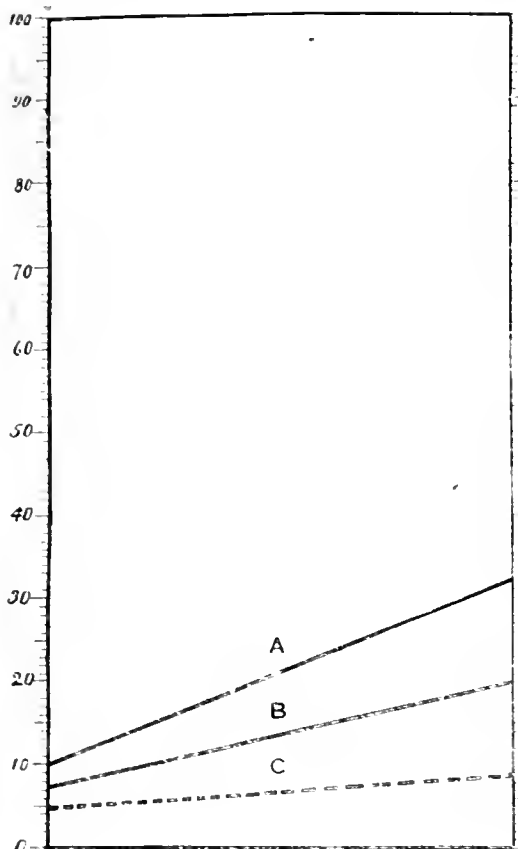


FIG 3

100 per cent. is taken as representing the whole energy of the steam, the area under A represents what might be turned into work by an ideally perfect engine working in an ideally perfect cycle (the left-hand side being a bad, the right-hand a good machine). The area under B represents an ideal engine working in actual cycle and under C the proportion actual engines turn into work. In Fig. 4, the ordinates of A are taken as 100 per cent., and the other two drawn in the relative places, the spaces showing the possibility of improvement in its real proportion. Professor Kennedy indicates that steam being in its working condition a vapour and not a gas, its temperature cannot be raised without at the same time raising its pressure. Considerations of safety and strength of materials become here very important, but even leaving these out of account, if you raise the maximum working pressure from 10 to 20 atmospheres, the theoretical maximum efficiency is raised only 10 per cent., clearly not a promising direction in which to work. The raising of the theoretical efficiency by the superheating of the steam is the direction in which Professor Kennedy seeks for improvement to be made, though it is only recently that the use of mineral lubricants has made it practicable. He mentions the fact that a little ordinary 4-h.p. engine, one cylinder, late cut off, moderate pressure, worked by steam

superheated 200° F., gave positively better results than a fine 100-h.p. Lancashire engine of the best Lancashire make, used with the same steam pressure and no superheating; under ordinary circumstances he would have expected the little one to use four times as much steam as the big one. Turning our attention now to gas engines, their theoretical efficiency is very high, about 80 per cent., the temperature of combustion in the cylinder may be as great as 2000° F. (so says Professor Kennedy, but 1800° C. was reached in Mr. Dugald Clerk's research experiments), yet the actual amount turned into work is less than in the steam engine (compare the lines C in Fig. 4, and B in Fig. 6).

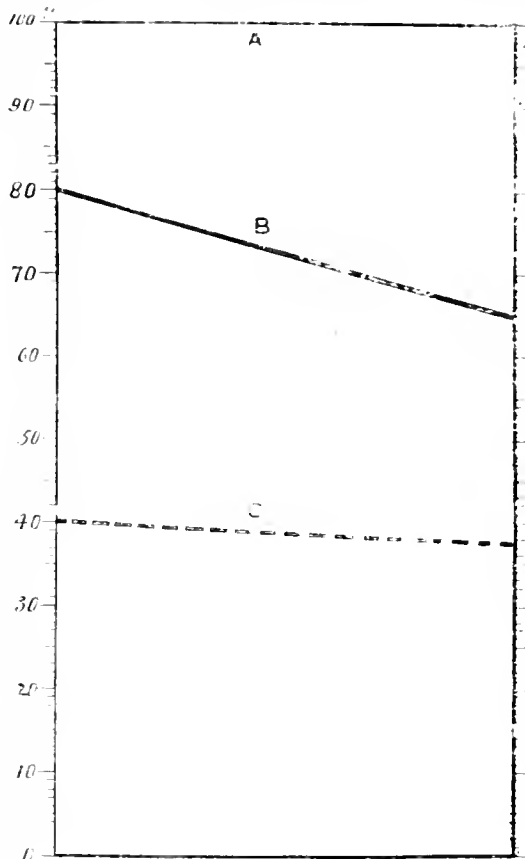


FIG 4

notwithstanding this, the actual energy utilized per thermal unit of combustion heat in a gas engine is very considerably greater than in a steam engine. The area above C in Fig. 6 represents the heat taken by the water surrounding the cylinder in the necessary water jacket. It is quite possible therefore for a gas engine of new type to appear in which only half the present quantity of gas will be needed. Possibly a reduction of initial temperature may be attended by economy and the use of producer gas with its lower thermal efficiency as compared with coal gas as may prove to be a step in the right direction, as many engineers (Messrs. Tangye, for instance) already declare to be proved. "A solution of the whole matter," says Professor Kennedy, "a solution which would also greatly help the steam engine problem, would be the discovery of some non-conducting material which was at the same time suitable for use in the construction of engines. Chemists have done much for us, but we still wait for our non-conductor, something as strong as iron and as easily machined, with the conductivity of sawdust is what we want, a material which at the same time must remain sensibly unaffected by very high temperatures and which will stand rubbing to any extent."



In Figs. 7 and 8 are contained a history of the gradually regenerative process by which the chemical energy of combustion is converted into electric light in a central lighting station; similar letters are used in each section of the figure, reading the transformations from left to right, for similar quantities of energy; in each section the losses of the section before are written off, and the quantity of energy actually carried forward is called 100 per cent.

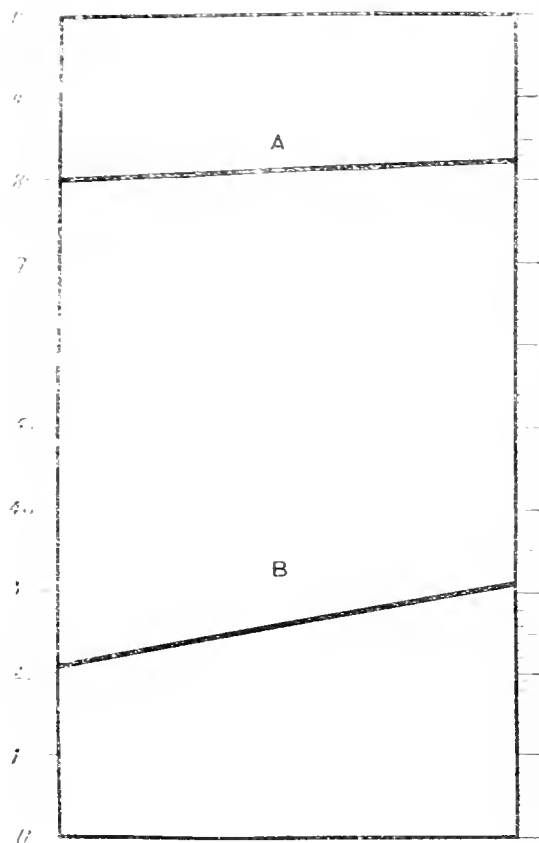


FIG. 5

In Fig. 7, K represents the losses in the boiler, H condensation in steam pipes and driving of pumps and other such losses inevitable in a central station. G is the part unavoidably lost from thermo-dynamic limitations; F the portion wasted by the engine, the remainder being turned into work; E the amount of heat-energy required to drive the engine itself, doing no work on the dynamo; D are the dynamo losses, C is expended in mains, transformers, &c., and of the energy reaching the incandescent lamps, B is heat and A only is light. Fig. 8 is drawn similarly for a gas engine worked by producer-gas; as regards the latter Professor Kennedy observes: "There seems no doubt the combination, although it does not much reduce the number of transformations, gives, under certain conditions, a very high economy of fuel indeed. I do not think the evidence before us is as yet sufficient, although I hope it shortly will be, to enable us to say how far under ordinary working conditions the actual combined efficiency of the whole plant will be distinctly greater than that of existing systems; the theoretical maximum efficiency is of course much higher." "To sum up," he says in conclusion of the examination of this subject: "the whole matter of possibilities and impossibilities, there does not seem to be anything very startling before us in the way of possible economies, except in the two directions of lamps as light producers, and of bringing up gas engines to their theoretical maximum."

We have heard much from enthusiasts during the last two years of the utilisation of town refuse in the generation of electric energy, the popular theory being that each urban community makes enough refuse with economical combustion to supply heat-energy enough to light the district with electric light. Our newspapers bear testimony to the judicious manipulation of the journalistic representative by the enterprising process promoter, and it is human nature to be enthusiastic about the conversion of dust-bin refuse into electrical energy under the soothing influence of a sumptuous lunch. But, looking at what can be reasonably expected of attainment, we must always bear in mind that the first essential in the disposal of town refuse is that the heat of combustion be so great, and the destructor or cremator be of such construction that all noxious gases have time as well as temperature for their final resolution into carbonic acid and water. This means steady feeding of the destructor through the 24 hours, while the demand for electric light is for a very limited proportion of the day. To charge the material into tubular steam boiler furnaces with the primary object of directly raising steam, and making the cremation of everything noxious the secondary consideration, is to run counter to all the advance that sanitary engineering has made in this department of recent years. I am very glad to be supported in this view by Professor Kennedy from engineering and thermal considerations, "of course," he says, "the stuff (town refuse) has the vast advantage that it costs nothing, even for cartage, but I cannot help fearing that like most things that cost nothing, it will be found to be worth very little more than it costs. I schemed sometime ago the boilers at a station in the north to try the experiment of using waste gases from a town destructor, but have not been sanguine enough to hope that I can do more in this way than save some of the cost of banking fires or running at very low load; I shall be delighted if I am mistaken in my estimate. In any case it has to be remembered that the

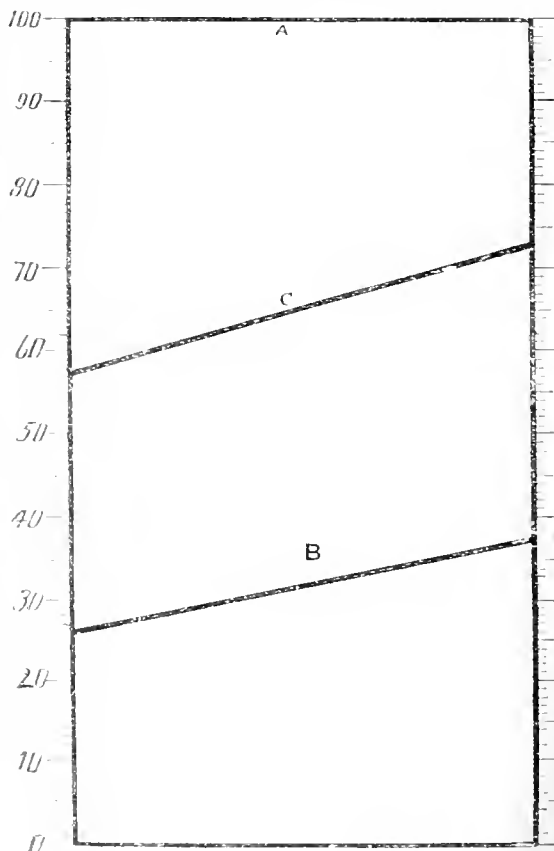
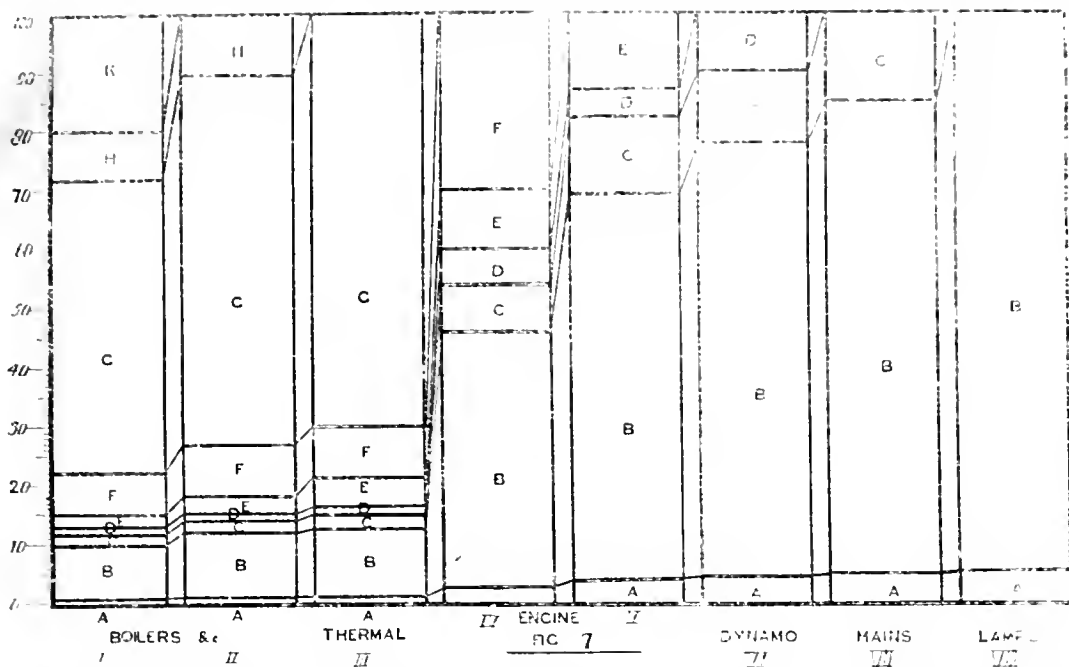


FIG. 6

dust destructor works on uniformly day and night delivering a uniform amount of heat, while the boilers of a lighting station want heat non-continuously in most variable fashion. If, therefore, more than a very small fraction of the destructor heat is to be utilised arrangements must be made for storing that heat somehow, so that it can be drawn upon as wanted; with thermal storage there is at least a

theoretical possibility of using most of the destructor's heat without storage. 70 per cent. of it is probably the largest amount that can possibly be utilised." With this quotation I take leave of one of the most suggestive, wise, and witty discourses that I have had the good fortune of recent years to come across in my reading. Those who have access to a file of "The Electrician" will find a good report of the



lecture in the numbers August 4, 11, and 18, 1893. I take this opportunity of expressing my thanks to the author for his kind permission to me to make use of the diagrams that so much aid in understanding the subject.

The question of possibilities and impossibilities in the development of electrolysis has received striking illustration by the publication by Mr. H. Y. Casner in the columns of *Engineering* and later of *The Chemical Trade Journal* of details of his electrolytic process for production of caustic soda and chlorine which has been continuously running at the works of the Aluminium Company, Oldbury, since August. It may be of interest to dwell for a short time on the developments that have taken place since 1888. In every paper that Dr. Hurter has communicated to our Society you will find the same high scientific qualities, and certainly his address to the Liverpool Section in November, 1888 (this Journal, Vol. VII. p. 719-726) is no exception to his high standard. The possibilities of electrolysis as they presented themselves to his mind at that time are there dealt with, and we are enabled now to see what amount of advance has been made in electrical efficiency since then. Taking the simplest electrolytic process known where the electrolyte does not change its constitution—the operation of copper refining—he found a loss of efficiency of 50 per cent., from Messrs. Siemens and Halske's statement of results. It is necessary to proceed with great caution in comparing the statements of electrical efficiencies obtained in different electrolytic processes as often entirely different meanings are attached to the word "efficiency." Dr. Hurter by his context takes the minimum voltage demanded by theoretical considerations and deduces his percentage loss by measuring decomposition per watt. By the custom of electricians, electrical efficiency is determined by the yield per ampère, a very different matter. Mr. Casner inclines to the latter view, he says, apropos of his process—"the electromotive force being 2.3 volts, it is evident any extra voltage is due to the internal resistance of the cell. As the amount of this internal resistance increases relatively to the

quantity or volume of the current passing, the whole matter resolves itself into one of cell construction, so as to arrange a cell that will be commercially practical by taking a large current without materially increasing the extra internal resistance. With almost any cell it is quite possible to pass a few amperes at almost theoretical voltage, but in attempting to increase the volume in order to obtain a practical result, the resistance increases so rapidly in proportion to the volume of the current as to necessitate the use of enormous cells which at best have a comparatively small output. If it is theoretically possible to pass one ampère of current through a cell at 2.3 volts, it certainly cannot be said that it is theoretically possible to pass 550 amperes at the same voltage, entirely irrespective of what is or is not the technical meaning of electrical efficiency."

And yet to my mind all this is a little academic, for the question of yield per watt, or, what is the same thing, its equivalent in mechanical energy or horse-power, is what will decide the investment of capital in trying a process.

It speaks, however, well for Mr. Casner's process that his actual voltage should be only 4, and ampère efficiency 88.5 for, this brings the watt efficiency of a decomposition admittedly far more complex than that of copper refining to somewhere approaching 50 per cent. of the power utilised. It is generally admitted that in the present state of the industry no electrolytic soda process would pay in this country at a higher voltage than 4.

The Greenwood and Le Sueur electrolytic processes described by Messrs. Cross and Bevan (this Journal, 1892, Vol. XI. p. 964, and 1894, Vol. VIII. p. 453) show ampère efficiencies of 80 to 85 per cent. working at 4.5 volts. These, I understand, are working in commercial scale in the United States. Mr. Hasenclever in his report on German chemical industry in 1892, described the remarkable progress of electrolysis of potassium and magnesium chlorides. Some processes therefore are practical.

Few of us realise the small amount of decomposition one ampère produces. Dr. Hurter states it in this way: "It

takes a current of 10,000 amperes day and night every day for three years to produce in one electrolytic cell one ton of hydrogen gas or its equivalent of any other substance. At the (1888) works of Messrs. Gaskell, Deacon, & Co., about four tons of 95 per cent. salt are decomposed per hour. To do that work with the theoretical amount of electricity

engine power equivalent to 5,728 horse power would be necessary, and it would require 100 large boilers to supply the necessary steam."

There is nothing in this statement to be modified by the light of to day except that the basis of the calculation was for the products that appeared H and Cl, 1.65 volts for the

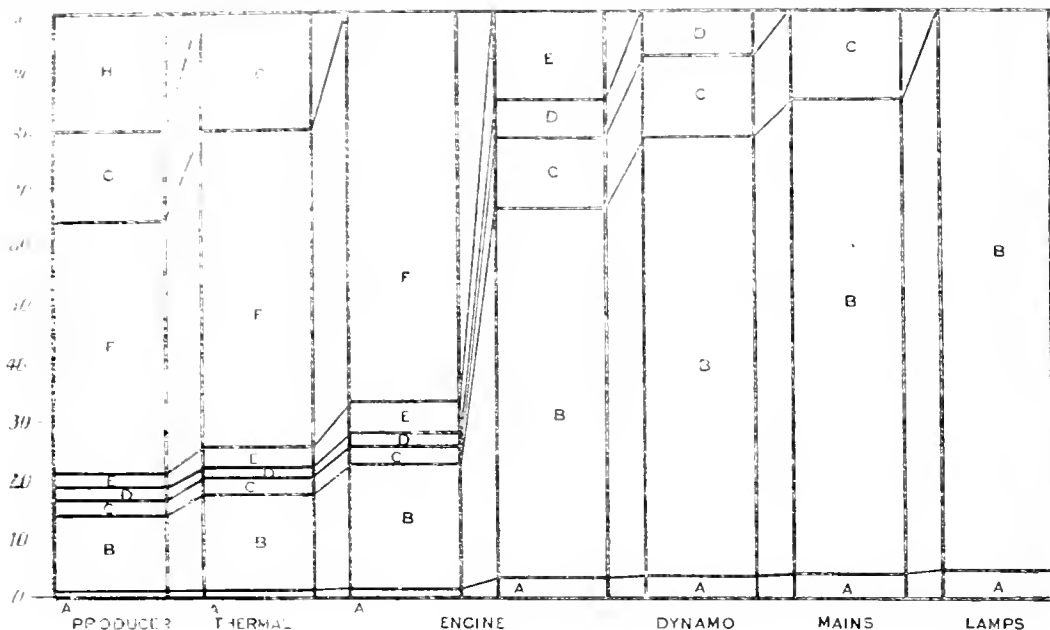


FIG. 2

back electromotive force, and 0.35 volts for resistance. This is correct. Dr. Hurter now states when no current is passing, but, while the current is passing the back electromotive force is 2.25 volts, without resistance, or 2.60 with —Mr. Castner's basis of decomposition (theoretical) is 2.3 volts.

You will notice that Dr. Hurter's remaining figures almost hold their own to-day, first the loss of heat during its conversion into mechanical energy, 90 per cent. (Kennedy, 85 per cent., Fig. 7, losses K. to F.). Second, a loss of conversion of mechanical energy into current of 26 per cent. (corresponding closely with Professor Kennedy's E. and D., Sec. V.). The only place in which I think, from my point of view, Dr. Hurter fell into somewhat serious error was, when speaking of the prospects of electrolysis ousting the Leblanc process. He says, p. 723: "I find that it would cost nearly 1/2 to decompose one ton of salt (electrolytically) and to obtain therefrom chlorine as gas, and a solution of salt containing caustic soda and other things for it could not be anything else. The Leblanc process sells the whole of the products of from 1 ton of salt for less than 6/."

The basis of Dr. Hurter's calculation was  $\frac{1}{2}$  ton of caustic, 70 per cent., and  $\frac{1}{2}$  ton of bleach; the former at 7/., the latter at 5/ per ton. But while electrolysis at that time had not done better than this the theoretical possibilities showed approximately,  $\frac{1}{2}$  ton of bleach and  $\frac{1}{2}$  ton caustic of 70 per cent. Surely in forecasting the future these possibilities should have been taken into account. As regards the Leblanc process no startling improvement is to be expected in the manufacture of caustic, but in that of bleach much has been done since 1888, especially by the Deacon-Hasenclever process to increase the yield of bleach per ton of salt, the limit of attainment is however by now pretty well known.

The only possible rival type in Mr. Castner's mind to his own process is the fusion type of process of which Mr. Vautin's is an example. (This Journal, 1894, 448—455), shortly to be in operation in Lancashire. Mr. Vautin found

in an experimental trial the voltage at which the current will pass 4 amperes to be 2 only, as compared with 1.4 amperes at  $3\frac{1}{2}$  volts in an exactly similar experiment in the wet way, and this is a marked feature in its favour, but the cost of maintenance of the chloride in a state of fusion, and the wear and tear of the carbon anodes are matters which only trial on working scale can prove. Mr. Castner appears to have been well aware of the rocks on which other electrolytic processes have broken down, of the necessity of getting a large number of amperes, or current of large volume, to pass at low voltage, necessitating the abandonment of diaphragms, of the accumulation where no diaphragm is used of hypochlorites and other impurities in the solution necessitating these being rejected from time to time, of the disintegrating effects of these on the carbon anodes (the hypochlorite itself electrolysing with evolution of oxygen from the carbon) and to have skilfully devised his plant so as to turn the flank of one and all these difficulties. Whether the introduction of mercury as a cathode involves a loss at this point by mercury passing into solution remains to be proved. This is the solitary point on which he is silent after the criticisms the process has received, and it is one to which working the process for a considerable time is the only answer. The voltage 4 at which the process is worked is as low as that of any working electrolytic process, if it is not lower, considering the number of amperes passing at that voltage, a very important point for commercial success. The future of the process will be watched with very great interest, and while the horse power necessary for an extensive work, if raised by boiler power, is large, yet, if the water power available at Niagara can allow the electrical syndicate formed to utilise it, to supply 1 e.h.p. for 5 dols. per annum,\* the cost will come to about 1/10 of the cost in England to day of 1 i.h.p. (viz.,  $\frac{1}{10}$  d. per hour), and we might expect great developments of

\* The latest advices I receive show this estimate to have been inaccurate, the actual figures at which 10,000 e.h.p. have been contracted for being 18 dols. per annum per e.h.p., equivalent to  $\frac{1}{10}$  d. per hour.

the process under conditions such as these at Niagara or elsewhere.

For those of us with a sufficient knowledge of mathematics Dr. Hurter's inaugural address to the Liverpool Physical Society in October 1893 on electrolysis will well repay study. The phenomena of conductivity of solutions, of secondary reactions, and the curious disturbing influence known as "migration of the ions" are passed in review and the results of a large number of most interesting and patiently conducted experiments are there recorded and explained.

It seems ungracious to envy the prosperity and efficiency of societies formed like our own for the promotion of technical knowledge and interchange of ideas among its members, but I sometimes entertain these sentiments towards the various district Societies of Gas Engineers, as chemical subjects connected with their industry would find ample scope for discussion amongst ourselves, and these papers tend naturally enough to go to the engineers rather than to come to our Society. I hope this session will not pass without our receiving one or more papers on chemical points connected with the gas industry, for this was never at a stage of development more interesting to the chemist than at the present time. Two movements opposite in principle are dividing the gas world; let me try and shortly summarise the position. In view of the tendency of parliamentary demands for increase of candle-power, and of the limitation of supplies of candle, coupled with the cheap price of petroleum oils, several processes which you will find described in our Journal have come more or less into vogue for increasing the illuminating power of gas; among others I might name the Peebles process, and the Tatham or oxyoil process. In others carburetted water-gas, as in the Lowe process, is adopted as an adjunct. I learn on the authority of Professor Vivian Lewes that last winter 10 per cent. of the gas supplied by the Gas Light and Coke Company, of London, was derived from this latter source, where the ordinary gas of 14 to 15 candles was enriched with the 22 candle water-gas and sent out at 17 or 17.5 candles to ensure getting the required 16 candles at the testing stations. That gas engineers should be alive to the advantage of having a plant that can be so rapidly put into operation as the one I mention is not to be wondered at when we remember the demands upon their plant that are sure to arise on the advent of fog lasting perhaps a week. It is impossible to heat up beds of retorts quickly enough for the requirements. The sanitary drawback, however, will at once suggest itself to you, it is not desirable to increase the percentage of carbonic oxide in our burning gas, as the increase of danger of accident by escape and use rises in direct proportion. I will do no more than mention this feature that should not be lost sight of. But there is another feature of greater interest to us as chemists. In proportion as the percentages of carbonic oxide and hydrogen are increased in such degree does the height of the flame of the gas lessen when burned, and to this cause more than any other does Professor Vivian Lewes attribute the complaints by consumers of defect of illuminating efficiency that have arisen. The following are some figures obtained by him in burning 5 ft. of different gases per hour in a standard argand:—

Gas.	Height of Flame in Inches.
Hydrogen.....	0.985
Carbon monoxide.....	2.206
Carburetted water-gas.....	2.5
Methane.....	1.25

This statement prepares us now to appreciate the argument of the other school of gas engineers that contains the eminent names of Mr. George Livesey and Mr. Thomas Newbigging, and receives the weighty support of Professor Vivian Lewes. Mr. Newbigging is perhaps the most recent exponent of the view that the enrichment processes are all

tendencies in the wrong direction. He distinguishes between illuminating power and illuminating effect, the former, of course is the photometric measurement and comparison of the light with that of standard candles, in which process the eye of the observer is carefully shielded from all direct sight of the flames. A light of high illuminating power may not have by any means so great a proportionate illuminating effect (take the arc light as an instance of this familiar to every one). As one passes from the south of England to Scotland the illuminating power of gas rises from 16 candles in London to 18 to 19 in Manchester and Salford, and to 22 and 24 in Edinburgh; will any one, says Mr. Newbigging, contend that it is possible to obtain proportionate increase of illuminating effect. The natural illuminating power, without enrichment, given by the Durham coals in use in London is about 14.5 candles, and Professor Vivian Lewes found hardly any difference in candle units per foot of gas consumed in No. 1 and No. 5 flat flame burners (which form 85 per cent. of those in domestic use) between 14.5 and 16-candle gas. The absolute figures of candle unit efficiency being No. 4, 1.74 and No. 5, 1.87; when I tell you that the maximum candle unit efficiency per foot of gas when burned in regenerative burners is 10 you will see that there is plenty of room to improve up to the best in the department of burners, and that it is quite arguable that the great cost of enrichment is so much money wasted. As an experiment the South Metropolitan Gas Company sent out for the period of a fortnight unenriched gas of about 14.5 candles without receiving complaint from a single consumer. The point I have previously mentioned as to size of flame has a distinct bearing on this question of illuminating effect. A natural coal-gas, containing its due proportion of methane will give a larger flame than an enriched one, and the illuminating effect of the larger flame will be as great as that of a smaller and more intensely brilliant surface of flame obtained from an enriched gas of higher candle power. In addition there are physiological reasons supporting the contention of this school, the eye automatically and involuntarily closing against admitting too many rays from a highly brilliant flame. It will be most interesting to watch the progress of the newer ideas. If, in Manchester, some of the money spent in enrichment were only diverted to purification from sulphur impurities we should (with all due respect to Professor Lewes, who is still unconvinced by the arguments this Section has brought forward) be deeply grateful to the authorities. Those of you who feel interested enough in the subject to make further acquaintance with it will do well to read the paper of Mr. Thomas Newbigging at the meeting of the Gas Institute this summer, and the discussion thereon to be found *in extenso* in that most excellent technical publication, "The Journal of Gas Lighting," July 1894. One drawback to the enrichment processes I have not named, the fact that the gas is the carrier of hydrocarbon vapours. This leads to stratification in the gas holders, and when the gas passes along the mains with the friction against the skin of the pipe, tendency to condensation and deposit of liquid hydrocarbons arises, so that to reach the consumer gas of proportionately higher candle power must be sent out from the works. We had some years ago, in Salford gas, during the period when the Hawkins process was in use, sufficient evidence of this phenomenon.

In covering so much and such varied ground this evening I have necessarily been able to touch but lightly on each subject. If my remarks have been at all suggestive that is all the merit that I hope for them to possess.

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Meeting held in the Durham College of Science,  
Newcastle-on-Tyne, on Thursday, November 8th, 1894.

MR. GEO. T. FRANCE IN THE CHAIR.

### PRACTICAL RESULTS OF THE LUNGE-ROHRMANN PLATE-TOWERS FOR CONDENSING HYDROCHLORIC ACID.

BY G. LUNGE.

AS ONCE of practice is better than a pound of theory—this, I think, everybody will admit, without in the least depreciating the immense services theory can render to practice, services which the writer is the very last person to hold cheap. After some tilting between Dr. Harter and myself in the pages of this Journal, in which theoretical reasoning played the greater part, and which I did not care to continue at the time, fearing that it might appear tedious, it will be appropriate to relate something about the *practical* results obtained with plate-towers (sometimes called "Lunge towers") on a large scale.

An opportunity for this arose in the following way. A little more than a year ago, Mr. Richard Curtius, managing partner of one of the oldest German alkali works, Messrs. E. Matthes and Weber, of Duisburg, had his attention drawn to the plate-towers. I believe principally through my friendly polemics with Dr. Harter. He made up his mind that he would go in for their use as hydrochloric acid condensers, not in a half-hearted way, which might very easily have led to an erroneous result, but as thoroughly as possible. He fitted up one of his salt-cake furnaces with a condensing plant based on that system, and almost at the outset obtained the most encouraging results. This furnace has been running ever since with plate-towers, and, as we shall see, produces a better yield of acid than any system which has been hitherto known. Most other manufacturers would have been contented with such a result, and would simply have continued to work on in the way in which they had succeeded from the first. But Mr. Curtius, with an enterprise and a zeal for technical chemistry which cannot be sufficiently praised (seeing that he is not in the least interested in making propaganda for other people's inventions), made it his task to try various modifications of the first system, in order to completely elucidate that subject, and he has not yet finished his experiments. But, as the works' manager, Mr. Lasche, with the consent of his chief, is about to publish the results hitherto obtained, and as I have had an opportunity of seeing his report before it goes to the press, I think it will interest English and American chemists to get a short summary of it several months before it would be otherwise accessible to them, together with some explanatory remarks of my own.

It is hardly necessary to point out that I give Mr. Lasche's results in an absolutely unvarnished form. The reader is therefore not to be treated to an *ex parte* statement, but to absolute *facts*, established at a first-class works, and to be published by its responsible manager, whom I had never seen or communicated with in my life before the termination of the experiments to be reported upon.

The salt-cake furnace in question is a muffle furnace, decomposing every 24 hours 10 charges, amounting in all to 4 tons 5 cwt., or 30 tons per week. The pan-gas and muffle gas travel separately through about 30 ft. of 16-in. pipes, and then enter into two small towers, 3 ft. wide and 10 or 13 ft. high, one of them empty, the other one filled with coke. Here a little impure acid, with much sulphuric acid, is condensed. The second tower was intended to serve as a wash-tower, by being fed with water or weak acid, but this was not done during the experimental period, both towers only working as air-coolers. The gas then enters an empty receiver, and then passes through six ordinary earthenware receivers, holding 66 gallons each, the connections being made by high bends, to increase the cooling action. In all, the gases travel over a distance of 130 ft. (reckoning horizontal and upright way) before entering the plate-towers. These consist of nine cylinders, 3 ft. wide and 3 ft. 3 ins. high. The bottom cylinder is empty; the next three cylinders contain 60 Lunge-Rohrman plates; the fifth cylinder is again empty; the sixth and seventh contain coke, resting on an earthenware grating; and the two top cylinders are again empty. The plates are held in the grooves of annular bearers, 2 in. high and tightly ground upon one another, so that a second column is formed within the first, the space between them being filled with pebbles. The diameter of the plates themselves is 2 ft. 2 in. The 15 bottom plates have holes of 12 mm. diameter; the next 35 plates, holes of 7 mm.; the last 10 plates, holes of 6 mm. The top of the tower is provided with an ordinary water-spreading arrangement, and just below there is an exit pipe, slanting a little upwards, and then leading downwards to the chimney flue, a "sight" being interposed, which at the same time serves for regulating the draught.

The acid formed in the towers flows through the six receivers and runs off from the first receiver. At first it was attempted to work without these receivers, which not merely aid in the condensation, but also serve for equalising the unavoidable variations of work. This attempt, as was to be expected, failed; the strength of the acid and its temperature varied within wide limits. But after the six receivers had been interposed, the specific gravity of the acid, when both that coming from the pan and from the muffle were united, varied only between 1.161 and 1.167, and averaged 1.164 at 15° C. All following specific gravities are also reduced to that temperature.

The temperature of the gases on entering the pan-set was from 15° to 55°, average 34° C.; in the muffle-set 15° to 59°, average 49°. It was always highest towards the end of an operation. On entering the plate-tower in the pan-set it was 14° to 37°, average 24°; in the muffle set 16° to 38°, average 28° C. The acid running from the first receiver in the pan-set showed sp. gr. 1.141 to 1.170, average 1.160; in the muffle-set 1.152 to 1.175, average 1.163; between the tower and the nearest receiver in the pan-set 1.136 to 1.162, average 1.168; in the muffle-set 1.132 to 1.162, average 1.145. During October 1893, for instance, there were made 181 charges of 8½ cwt. rock-salt each, or 119 tons 8½ cwt., which yielded 218 tons 16½ cwt. of hydrochloric acid of sp. gr. 1.162, or 183.2 parts of hydrochloric acid for every 100 parts of rock-salt decomposed. This is a yield not attained by *any* other condensing plant in any part of the world, as far as my knowledge goes. I have taken some trouble in collecting information on this point, for use in my treatise on Sulphuric acid and Alkali, and the highest yield I have heard of from anywhere is only 180 parts acid of sp. gr. 1.16 (20° B.) on 100 rock-salt.

The temperature of the exit gases in the pan-set was 30° to 40° C. Mr. Lasche also tested their acidity, but his figures admit of no comparison with those given by the English alkali inspectors, as he only tested for the total

acidity including  $\text{SO}_2$  and  $\text{SO}_3$ , and took his tests immediately behind the towers, whilst the alkali inspectors test the condensers only for  $\text{HCl}$ , and the total acidity in the chimney outlets, where the condenser gases are diluted with other gases, before issuing into the outer air. So much is certain that, tested in the English fashion, the Duisburg towers must have shown themselves to be equal to the very best of English condensing plants, since the acid really escaping from them into the air is notoriously less than in any other known case.

At the plant in question an arrangement had been made for taking samples of the acid coming down from the small coke column, before reaching the top plate of the plate-tower proper. This acid, very remarkably, at the pan-tower showed sp. gr. 1.108 to 1.158, average 1.133; at the muffle-tower 1.067 to 1.121, average 1.094. It seemed from the outset hardly possible that a corresponding quantity of  $\text{HCl}$  should have escaped condensation on the plates, and quite impossible that it should have been all condensed by a coke-column 2 ft. wide and 6 ft. high. That must be from the first the conclusion of people not prejudiced at the outset against the plate-towers, but inclined to give them a fair chance. Mr. Lasche indeed came to the conclusion that very much liquid acid must have been carried away mechanically by the draught from the plates, and mechanically retained by the coke. The experiments to be described anon, where the coke was left out, and it was tried to avoid the mechanical loss in other ways, did not yield as good results as the original plan, with a little coke at the top; but, as we shall see, the yield even then was so good that it is utterly out of the question to attribute to the coke much more than the mechanical part above mentioned.

The Duisburg condensing plant, as above described, had given such excellent results that it could not be expected to improve them further. Mr. Curtius, however, in the interest of technological science, resolved to try some modifications of the plant, in order to completely elucidate the working of the plate-tower system, and in particular the part played by that small column of coke. The work was stopped, the coke was taken out, and work was recommenced with the plates alone. The draught was regulated by means of the "sights" in such manner that the anemometer in the exit pipe showed an inward pressure of 6 mm. water; this draught was found to answer best, and to obviate any escape of gas at the furnace doors. It was at once found that the distribution of the water on the top had to be improved, and even then the yield of acid was now only 169.6 per cent. of sp. gr. 1.157. Such a yield is still superior to that of a very large number of works, and some years ago it would have been considered first-class at most places, but they could not think at Duisburg of leaving matters at that point, since after removing the coke nearly 8 per cent. less acid than before had been obtained. The aim was now to see how many additional plates would be required to attain the same yield as with the coke. The first trial was made by putting three additional plates on the top of the others, with holes of 5 mm. diameter; but this would not work at all, as the draught was too much interfered with. Still this experiment was very valuable in settling the lowest limit for the size of holes for this purpose. Evidently holes 5 mm. wide are too narrow to allow the passage of the gas at the same time as that of the comparatively large quantity of water running down in a hydrochloric acid condenser, although long experience has shown that such holes are most suitable for apparatus employed for recovering nitric acid from the lower oxides of nitrogen, in which case only a very small feed of water is used. Hence the minimum size of holes for  $\text{HCl}$  condensers must be placed at 6-millimetre holes, and this may be the proper size altogether, but plates with 7-mm. holes are also doing very good work. Any width above this is decidedly objectionable, whilst for sulphuric acid apparatus, owing to the greater viscosity of even chamber acid, the minimum size of holes has been ascertained by experience to be 8 mm. The 15 plates at the bottom of the Duisburg towers, with 12-mm. holes, evidently did practically no condensing work at all; this part of the towers always remained quite cold, the heating only commencing where the plates with 7-mm.

holes started. This is no matter of surprise; the 12-mm. holes leave hardly any margin for the acid to stand on the plates, which is one of the guiding principles in the construction of the Lange plates; they act merely like sieves, with hardly any condensing power, so that the space occupied by these plates has very little more efficiency than if it were empty.

The plates with 5 mm. holes were now taken out, and 11 other plates were put in, 10 with 7-mm. and one with 6-mm. holes. The draught had to be increased to 12 mm. water pressure on the pan-tower and 14 mm. on the muffle-tower. The yield now rose to 177.7 parts acid of sp. gr. 1.160, the specific gravity varying from 1.144 to 1.161 on the pan side, and from 1.150 to 1.164 on the muffle side. This yield most assuredly exceeds that obtained at a great majority of works, but it is about 3 per cent. less than that obtained with the original plant, that is, with the small coke-column at the top of the plates. The acidity of the gases was also decidedly greater than before. Mr. Lasche points out that the yield would probably have entirely equalled that obtained before, if the 15 plates with 12-mm. holes had been replaced by such with 7- or 6-mm. holes, and that in this case the coke would have been found quite unnecessary.

Even if it should be found that by some such arrangement the coke could be entirely dispensed with, I am at the present inclined to advocate retaining such a small column of coke at the top of the plates. The additional two cylinders and the few cubic feet of coke cost very little indeed; but although they may not, and indeed cannot, exercise any essential condensing action in the proper sense, they are evidently very useful in perfecting the equal spreading of the water before it reaches the top plate (which is very important), and in mechanically retaining the liquid acid drops carried upwards by the draught; also in counteracting the tendency of the feeding water to be drawn towards the exit pipe, which is placed just below the cover.

I must point to a drawback of the plate-towers as erected at Duisburg (and elsewhere) which will not occur in future. I have explained above that the annular earthenware bearers form a complete inner cylinder within the outer shell. The space between was loosely filled with pebbles, but formed, of course, a non-conducting air-jacket. This, together with the double earthenware envelope, must be very prejudicial to the dissipation of the heat produced within the tower by the action of  $\text{HCl}$  upon water, and must decidedly lower the efficiency of the tower. In future the towers will be constructed with a new form of cylinder, provided with projections for bearing the plates, the present annular receivers being entirely done away with. The cooling action of the air will then be much more complete, and it may be expected that the same excellent results as hitherto will be obtained with a smaller number of plates.

The experiments at Duisburg are now being continued, to try the action of towers of larger diameter with different kinds of plates, and so forth, and we shall in due time hear more about it. But already now, as Mr. Curtius states, the Duisburg trial has absolutely established the success of the plate-tower system for condensing hydrochloric acid. This small apparatus has shown itself more than equally efficient with either the ordinary huge coke-towers, with or without the addition of some cast-irons, or else the 100 stoneware receivers, followed by small coke-towers, as usual on the Continent. These trials have also fully justified my assertion, formerly impugned on theoretical grounds by the high authority of Dr. Hurter, according to which a plate-tower possesses from 10 to 20 times the condensing power of an equal space of coke-tower (compare this Journal, 1893, 330 and 421). Even if we comprise in our calculation those inefficient 15 plates with 12-mm. holes, the inner space of the Duisburg towers, so far as it is filled with plates, is only 2 ft. 4 in. wide and 10 ft. high, equal to just 74 cub. ft., for both towers together. With a decomposition of 30 tons per week this is = 2.46 cub. ft. per ton per week. In well-conducted English works the condensing coke-tower space averages about 50 cub. ft. per ton per week. It is true that this comprises the empty spaces above and below the coke, but these are more than allowed for by our taking in on the other side the 15 plates with 12-mm. holes. For the sake of this comparison we

must deduct from the 50 ft. the space occupied by the sturdy coke-column in the plate-towers, to which we must of course assign neither more nor less efficiency than to an equal space in an ordinary coke-tower. The small coke-column in the plate-towers amounts, for both of them together, to a space of 19.3 cub. ft., or 1.61 cub. ft. per cent per week. Deducting this on both sides, we find 2.46 cub. ft. of plate-tower space equivalent with 48.36 cub. ft. of coke-tower space; that is, a ratio of 1 to 20. If due allowance were made for the 12-mm. hole plates, and for the present insufficiency of cooling action in the Duisburg towers, the comparison would be even more favourable to the latter. They furnished not merely a better yield of acid than the most gigantic coke-towers, but even the variations in the strength of the acid were no greater in the former than in the latter case. This result at the same time amounts to a distinct proof that the preliminary cooling of the gases, before they reach the condensers, really plays the very important part assigned to it by the late Dr. E. Angus Smith, and that the unavoidable rise of temperature, caused by the absorption of HCl in water, can be coped with much more easily and in a much smaller space than both some modern theories and the practice of some Widnes alkali-makers have allowed. Just in this particular respect, as we have seen, the Duisburg towers were decidedly not quite as efficient as they might have been, and as their successors will be in future, and yet they have completely attained their purpose.

#### DISCUSSION.

Dr. HURTER writes as follows:—Professor Lunge has given in his paper a number of facts from a similar paper by Mr. Lasche; and draws sweeping conclusions from facts, which, if they prove anything, prove the failure of the Lunge plates as compared with coke.

Usually the conclusions of a paper are the only part which most readers look at, leaving the tedious details to be studied by those whom it may concern. The concluding paragraph of Dr. Lunge's paper will undoubtedly lead the readers to think, that at Duisburg they have managed to condense the hydrochloric acid from 30 tons of salt per week in the very small space of 74 cub. feet, and that this result had been accomplished solely by the aid of the great efficiency of the Lunge plates, which, he states, are proved to have been twenty times as efficient as an equal space filled with coke.

The plant consisted, for a decomposition of 30 tons of salt per week, of a pan and muffle furnace and the following condensing systems, of which there were two sets, one for the pan, the other for the furnace-gases.

The two sets were identically alike, and comprised between them the following parts:—

- (1.) Cooling pipes, about 80 yards of 16-inch pipes.
- (2.) Two empty towers (one for each system) 2 feet 8 inches diameter and 10 to 12 feet high.
- (3.) Two towers filled with coke, but not fed with water (one for each system), same diameter, 17 feet high.
- (4.) Four large Bombonnes kept dry to collect all acid condensed up to this point (two for each system). No water was allowed to flow through these.
- (5.) Twelve large Bombonnes (six in each system), through which all the water from the following towers was flowing. These were found to be indispensable; without them the system would not work.
- (6.) Two Lunge plate towers (one for each system) 2 feet 8 inches diameter, about 30 feet high. The lower part of these towers contained at first 60 plates, and above them about 6 feet of coke; later on the coke was removed, and still later 11 plates were substituted for the coke.

The acid condensed in these towers flowed through the Bombonnes.

It is impossible for me to give the accurate cubical capacity of this compound system, but approximately I take it as about 1,100 cub. feet, 1,000 of which are in front of the Lunge towers. It is surely idle to talk of the wonderful results obtained with 74 cub. feet of plate room when these are preceded and assisted by 1,000 cub. feet of

space in front of them, unless indeed absolute proof were furnished that the space in question really does the greater part of the work.

The results we have at our disposal (as found in Mr. Lasche's paper) give, however, the direct contradiction to the statement that the 74 cub. feet of space filled with plates have contributed any extraordinary amount to the condensing power of the system.

We are told that the acid obtained at the outlet from the Bombonnes tested on an average 32.8° Tw. As the gases arrived at the Bombonnes with an average temperature of only 93° F., most of the steam had already condensed, and we may take it that the amount of water flowing from the Bombonnes was also the water flowing down the Lunge towers. Acid of 32.8° Tw. contains 32.3 per cent. of HCl and 67.7 per cent. water. Thus for every 32.3 parts of HCl condensed 67.7 water were flowing down the towers.

In the tower, above the plates, there was a layer of coke of 6 ft. depth, placed there probably to catch the last traces of hydrochloric acid which might escape from the plates, and also for the better distribution of the water.

Mr. Lasche had wisely made provisions for testing the strength of the acid flowing from the coke on to the plates. The result seems to have surprised him, for he says:—

"*Marvellously* the following strengths were obtained: at the pan condenser the acid flowing from the coke had on an average 26.6°, as minimum 21.6°, as maximum 31.6° Twaddel."

The acid from the tower, at the bottom, flowing into the Bombonnes, had an average strength of 29.6° Tw., minimum 27.2° Tw., maximum 32.1° Tw.

With these data it is easy to calculate that the 67.7 of water flowing down the tower absorbed in the coke space 21.2 parts of HCl, in the plate space 3.8 parts of HCl, and in the Bombonnes 4.3 parts of HCl.

The work of the system may thus be represented as follows:—

	Per Cent.
Acid condensed in the coke space .....	74.9
" " plate space .....	11.8
" " Bombonnes .....	13.3

It would be incorrect to draw the conclusion from these results that about 50 cubic ft. of coke space had done six times the work which was done by 74 cubic ft. of plate space.

The fact is that the work done by any part of a condensing system is not measurable at all, either by the acid condensed therein or by the changes of temperature which occur therein, but solely by the number of units of heat which the particular part of the system can disperse. In every condenser, as far as I know, without exception, the large bulk of HCl gas disappears just near the entrance of the condensing fluid. But the liquid acid there formed cannot be strengthened to the required degree, unless the heat of condensation be dispersed and the temperature of the acid lowered. It is the function of the other parts of the apparatus to do this.

For this reason the Duisburg system did not work at all without the Bombonnes; the Lunge tower was not able to cope with this task. Again, if on the top of the six feet of coke another six feet had been placed, the disappearance of the large bulk of HCl gas would have occurred in the upper six feet, and the condensation in the lower six feet of coke would have been a very small fraction, and not, as before, 75 per cent. After the removal of the coke the work done by it now devolved on the plates, and they were unable to cope with the work, as the results clearly prove.

The results after removal of the coke were very bad indeed, but they will not serve as a correct basis for the calculation of the comparative efficiency of coke and Lunge plates. None of the results detailed in Mr. Lasche's paper will serve that purpose, and until the managers of Duisburg will permit the whole of the plates of the Lunge towers to be removed and to be replaced by a rational coke packing we shall not be in a position to know that comparative efficiency. I devoutly hope the managers of the Duisburg works will in the interests of technical science make that most interesting experiment, and prove once for all the truth of Professor Lunge's assertion that the same space will do 20 times more work with plates than with coke.



As far as the managers at Duisburg went in the substitution of plates for coke, the results are decidedly in favour of coke, and they are alive to that fact.

The following comparison of results obtained (1) while working with coke, (2) while working without coke, (3) while working with 11 Lunge plates instead of coke, show the very marked superiority of coke over the plates:—

One hundred parts of rock salt yielded in form of liquid acid—

	Pure HCl.
(1) when working with coke.....	58.8 per cent.
(2) " " without coke.....	39.6 "
(3) " " with plates instead of coke.....	55.6 "

The exit gases contained free acid, which was expressed as  $\text{SO}_2$ , and which I give in grains per cubic foot. It will be seen that the substitution of 11 plates for the 6 ft. of coke caused the exit-tests to be doubled.

#### Pan Gas Exit-Tests (Average).

(1) When working with coke.....	1.24
(2) When working without coke.....	?
(3) With 11 plates instead of coke.....	2.44

#### Furnace Gas Exit-Tests.

(1) With coke.....	4.5
(2) Without coke.....	?
(3) With 11 plates instead of coke.....	12.7

It is a great pity that we are not in possession of sufficient data to calculate exactly in what part of the system the great loss of hydrochloric acid happened after the removal of the coke and before the introduction of the additional 11 plates in its place. There is little doubt, however, that most of this loss occurred in the furnace condenser. Even after the coke had been replaced by plates the production was bad, and the following results obtained from Mr. Lasche's figures by a calculation similar to the one already made, show that it is the furnace condenser where this loss occurs. Pan gases are comparatively easily dealt with, while the furnace gases frequently give trouble.

While the furnace condenser was working with coke in the upper six feet, the condensation of the hydrochloric acid was distributed as follows:—

	Per Cent. of Total condensed.
Condensed in Lunge towers.....	31
Condensed in Bombonnes.....	9

After 11 plates had been substituted for the six feet of coke the distribution was as follows:—

	Per Cent. of Total.
Condensed in Lunge tower.....	69.9
Condensed in Bombonnes.....	30.1

As the alteration in the tower could affect the condensing power of the Bombonnes but little, the acid flowing from them being of the same strength, there must have been quite an appalling state of affairs in the tower after the substitution of plates for the coke.

It is in the condensation of the furnace gases that the great value of coke will always make itself felt, and there no Lunge plates will ever compete with a rational coke packing.

Mr. MARTYN thought that a careful comparison of the relative merits of the two systems of condensing hydrochloric acid must result in a pronouncement in favour of the coke condensers. The points for comparison in each case were, firstly, the original cost of the plant; secondly, the relative efficiency of the systems; thirdly, the expense of maintenance and depreciation; and lastly, the cost of the supervision. As four plate towers were required to do the work of one coke tower, the first cost of the system could be but slightly in favour of the plate towers. With respect to efficiency, he (Mr. Martyn) thought that coke towers would be hard to beat. As a coke tower would last some 30 years, the item for maintenance and depreciation would naturally be less than in the case of the plate towers, subject, as the latter would be, to very great variations in temperature. Naturally also four plate towers would

require more supervision than the single coke tower, and though for some purposes plate towers might be found to be peculiarly suitable, yet it was unlikely that they could be generally substituted in this country for the coke condensers.

Dr. BRIDGES pointed out that the large cooling space through which the hot gas had to pass before reaching the plate tower must contribute largely to the efficiency of the system. He should like to know whether the effect of externally cooling the gas by a water spray had ever been ascertained.

The CHAIRMAN, replying to Dr. Bedson, said that though the effect of a water spray in actual contact with the hot gas had been tried and was known, he had never heard of the external application of water merely as a cooling agent.

Dr. LUNGE replies by letter as follows:—

An advance copy of Dr. Hurter's criticism on my paper on Practical Results of the Lunge-Rohmann Plate-Towers having been sent to me by the Hon. Secretary of the Newcastle Section, I beg to reply to it herewith.

Dr. Hurter estimates the gas space in front of my towers at 1,000 cub. ft. for pan and muffle combined, and he contrasts this with the "wonderful results" alleged to be obtained with 74 cub. ft. of plate room, hinting that the space in question really does the greater part of the work. I can satisfy him on this point. The pipes, empty towers, and bombonnes in front of my towers (except the last six for each set) were provided by the desire of the owners of the works, and acted entirely as cooling space, such as the great majority of English and other alkali works provide in front of their proper condensing apparatus. I never asserted that this cooling space could be diminished when adopting my system; I always thought it very irrational to neglect a thorough preliminary cooling of the gases, in full accordance with the views of the late Dr. Angus Smith, although perhaps not with some modern theories and practices which I cannot profess to refute on this occasion (it is done in Vol. II. of my "Sulphuric Acid and Alkali," which is to be published shortly). This cooling space at Duisburg condenses most of the sulphuric acid, but only a few per cent. of the HCl.

The six bombonnes (66-gallon receivers) in front of the plate-tower, as is clearly proved in my paper, served almost entirely for equalising the unavoidable variations of work. I quote the absolute figures from my paper, only converting the specific gravities into degrees Twaddell: acid running directly from the pan-tower 29.6° from the muffle-tower 29° after having passed through the six bombonnes, 32° and 32.6° respectively. That is all the work they did in connection with my towers, whereas Dr. Hurter knows very well that those continental works which condense in bombonnes generally use a hundred such for a pan and furnace with a coke-tower at the end. I have from the very first demanded that a few such receivers should be placed in front of my towers: 1st, to equalise the variations of work; 2nd, to bring up the strength of the acid to that required for sale; 3rd, to serve as an acid store, exactly as is done in the case of ordinary coke-towers; the only novelty in the Duisburg apparatus being that as few as six receivers of 66 gallons each have been found to be sufficient, whereas I formerly thought a few more were required for the above purposes.

Mr. Lasche's paper shows that the coke can be entirely dispensed with by providing a better dividing apparatus at the top of the tower.

Dr. Hurter makes a calculation according to which—judging from the strength of the acid flowing from the coke on to the plates—the coke, amounting to about 50 cub. ft. for the pan and muffle together, would have done 74.7 per cent. of the whole condensing work, the plate space 11.8 per cent., and the bombonnes 13.3 per cent.

My explanation was originated by Mr. Lasche, and proved to be at least partially correct: that the result obtained after removing the coke was not, as Dr. Hurter says, "very bad indeed," but 170 parts acid of 32° to 100° of salt, which is probably superior to the majority of works; that by adding a few more plates the yield, without any coke, rose



to 17.8 acid of 32, which is probably superior to the *bona fide* results of nearly all other alkali works, although Dr. Hurter composedly still calls it "bad"; that the 15 bottom plates in my towers, with 12-mm. holes, were practically inactive, and Mr. Lasche considered that if, in lieu of them, plates with 7-mm. holes had been present, the coke would have been entirely useless; that the exit pipes were only a few inches wide, and any gas tests taken in them cannot for a moment have been compared with the English alkali inspectors' chimney tests; that the owners of the Duisburg works are extremely satisfied with the results obtained, and have put up another plate-tower set, undaunted by the "appalling state of affairs" which Dr. Hurter says must have existed.

1. Dr. Hurter says: "As far as the managers at Duisburg went in the substitution of plates for coke, the results are decidedly in favour of coke, and they are alive to that fact." That sounds, and evidently is intended to sound, as if the Duisburg people had generally convinced themselves that a coke-tower is superior to a plate-tower, the truth being that they do not dream of employing more than that trifling quantity of coke space (about  $\frac{1}{3}$  of an English coke condenser) above the plates, as is distinctly said in Mr. Lasche's paper, and that the managing owner at the last meeting of the German Society of Chemical Industry, loudly declared it would be simply foolish not to apply my system in future.

2. Dr. Hurter says: "100 parts of rock-salt yielded in the shape of liquid acid—

	Per Cent. of pure HCl.
(1) When working with coke.....	58.8
(2) " " without coke.....	32.6
(3) " " with plates instead of coke..	55.6

The first and third figure are right: the first representing an actual yield by my system of 96.2 per cent. of the theoretically possible yield from 98 per cent. rock-salt. But for No. 2 Dr. Hurter has chosen the figure obtained at the very first trial after removing the coke, and he glides over the fact that the yield at once rose to 52.4 per cent. pure HCl by simply improving the distribution, exchanging some plates for others with narrower holes, and adding one plate for the better distribution of the water, but before putting in the additional ten plates intended to replace the coke.

3. Dr. Hurter says: "If on the top of the 6 ft. of coke another 6 ft. had been placed, the disappearance of the large bulk of HCl would have occurred in the upper 6 ft., and the condensation of the lower 6 ft. of coke would have been a very small fraction, and not as before, 75 per cent." My readers will probably think that I must have misquoted Dr. Hurter, in crediting him with the assertion that the large bulk of the HCl is not condensed in the lower part of a coke-tower, as the world had hitherto believed, but in the last 6 ft. of coke at the top; but I have quoted verbatim from the copy supplied to me, and I would rather not weaken the effect of that passage by any comment of mine.

In reply to Mr. Martyn, it is scarcely correct to say that four plate-towers are required to do the work of one coke-tower. There is only one small plate-tower for the pan and another for the muffle, and the cost of maintenance for these is no greater than for coke-towers. Nor is there any reason why the item for supervision should be greater in the one case than in the other, the small size of the plate-towers making them more easily accessible and manageable than coke-condensers.

In concluding the meeting the CHAIRMAN said that he wished to express his regret at the great loss the Section had sustained owing to the removal of Dr. Dunn to another part of the country. During 14 years he had rendered invaluable service as honorary treasurer and secretary to the Section, and at a special meeting of the Section shortly to be held, the members would have an opportunity of expressing in a material way their sense of the importance of these services.

## Scottish Section.

Chairman: Robt. Irvine.

Vice-Chairman: J. Clark.

Committee:

G. Beilby.	J. S. Macarthur.
R. Cox.	E. Ostlere.
C. J. Ellis.	T. L. Patterson.
C. A. Fawsitt.	R. Pullar.
D. Harris.	J. B. Readman.
G. G. Henderson.	H. Rose.
R. A. Inglis.	E. C. C. Stanford.
W. I. Macadam.	D. R. Stewart.

Hon. Secretary and Treasurer:

J. Stanley Muir, Chemical Laboratory, University of Glasgow.

SESSION 1894-95.

Dates of Meetings.

Glasgow.—Philosophical Society's Room, 207, Bath Street. January 8th; March 5th; May 7th.

Edinburgh.—Philosophical Institution, 4, Queen Street. December 4th; February 6th; April 2nd.

Meeting held in the Philosophical Society's Rooms, 207, Bath Street, Glasgow, on Tuesday, November 6th, 1894.

MR. R. IRVINE IN THE CHAIR.

### CHAIRMAN'S ADDRESS.

I HAVE to thank you for the honour you have conferred upon me in appointing me Chairman of the Scottish Section of the Society of Chemical Industry. It will be my earnest endeavour to uphold and increase its position and influence for good.

The able presidential address, delivered by Mr. E. C. C. Stanford at the Edinburgh meeting last July, must have impressed all who heard it, or have since read it in our Journal, with the vast and far reaching horizon of the work in which the Society of Chemical Industry is engaged; the beneficent influence it may have upon mankind in the present, and far more so in the unknown future of chemical science, as applied to its commercial developments.

Such an address deserves a distinguished place in the annals of our Society.

While entirely agreeing and sympathising with his views, it is somewhat trying to have to meet you in the emasculated condition the comprehensiveness of such an address leaves me. Therefore, instead of following the usual rule of confining my remarks entirely to the enumeration of new chemical creations during the past year, I may be excused if I occupy a part of your time in recalling some pleasant reminiscences in connection with our meeting in Edinburgh, where many old friendships were renewed, and from which many new and pleasant acquaintanceships originated.

Although Edinburgh and its immediate vicinity cannot claim to be a centre of chemical industry, her principal manufactures being alcoholic liquids, chloroform, and drugs, paper, printing-ink making, &c., still considering the interesting nature of the ancient town, with its beautiful surroundings, the hospitality of its magistrates and citizens (which was only bounded and controlled by the absurdly short time to which the meeting was confined), I am sure that visit must have come to many weary chemical souls as a pleasant change.

It was unfortunate that owing to the small-pox scare the attendance of members at this meeting was comparatively small, doubly so, considering the complete nature of the preparations made by the acting committee for the comfort

and convenience of a much larger number of guests; which arrangements, at the last hour in many cases, had to be entirely altered to suit the altered circumstances. Railway, coaching, and other arrangements were all dislocated, and general confusion must have resulted had it not been for the energy and administrative ability of the local secretary and treasurer.

The fact came home very forcibly to the committee that the three days set apart for the annual meeting were quite insufficient for the programme they had prepared.

Everything had to be so nicely timed that there was no room for expansion in the arrangements when any hitch arose. When next we welcome the Society to Edinburgh we must do our best to have the time extended so as to give members a reasonable opportunity of realising that the amenities of the town are not confined to refuse destructors, breweries, distilleries, &c.

As part of our programme, and in order that our guests might see something of the venerable "Old Edinburgh" the committee arranged to have "personally conducted tours" through that portion of the town lying between the Castle Rock and Holyrood House, representing as it does in its many relics much of the ghostly past in Scottish history. I had the curiosity to inquire how many took advantage of this opportunity, and found that only two members had been able to perform this interesting journey. The university, law courts, industrial museum, theatres, cathedrals, clubs, and citizens generally held open hands and doors to the harassed members in vain. Every moment of their time was otherwise occupied. Engagements, represented by numerous varicoloured tickets demanding their presence at so many places at one and the same time.

It is unnecessary to do more than thus casually refer to the holiday aspect of our meeting, and if some difficulties occurred, or mistakes arose, causing disappointment to individual members, these were the result of unfortunate circumstances over which the "hurricane committee" (as our local secretary quaintly designated it) had no control.

Before passing to review some of the chief industries visited in our excursions, it may not be invidious, however, to make an exception by recalling our very pleasant excursion to Perth, where the Messrs. Pullar and the magistrates of that town treated us as their guests with such princely hospitality.

#### Shale-Ore Industry.

In a former number of our Journal the general processes as carried out five years ago at Broxburn Oil Works are described in a paper by Mr. D. R. Stuart.<sup>†</sup>

Through the kindness of Messrs. Kennedy and Henderson, managing directors of that company, I am enabled to lay before you a concise account of the latest advances in the processes and manufactures by which means that great company has been able, "in a manner," to hold its own in face of the overwhelming competition of American and Russian natural oil supplies; with valuable statistics of the past and present condition of the mineral oil trade in Scotland, accompanied by a *history* of this industry, to which I will first refer.

TABLE I.

#### HISTORY OF THE MINERAL OIL INDUSTRY.

1830. Ireland, Peat Co. incepted by Mr. Moir Crane. First paraffin for candles.

1848-50. Alfreton Spring. Worked by Jas. Young. 300 gallons a day. Object—lubricating oil. Playfair, December 1847, letter to Jas. Young.

1851. Young's patent. Torbanchill coal, or Boghead.

1862. Boghead coal exhausted. 1862 Robert Bell worked shale at Broxburn.

1859. Petroleum. Colonel Drake.

Scottish trade.

1st period, 1850-64. Young's patent.

2nd period, 1864-72. Development of petroleum—(in 1871 there were 51 works).

3rd period, 1872-78. Great struggle for existence in 1873 (21 works gone). Smaller works disappear. Production concentrated.

4th period, 1878-87. Lucrative. 25 per cent. dividend at Broxburn. New oil fever. Over production—collapse increased production of ammonia becomes important.

5th period. Combination period. 13 works.

TABLE II.

The following table gives details of the progress of this manufacture from 1871 to 1894:—

	1871.	1878.	1887.	1894.
	End of 1st Period, 51 Works.	End of 2nd Period, 18 Works.	End of 3rd Period, 13 Works.	
Shale.....	Tons. 800,000	Tons. 850,000	Tons. 1,800,000	Tons. 1,947,812
	Galls. 25,000,000	Galls. 26,000,000	Galls. 52,500,000	Galls. 48,600,000
Crude oil.....	11,250,000	11,400,000	21,680,000	26,452,341
Naphtha and burning oil and gas.	2,500,000	5,000,000	9,000,000	8,745,253
Lubricating oil.....	Tons. 8,500	Tons. 9,200	Tons. 22,800	Tons. 19,150
Paraffin solid.....	2,250	4,750	18,483	28,000
Sulphate of ammonia		Capital, £1,300,000	Capital, £2,000,000	

TABLE III.

SHOWING OUTPUT OF SHALE IN SCOTLAND AND PRODUCTS FROM 1873 TO 1891 BY R. T. MOORE, C.E.

Output of Shale in Scotland from 1873 to 1891 (from paper by R. T. Moore, C.E., 1892—Federation Institute of Mg. Engs.)

Year.	Tons.	Year.	Tons.	Year.	Tons.
1873	524,005	1880	759,777	1887	1,300,320
1874	560,910	1881	912,171	1888	1,052,292
1875	424,023	1882	934,487	1889	1,086,500
1876	541,273	1883	1,133,720	1890	2,180,183
1877	684,118	1884	1,469,049	1891	2,337,302
1878	645,950	1885	1,741,750	1892	2,077,076
1879	712,428	1886	1,900,144	1893	1,917,842

Net prices realised at works (from paper by R. T. Moore, C.E.).

Year.	Naphtha and Burning Oil per Gall.	Lub. Oil per Gall.	Med. Oil per Gall.	Seal. per Lb.	Sulphate of Ammonia per Ton.
1877	Pence, 9.275	Pence, 11.004	Pence, 6.668	Pence, 4.31	£ s. d. 17 5 6
1882	4.313	7.347	4.005	2.49	18 2 5
1887	3.160	2.200	1.060	2.125	19 5 0
1891	4.176	3.715	2.806	2.262	19 7 1
1892	3.400	3.005	2.250	1.750	10 0 0
1893	2.730	2.950	2.100	1.750	11 12 6

Although the figures from 1889 show the production of shale to be larger, the quality has been deteriorating so far as oil yield is concerned. There is a great improvement in the quantity of sulphate of ammonia from the newer methods at work.

\* There seems to be a discrepancy between Tables II. and III., as to the output of shale. I think the figures of Table II. may be accepted as the more accurate, looking at the output of crude oil.

<sup>†</sup> Vol. VIII., p. 190. February 28th 1889, D. R. Stuart.

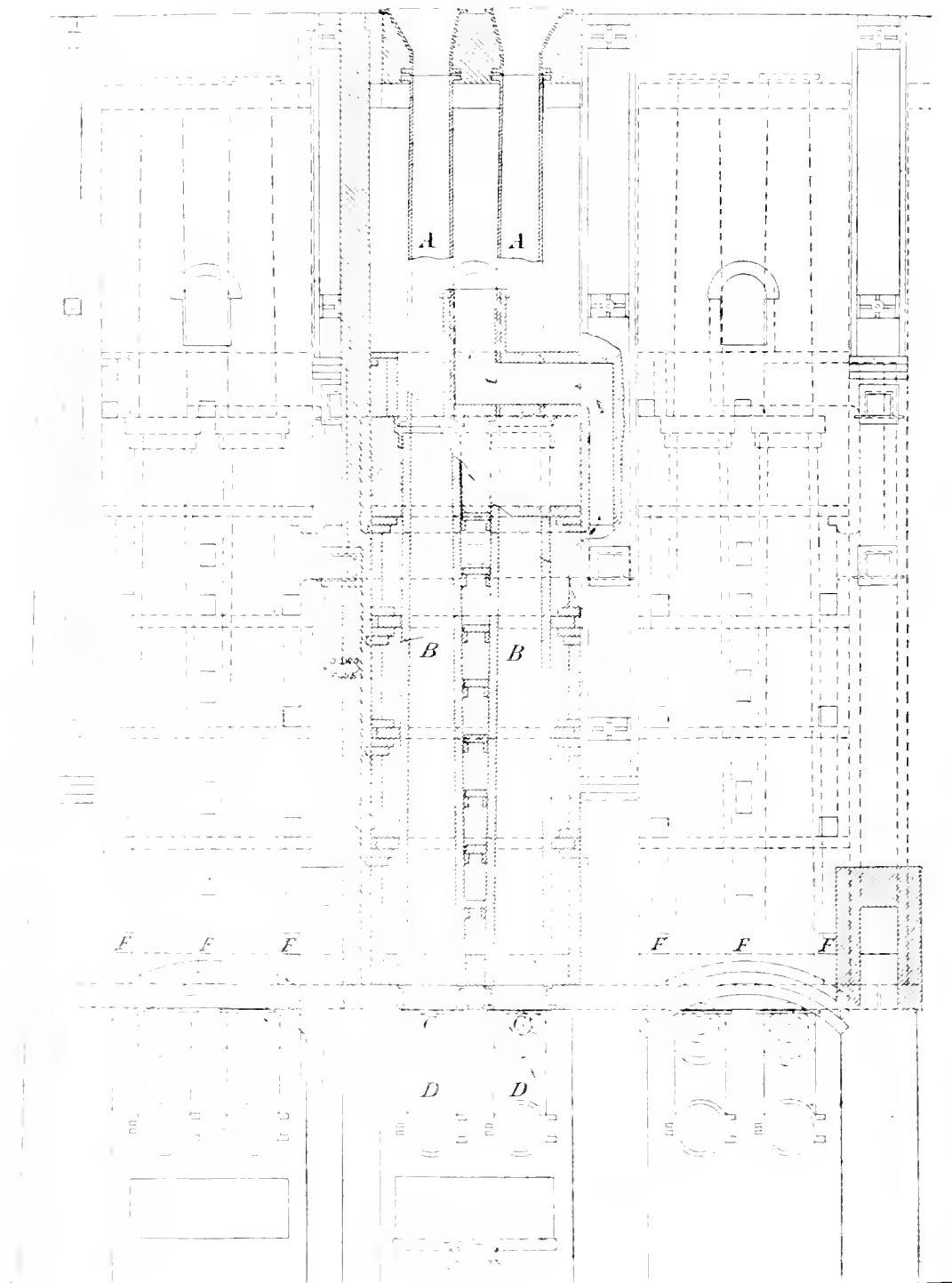
When first this great business was introduced the principal object was to obtain burning oil (then selling about 9d. per gallon), but competition, and more especially the flooding of the world's markets with burning oil from America and Russia, has now reduced the price of Scotch oil to less than one-third, namely, 2·730d. See Table III.

As has been the case with the coal gas and many other industries, so also in this, the residual products, which

at first had been considered of little importance, more and more came into prominence, and had to bear the burden of this reduction in prices, and consequently these have now practically taken the premier position as money earners for such Scotch oil companies as are now able to pay dividends.

In this respect the present position of matters at Broomburn is, that the sulphate of ammonia obtained from one

Fig. 1.

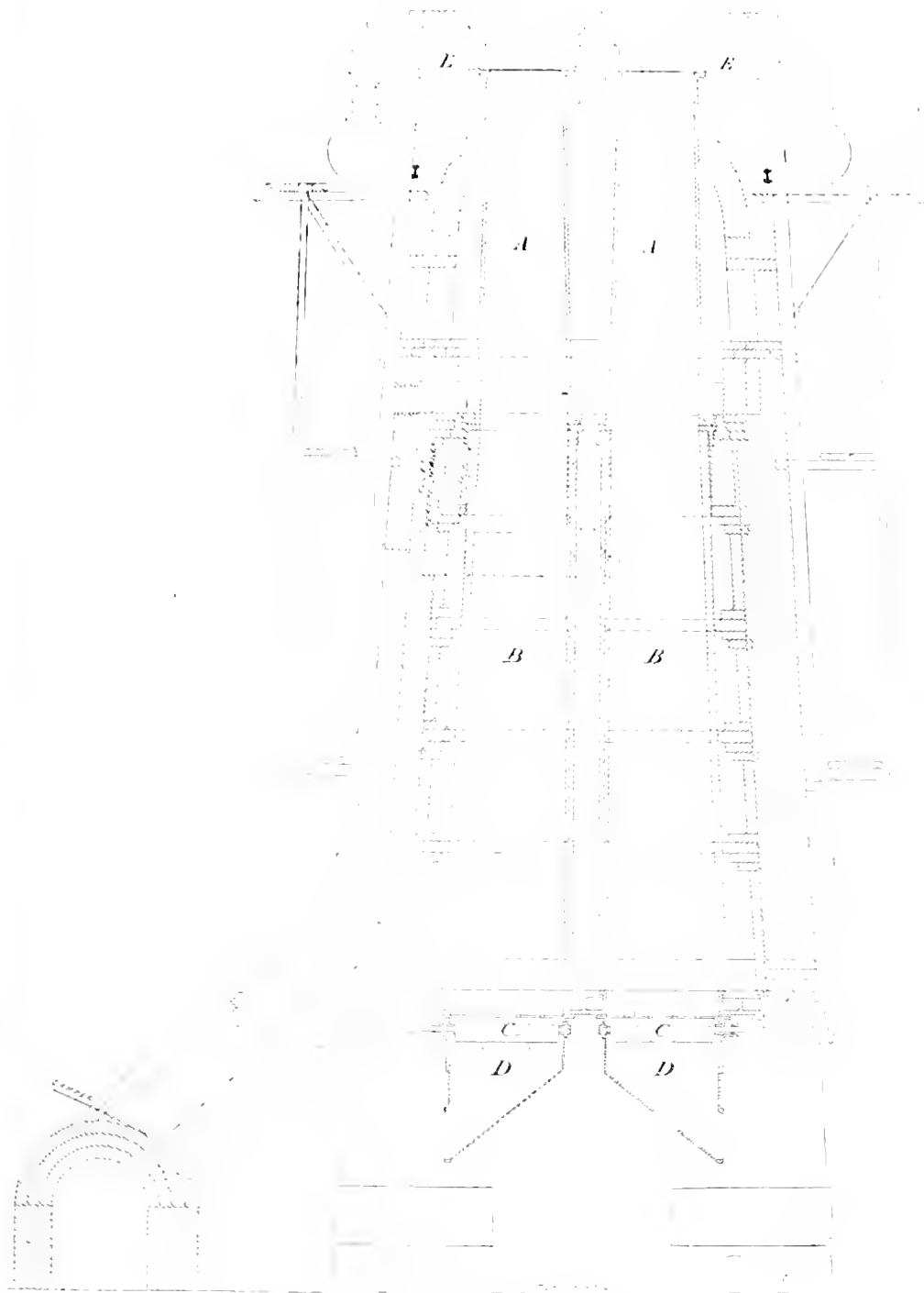


ton of shale (72 lbs. in weight) pays all expenses, including royalties, mining, and retorting, so that their crude oil costs them practically nothing, and consequently the shales, which in old days were considered too poor to work for oil alone, are now preferred, owing to the large amount of ammonia they produce.

Young and Bielby were the first to realise this important fact, and the success attending their investigations, and the practical results obtained by the use of their system of

shale distillation, must be patent to all conversant with the history of this manufacture, and I need not refer further to their process of retorting, as the following description of the new "Henderson retort" embodies their idea of a continuous distillation of shale, beginning at a low temperature, 200° F., and ending by exposing the shale char, from which the oil has already been extracted, to a very high heat, 1,500° F., in the presence of superheated steam.

Fig. 2.



HENDERSON'S IMPROVED PATENT RETORT.

At Broxburn Comp's Works we saw the new retort (patented by Mr. Henderson) in operation, and what must have struck all, with admiration, was the perfection of the apparatus, when appeared to do its work automatically, only requiring the guiding hand of man in trifling details.

TABLE IV.

DESCRIPTION OF DRAWINGS AND WORKING OF HENDERSON'S IMPROVED RETORT (Patent No. 6,726, A.D. 1889) FOR THE DESUBJECTIVE DISTILLATION OF SHALE, &c.

"The object of this improved retort is to get a larger yield of sulphate of ammonia without deteriorating the quantity and quality of the crude oil. The retorts are vertical, 28 ft. long. In the upper cast-iron portion of the retort A, the oil is first distilled off the shale at a temperature of about 900° F. The spent shale is then passed downwards into the brick portion of the retort B, which is kept at a constant temperature of about 1,300° F. The shale, in passing down through the retort, is kept in constant motion by a roller C at the bottom, which delivers it into a hopper D below, at the required rate. This continuous motion largely increases the yield of ammonia and permanent gases from the carbon of the shale; it also regulates the working of the quantity of shale to be put through, and prevents the exhausted material from fusing and sticking in the retort under the higher temperature. All the products of distillation are drawn off the top of the retorts through the branch pipe E.

"The retorts are heated with gas made from dross coal made in gas producers, and introduced at the ports F. The incondensable gases are also returned to the heating chambers, and mixed with the producer gas to assist in heating the retorts.

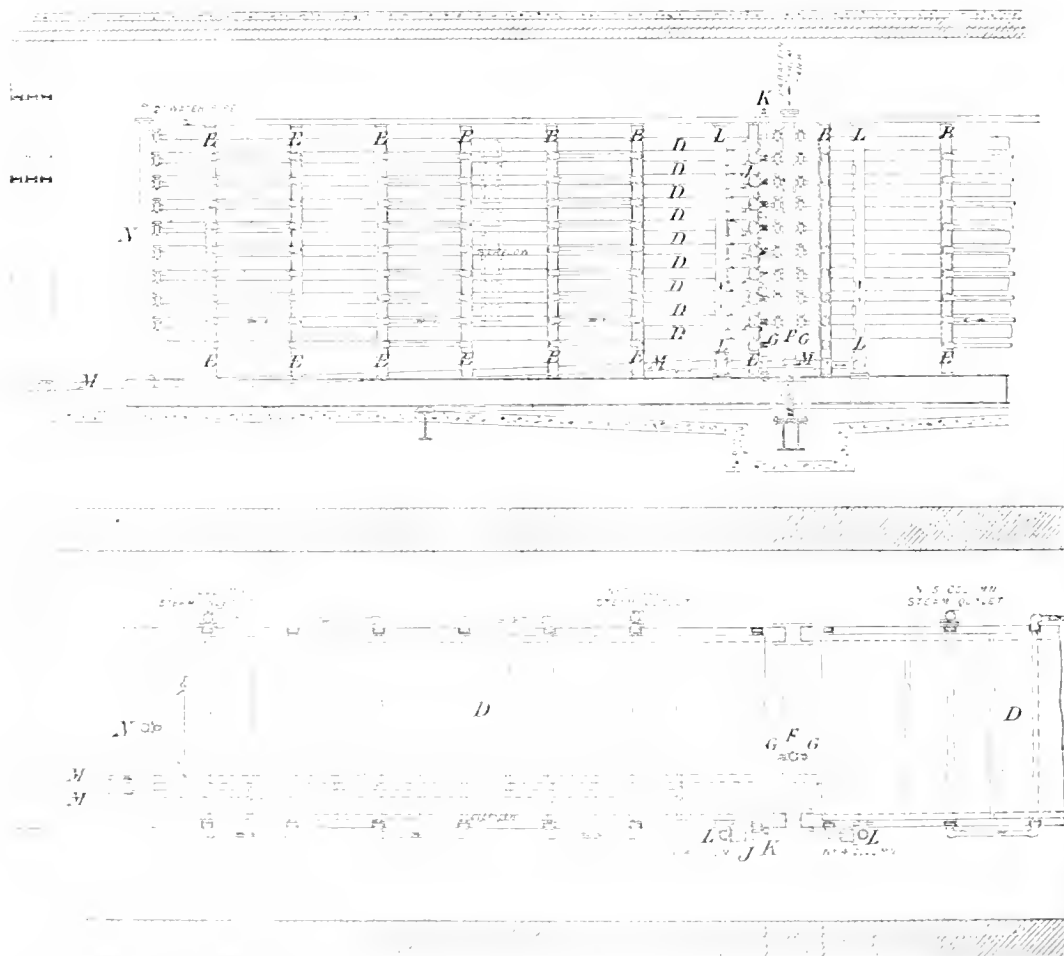
"On the tops of the retorts are fitted large malleable iron hoppers, capable of holding about 18 tons supply of the raw shale. These have been found to be of great advantage in the regular working, in dispensing with all night and Sunday labour.

"The above retorts are now adopted by the Broxburn Oil Company, Limited, and have proved most satisfactory, giving the maximum yield of products with the minimum of labour."

*Comparison of Past and Present Retorts, as worked at Broxburn.*

"The old Henderson patent retort of 1873, gave, with the Broxburn shale, about 31 gallons crude oil, 15 to 17 lbs. of sulphate of ammonia, 75 per cent. spent shale containing 12 or 13 per cent. of combustible matter, which was dropped hot into the furnace below for fuel; and about 2,000 cubic ft. of permanent gas. With the present retort, after extraction of the crude oil, 31 gallons to the ton, the spent shale passes into the brick retort below to be acted on by steam at a high temperature. This gives, for fuel, gas (like producer gas) of about 15,000 cubic ft. per ton of shale, and the sulphate of ammonia is increased to 44 lbs. per ton. With Drumshorland shale at the Broxburn Oil Company's

Fig. 3.



Roman Camp Works, the crude oil got is about 20 gallons per ton, and sulphate of ammonia about 72 lbs. Spent shale contains 26 per cent. of alumina, equal to about 14 per cent. of aluminium."

From the hurried nature of our visit to Broxburn, it was difficult to carry away a clear idea of the various processes now in use there, but all must have been especially impressed with the wonderful ingenuity displayed by Mr. Henderson in his new adaptation of the old sweating process for the purification of the dirty looking crude paraffin scale, into the pure translucent wax, entirely devoid of colour or smell, and directly fitted for the manufacture of candles. Subjoined are detailed explanations of the new system.

### Purification of Paraffin Wax.

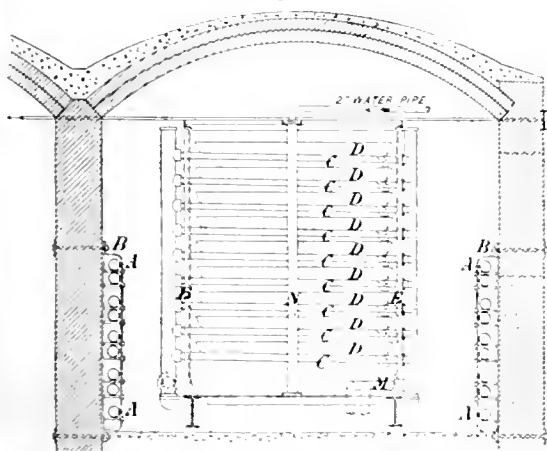
#### DESCRIPTION OF WORKING.

"The crude solid paraffin from the filter presses, after being melted in a tank, is pumped direct through the vertical charging pipes and through the small cocks into the horizontal trays of the sweating apparatus."

"Before charging the trays, the diaphragms are covered with about half-an-inch of water from the small cocks on the charging pipes N which prevents the melted wax from running through the diaphragms, when the trays have been charged, the crude paraffin wax is allowed to congeal, then the water is run off from the bottom of the trays through the same swivel nozzles into its own receptacle. The chamber is now shut up and the suitable heat applied. Complete arrangements are made at the end of the horizontal pipes M for changing the various products into their own receptacles; beginning with the water, then with the various grades of paraffin, up to the refined wax. This can be regulated to any grade of paraffin required."

"This process has been largely adopted, and is now the only one used in the Broxburn Oil Works, being a great saving in labour over the old methods. In the old method of working, the crude paraffin wax was passed from the filter presses and further put through hydraulic plate presses, which entailed much more expense in machinery and labour than are necessary with the above improved process."

Fig. 4.



LONGITUDINAL ELEVATION AND CROSS SECTION OF APPARATUS FOR TREATING AND PURIFYING PARAFFIN WAX. (Patent No. 11,799, A.D. 1891.)

"The drawings accompanying this description show an apparatus capable of refining 14 tons crude paraffin wax at a time, and which can be charged from 2½ to 3 times a week according to circumstances."

"The sweating apparatus is arranged in a horizontal elongated chamber about 52 ft. by 13 ft. by 10 ft. high constructed of brickwork with a suitable roof, and fitted with pipes heated by steam. Besides the heating pipes A,

ranged along the sides of the chamber B, there are transverse heating pipes C, which also serve the purpose of carrying the trays D, being fitted in vertical standards E. The charging pipes F are fitted with cocks G. In the bottom of the horizontal trays D there is fitted a wire gauze diaphragm H. These diaphragms are fixed with bolts to the ends of the trays, and rest on cross bars I. The bottom of the trays communicate with short pipes with swivel nozzles J, constructed to work with worm wheels from worms on a vertical shaft K, this arrangement allowing all the swivel nozzles from the bottoms of the trays to be turned at once."

"The various liquids separated in the refining of the wax are drawn off through the swivelling nozzles into cups or hoppers on the standard pipes L, which lead them into horizontal pipes M, through which they pass to suitable receivers for collecting the various qualities of products."

The history of this oil industry is an object lesson. Not only so in respect to the civilizing influences it has had upon the mass of the people of the world, but of the unattainability of brightest hopes and prospects of those engaged in it.

As regards Scotland, we had her oil companies paying till within a few years from now, splendid dividends to their shareholders; then the clouds of misfortune descended, dividends ceased, and most men would have given up the unequal battle in despair, in place of which, however, skill and indomitable perseverance have overcome difficulties which seemed insurmountable, and with the improved processes now at work there seems a fair chance of a return of prosperity.

### Flash-point of Mineral Oils.

If I refer to the vexed question of the flash point of mineral oils used for burning in lamps, I do not seek to complicate a question which is at present "sub judice."

The subject is beset with a variety of complications which affect great commercial interests, but upon this part of the problem of course I have nothing to say.

Taking the question in its utilitarian aspect there can be no doubt that much of the oil sold in this country for household use is of an unsafe nature, and the *petroleum death rate* now ranks with other forces inimical to the human race.

Much has been, and is being written upon this important topic, but the supineness on the part of the public, coupled with the general want of interest in this vital question, makes it careless as to an evil and danger which happily only comes home to comparatively few.

Outside the Government commission, and apart from petroleum party confederations, the public should take up the matter and only buy and use such oil as is practically safe, and if insurance companies, who suffer so much from fire losses directly caused by the use of low flashpoint oil, were to bar the use of dangerous illuminants, this, to them, ever present danger would soon disappear.

Trifling causes generally sway the public taste, and the less unpleasant smell of Russian and American burning oils, which can be had at a cheap rate, keep them in favour, in defiance of any risk attending their use.

In this respect, with safe Scotch burning oil prepared from shale, the comparison is unfavourable. I venture to suggest that efforts should be made to overcome this sentimental (?) objection, and I have no doubt that sweet-smelling Scotch oil, with a flashpoint of 100° F., would then be preferred to the more dangerous varieties.

If the Scotch maker can accomplish this (and will do it), the Russian and American producers would in self-interest be bound to follow their excellent example by only selling safe oil. In America I understand the 100° F. flashpoint oil is in general use. Of course, there are many imported oils perfectly safe, but prices for such are high. The whole question relates to the oil supplied to the poor—not to the rich. Of course, opinions differ as to the cause of lamp explosions—Lamp *c.* Oil here—Oil *c.* Lamp there—one side believing that badly constructed lamps make it unsafe to use any mineral oil however high its flash point may be: the other side arguing, with some justice, that bad oil will be unsafe in lamps of the best construction.

Are we in Scotland not making too much of this low *v. g.* flash-point question? Regarding the above circumstances should we not rather try to meet the difficulty of dealing with low flash-point oils by a change in the construction of the lamp in which such oils are burned?

As presently constructed almost all of these lamps consist of a metallic burner and fittings, supporting the ink communicating directly with the oil reservoir, upon which the burner rests; only one or two inches of space intervening between a flaming heat of 1,000° F. and a considerable quantity of a very inflammable liquid.

Is it impossible to construct a lamp with the burner 6 or 8 inches above the oil reservoir (which could then form a convenient base for the lamp-stand), and thus remove the real source of danger, or let the oil reservoirs be the same distance horizontally from the burner?

#### Oil Gas.

At Perth Gas Works, the latest adaptation of the process for gas enrichment was seen in operation, by means of the addition of mineral oil gas to coal gas.

By this means the illuminating power of poor gas is raised to a degree beyond what would be expected taking the "*Light Values*" of both into consideration. This is a matter now becoming of great and essential importance, owing to the fact that most of our valuable and high-class qualities of *gas coal* are exhausted. The large supply of mineral oils now so low in price provide not only a cheap material for the enrichment of poor coal gas, but also (as practised in America) render non-illuminating water-gas (so called, obtained by passing steam through incandescent coke) of value as an illuminant.

There can be no doubt that this process which is a simple and economical one, will be universally adopted in the future and in many cases, this high-class gas will hold its own with its charming rival the electric light.

#### *Remedial Measures for purifying Water in connection with Paper Mills.*

At the mills of Messrs. A. Cowan and Sons and J. Brown and Co., Penicuik, the members had an opportunity of studying in detail the "Remedial Measures" now in common use in most paper mills in the vicinity of Edinburgh, especially those of the "Esk Waters" for the prevention of pollution.

These measures have been so successful, and the fouling of the water of these rivers has been reduced to such an extent that we may soon expect them to be teeming with trout (and even salmon), and a return to the condition of "fifty years ago" (before esparto boiling was dreamt of), when the disciple of Isaac Walton practised their gentle craft.

Dr. Stevenson Macadam, a prominent member of our society, may well be proud of his share in this great improvement.\*

The following details will be of interest not only to this Section but to the members of the Society generally. They apply to all the paper mills situated on the North Esk River, in the neighbourhood of Edinburgh, by whom they are strictly observed.

It is pleasant to reflect that such an agreement has been arrived at between the riparian proprietors and the paper-makers by which not only has the river water been rendered comparatively pure, but at the same time to the profit of the manufacturers, who have adopted the plans formulated by Dr. Macadam.

It is to be hoped that before long the conciliatory policy, which has in this case actuated the parties interested, may become of general adoption, not only for the prevention of this, but of all other forms of river pollution, which at the present day is a disgrace to our civilization.

The following are the rules observed by the paper-makers on the North Esk:—

"The general principles which regulate the mode of dealing with the impure discharges from the paper mills on the river are applicable to all the mills under the agreement,

but are sufficiently elastic to admit of considerable variation in the mode of working, so as not unduly to interfere with the individual methods of manufacture, or with the special arrangements of each mill. The separate schedule of regulations issued under the agreement to each mill is intended for the guidance of that mill alone, and may be regarded as the special method of carrying out the necessary preventive processes for dealing with the polluting materials.

The general remedial measures applicable to the whole mills on the river may be summarised as follows:—

1. All isparto boilings and second waters being first coolings go to the roasters, and none flow, directly or indirectly, through the ponds or otherwise to the mill lade or the river.
2. All other esparto cooling waters and stronger steeps, whether re-used or not, go ultimately to the roasters, and none pass, directly or indirectly, to the lade or the river.
3. All weaker esparto steep waters containing at the maximum not more than 200 grains of total residue in the imperial gallon, and on the average not more than 100 grains of total residue, and not more than five grains of alkalinity calculated as hydrated caustic soda in the imperial gallon, may be discharged through the pond system to the general outflow, provided no washing waters from the esparto breaking engines are passed away from the same mill.
4. Where no steep waters are discharged through the pond system to the general outflow, then washing waters from the esparto breaking engines may be sent through the ponds and general outflow instead thereof, provided these washing waters conform to the standard of quality given in paragraph 3.
5. When rags are used in esparto mills the boilings at least go direct to the roasters, and coolings, either directly or after re-use, go also to the roasters. Steeps from rags and washing waters from the rag-breaking engines may go through the pond system to the general outflow.
6. All spent bleach liquor to be re-used as much as possible, and the residual liquid may be sent through the pond system to the general outflow.
7. Presse-pâte waters and ordinary machinery waters to be re-used as far as possible, but when discharged to go through the pond-settling system and thence to the general outflow.
8. All discharge liquids flowing from the ordinary working operations of the mill to pass through the settling pond system and by a common outlet to the lade or river, and the *bona fide* combined effluent shall not be diluted with extra water at any stage, and shall not contain more than 100 grains of total residue in the imperial gallon. It should be neutral, but if alkaline, shall not exceed alkalinity equal to five grains of hydrated caustic soda in the imperial gallon of the discharge.
9. All drainings from the pond sludge ash filters to flow by a visible outlet to the lade or river, and shall conform in quality to the standard given in paragraph 8 for the general overflow.
10. Spent bleach sludge, settling pond sludge, causticising lime, ashes, founds of buildings, and other rubbish not to be deposited on the banks of the river or lade, or within reach of the water at any time.

The discharges from all the mills are repeatedly inspected during each week, in some cases every day, and occasionally several times in the day. Whenever the appearance of the outflow, or its alkalinity, or its effect in the lade or river indicate that the discharge is unduly polluting, then a sample is taken, and on subsequent examination a number of these samples are selected for analysis. The results prove that the discharges from the mills under the agreement practically fulfil the conditions of paragraph 8, alike as to total residue from the imperial gallon and as to alkalinity. The discharges which fail to come within the

limits of paragraph 8 are comparatively few, and are found to be unduly polluting from observed inattention to the practical remedial measures.

There can be no doubt, therefore, that with the employment of properly constructed remedial measures and with ordinary care and attention in the working of such, there need be no difficulty in keeping the quality of the discharge from any paper mill well within the given standards."

#### *Treatment of Town Refuse.*

To attempt to master even the literature (if I may use such an expression) of the sewage question would be almost as hopeless a task as the practical treatment of the subject has proved to be. Apparently only nature can regulate this vast volume of waste (?) material in her own economic ways. That great brooding mother—the ocean—into whose bosom the "waste matter" of the world is carried returns to us what we think we have lost. Thus, literally, we cast our bread into the waters, and it returns to us in the liberal food supplies she affords. With solid refuse of towns it is otherwise, farmers will not buy it, and we now burn it—sometimes not in a very skilful manner—in what we truthfully, and to our shame, call destructors. All the valuable nitrogenous matter, tar, &c., goes up the chimney, often to the annoyance of the public. Why is the refuse not retorted in the same manner as shale is, and the ammonia saved? Surely as much sulphate of ammonia would be obtained from town refuse as would make its treatment profitable instead of a burden upon the town rates.

#### *Gold Extraction.*

The importance of the increased production of gold during the last few years must have startled all who have watched the development of the gold getting industry, more especially in Australia and Africa.

Alluvial deposits have long since been practically worked out, and it is now exceptional to find gold in mass. The more troublesome and costly, although perhaps more certain, method of extracting the metal from its original matrix (of quartz or other rocks), or from the heaps of waste material—the residues of processes formerly in use and known as tailings—by chemical in place of mechanical means has given rise to a variety of new methods, some of which are now in successful and extensive operation, in gold bearing districts.

The well known "chlorination" process is suitable for certain classes of ores and for these is in common and profitable use, but in many cases, especially where the ore is of a basic nature, much chlorine is lost as it decomposes and combines with the basic matter before combining with the gold.

By far the most important and successful application of the many chemical processes for the extraction of gold, and especially suitable in the case of even the poorest tailings and most obdurate ores is the "cyanide of potassium" method, known after its introducers as the Macarthur-Forrest process. This may be termed a development rather than an invention, for it has long been known that gold was readily soluble in (or would combine with) solutions of alkaline cyanides. Be that as it may these gentlemen have succeeded where others have failed in developing what was merely a laboratory reaction into a profitable and important new industry.

They employ a very weak aqueous solution of cyanide, *about one tenth per cent.*, 2 lbs. of the salt being usually sufficient to dissolve the gold in one ton of ore or tailings, and this by simply filtering the cyanide solution through the powdered material. The lixivium so obtained is passed through filter boxes filled with the finest zinc shavings where the gold is deposited as a brown mud, the cyanogen which held it in solution passing away in combination with the zinc. Thereafter this mud is melted into bullion.

The following figures show what rapid progress this process has made in the comparatively short period of three years, and it seems to me if such a splendid advance continues to expand to its legitimate end we shall have in the future what will be equivalent to the discovery of such new sources of wealth as astonished the world in the

middle of this wonderful century—and what happened then as the result of great additions to our gold supplies will unquestionably hold good in the near future with the *additional supply of gold; land, and its animal, vegetable, and mineral products* will again rise in value, and the abnormal conditions, which to many in the past have suggested a bimetallic standard as the only way out of the scarcity of our real standard metal gold, will cease. Consequently wages will rise and commerce will be conducted on more sound and legitimate lines. This is not the language of romance. One company *alone* (to quote from actual figures, see below) if they continue their operations on the lines of the last three months will by means of the Cyanide Recovery process add nearly three million sterling per annum to the gold supply of the world.

The African Gold Recovery Company (Limited) announce that 55,000 ozs. of gold have been recovered at the Randt, and 7,100 ozs. in other districts, a total of 62,100 oz. during the month of September, by means of their Macarthur-Forrest cyanide process. The August total was 64,258 ozs., and that for July 57,500 ozs. For the past three months (the first three months of the company's financial year), the amount of gold obtained is 183,858 ozs., as against 102,390 ozs. for the same period of last year. The following are the details monthly to date since the company began operations:—

	1894.	1893.	1892.	1891.
	Ozs.	Ozs.	Ozs.	Ozs.
January.....	51,000	17,500	9,700	..
February.....	51,500	16,963	10,366	..
March.....	56,500	20,000	11,700	..
April.....	56,500	20,050	13,500	..
May.....	57,850	24,000	13,500	..
June.....	58,000	27,100	16,045	..
July.....	57,500	20,300	16,451	..
August.....	64,258	56,000	16,648	..
September.....	62,100	37,000	16,986	..
October.....	..	38,000	17,771	3,300
November.....	..	40,310	17,305	5,732
December.....	..	44,200	18,000	8,300

The enormous "placer" finds of gold during the period of 1850 to 1870 reached 30,000,000*l.* to 40,000,000*l.* sterling per annum, but these have now given place to a later yearly average output of 15,000,000*l.* to 20,000,000*l.* since that auriferous period gave out. Taking the later figures to represent the actual gold output of the world since then, we see the one Gold Company, quoted above, giving to the world from one-fifth to one-seventh of this whole amount; and that principally recovered from hitherto useless waste or refuse material.

By the application of the Macarthur-Forrest process to fresh ore (presumably richer in gold than these waste heaps of already partially exhausted material) we have reasonable grounds to infer that, apart from finding sporadically rich-bearing districts, the future gold extracting industry will yield to the world a very largely increased amount of bullion.

The successful adoption of the Macarthur-Forrest process has increased the demand for alkaline cyanides. This in turn has revived the old dreams of its preparation from atmospheric nitrogen—the El Dorado of many sanguine chemists,—but so far nothing has been done in finding a practical and profitable process. How soon success may attend these efforts, who can say?

A fresh source of the salt had, however, to be found for instant requirements as the output from the old "Prussiate"

\* We may be nearer a higher range of prices than many think.



process and from gas residual products was insufficient. To meet the new demand Mr. George Beilby, one of our distinguished members, with much patient work and great chemical ability has, I am informed, succeeded in preparing this substance on a practical scale in a condition almost chemically pure. For obvious and the best of reasons, my own ignorance, I cannot go into particular details: I can only thus briefly refer to his successful work by stating that he uses ammonia as the source of nitrogen in this new manufacture. Many others are working on the same lines, but as yet there seems to be little practical success outside his process. The field, however, is a wide one, and the workers are many, and we may any day hear of the discovery of even a simpler process than that invented by Mr. Beilby.

### *Coal Strike.*

This Section of the Society cannot fail to be interested in a result arising out of the late coal strike in Scotland.

Lamentable as its effects have been, still it has taught, or perhaps brought into greater and more pressing prominence, what most of us hitherto but dimly realised, viz., the superior value, as a fuel, of English as compared with the generality of Scotch coal. From a number of tests conducted on a large scale, my experience is that for steaming and heating purposes, quantity for quantity, four-fifths of English (Newcastle-on-Tyne) is equivalent to five-fifths of ordinary good Scotch steam coal. Prices being equal, therefore, Scotch coal is about 20 per cent. less in working value.

It must be a sorrowful reflection to the Scotch miners that English coal has now partly taken the place (in many instances with profit) of Scotch coal, and that such an anomalous condition of matters is not unlikely to continue. Also the coal fields of America, India, Australia, China and Japan, are gradually but surely cutting off the home markets.

There are many other matters it would be important to notice which I must pass over, but I cannot let slip this opportunity of addressing you, without advancing some personal opinions as to our procedure as a Society. Our Journal gives such full reports of the papers read at the several meetings of the Sections, and the discussions consequent thereon, that I need not make any special reference to our past work. But, looking forward to the session just begun, I would earnestly urge upon members the necessity of regular attendance at the Section meetings, and also in taking an active interest in the great object the founders of our Society had in view when it was established, namely, the communication of original papers, and thus diffusing useful information to our members.

But here a difficulty I have long recognised and to which I have before called attention comes into prominence. The interests of the Society corporate may often and naturally do clash with those of its individual members, in so far as inventors of new, or, of improvements in details of old processes, which it may be impossible properly to secure by patent rights, cannot be expected to lay the results of their hard earned work, by communications which our Journal thus lays open to the world over which it circulates. From such we cannot hope to have papers.

Young men fresh from the laboratory, a daily increasing host and for many of whom there is at present little or no scope, when engaged to act as work's chemist are generally bound down by agreement (legal or otherwise) not to disclose the processes they are engaged to watch or improve.

Work's managers are generally in the same position with the additional burden that they have to provide dividends for the companies they manage, so that the temptation here is all the other way. So it very much comes to this that the papers read before our Sections, with some bright exceptions, only relate to general matters, or are merely the description in bare outline of some chemical process with all the useful details necessary to success, carefully left unnoticed. Or they may be merely a rechauffé of old material in an encyclopaedic form. Or we may have sometimes wild schemes propounded in which chemical processes and known facts are alike disregarded. The wise member holds

his tongue as to what he knows or hopes to effect, for we can never forget that almost the entire membership of this Society are traders, united it is true to science by its noblest developments, but still traders, who have to make money, or to pay their chemists and managers to make it for them, and it is clearly against self interest to diffuse the means or processes by which we are, or would be, enriched. Thus one of the principal ends aimed at by our Society becomes in part its weakness and may be the cause of its failure in effecting the object for which it was established.

As time goes on, technical colleges and schools will more and more change this condition of matters. Already we have evidences of good work done in several important branches in the Arts, notably those of dyeing, calico printing and brewing industries, the results of which are published in journals dealing with these industries. Is it not our duty to affiliate such efforts more closely with our own?

Our late president referred to the admission of students or associates on easier monetary conditions than are now required, since the alteration of Rule 27 came into effect, which entails the payment of 2*l.* 5*s.* 0*d.* as admission fee, and the first year's subscription—a sum which although not high in itself may practically exclude a goodly number of young men to whom even such an expenditure would be a burden. I think it will be a great pity if this valuable suggestion be allowed to drop, as we must look to many of these young men not only ultimately to become members of our Society, but to fill and build up the ranks of the great army of Chemists and Managers of the Chemical Factories of our country.

The trying time for the student is when he leaves the scientific laboratory where he has been taught the theoretical rules and laws which govern chemical processes, but has had no opportunity of gaining experience of their practical developments, and I think it is the special duty of this Society to encourage, by every means in its power, any plan by which the young chemist may gain that experience which is required to fit him for his future work. In this the Chemical Society have an equal, if not a greater, responsibility than we have. I am delighted to see this is recognised by Dr. Armstrong in his address, delivered this year to that body, which forecasts the hope of a closer union and co-operation of chemical science, and its practical development represented by both Societies.

This may be met in part by Mr. Stanford's scheme which would provide for the sale of our excellent Journal along with the associateship of the Society at a small cost.

I would suggest a similar scheme should be adopted by the Chemical Society.

I confess that the great number of young chemists so situated who are eagerly clamouring for employment is a difficulty a way out of which in the present circumstances I cannot see the end, and which is becoming every year more accentuated. Either from fear of disclosure of secrets or the grudging of reasonable salaries, not only in chemical works, but also in many manufactories which, not being purely chemical in their nature, still have to deal with chemical processes, there is but one back analyst, if any, whereas, as is well known, in most chemical and cognate industries in Germany, Austria and France, every process is watched at every stage and carefully regulated by chemical skill. As an instance, in the "Badische Anilin und Soda Fabrik," employment is given to *eighty-one* manufacturing and research chemists at a very great annual cost, or for every fifty labouring men employed there is one chemical superintendent.

Would our neighbours continue such a system at what must be an enormous expenditure, if it did not pay? Did British chemical manufactures follow their example, firstly, adulteration of products would not be possible; secondly, partially manufactured material would not be sent out of the country to be returned fitted for the consumption of for example our dyeing and calico printing industries, as is the case with benzol, toluol, and other crude tar products; china clay, which we sell at 10*s.* or 20*s.* per ton, comes back to us as ultramarine, having a value 100 per cent. above that of the material from which it is made, and so on.

We are too fond of doing things in the "rough," content with selling coal and iron which require no particular skill in obtaining. Our alkali trade of such great importance (even in its present decadent condition) requires a chemical union to keep it alive, whereas the "Solvay" process invented and nurtured in France pays enormous profits.

Were our chemical works properly or correspondingly equipped with a staff of chemists equal to our foreign competitors we would not only manufacture our own material into its highest derivatives but provide means and even wealth for our other "raw" material in the multitude of well educated chemists which our universities every year pass into the workaday world.

It is by such means our foreign brother chemists obtain, and retain, their pre-eminence as chemical manufacturers, whilst we in Britain sustain a double loss in men and material. We have besides to contend with subsidies by their governments to foreign manufacturers, tariffs, taxes, cheap labour, all handicapping the British chemical manufacturer, who, in too many instances, will neither sow the seed for a future harvest, or even when niggardly sown refuses to stimulate it with sufficient nurture.

## New York Section.

Chairman: Alfred H. Mason.

Vice-Chairman: Arthur McGeorge.

Committee:

G. T. Bruckmann.	John McKesson.
T. Lynton Briggs.	W. H. Nichols.
H. Comer.	Francis J. Schleicher.
H. Endemann.	Jas. H. Stebbins, jun.
Jos. D. Geisler.	T. B. Stillman.
Jas. Hartford.	J. H. Wainwright.
E. G. Love.	

Hon. Treasurer: R. C. Woodcock.

Hon. Local Secretary:

Dr. H. Schweitzer, 159, Front Street, New York, U.S.A.

The following gentlemen have promised papers:—

- Dr. G. Archbold: "Technology of Starch."  
 Mr. H. Comer: "On Croosote."  
 Mr. E. N. Dickerson: "The relation of the U.S. Patent Laws, and Decisions thereunder, to American Chemical Industry."  
 Dr. H. Endemann: "Magnesia as a Waste Product, and its Uses."  
 Mr. E. E. Lungwitz: "Commercial Analysis of Whale Oil."  
 Dr. R. C. Schuphaus:  
 (1) "Evolution of Smokeless Powder."  
 (2) "Chemical Nature of Celluloid."  
 Dr. H. Schweitzer: "A New Reaction for the Detection of Soap in Lubricants."  
 Dr. Wm. G. Ungerer: "On Perfumery."

## Nottingham Section.

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J. B. Coleman.	C. Taylor.
H. Forth.	G. J. Ward.
J. F. Kempson.	

Treasurer: J. T. Wood.

Hon. Local Secretary:

R. L. Whiteley, University College, Nottingham.

SESSION 1894-95.

Wednesday, December 12th, 1894.—Mr. F. J. R. Carulla: "Recent Experiences when putting up a Steam Boiler."  
 January 1895.—Mr. Jas. O. Sullivan: "Note on the Estimation of Cane Sugar."

## Journal and Patent\* Literature.

Class.	Page
I.—General Plant, Apparatus, and Machinery .....	1047
II.—Fuel, Gas, and Light .....	1049
III.—Destructive Distillation, Tar Products, &c. ....	1053
IV.—Colouring Matters and Dyes .....	1054
V.—Textiles: Cotton, Wool, Silk, &c. ....	1056
VI.—Dyeing, Calico Printing, Paper Staining, and Bleaching .....	1057
VII.—Acids, Alkalis, and Salts. ....	1057
VIII.—Glass, Pottery, and Enamels. ....	1060
IX.—Building Materials, Clays, Mortars and Cements. .	1062
X.—Metallurgy .....	1063
XI.—Electro-chemistry and Electro-Metallurgy .....	1067
XII.—Fats, Oils, and Soap Manufacture. ....	1068
XIII.—Pigments and Paints; Resins, Varnishes, &c.; India-Rubber, &c. ....	1071
XIV.—Tanning, Leather, Glue, and Size .....	1072
XV.—Manures, &c. ....	1075
XVI.—Sugar, Starch, Gum, &c. ....	1076
XVII.—Brewing, Wines, Spirits, &c. ....	1077
XVIII.—Chemistry of Foods; Sanitary Chemistry and Water Purification; Disinfectants .....	1082
XIX.—Paper, Pasteboard, &c. ....	1085
XX.—Fine Chemicals, Alkaloids, Essences, and Extracts ..	1085
XXI.—Photographic Materials and Processes .....	1088
XXII.—Explosives, Matches, &c. ....	1088
XXIII.—Analytical Chemistry .....	1090

## I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

### PATENTS.

*An Improved Hoffman or Continuous Kiln for Drying and Burning Bricks and other Goods.* R. Cook, Kensal Rise, Middlesex. Eng. Pat. 19,848, October 21, 1893.

THE kilns are worked continuously with two rows of firing chambers, separated by a wide tunnel gangway which has three lines of rails running longitudinally and a smoke flue beneath, divided from the tunnel by air-tight iron plates. The firing chambers are so connected with flues and damper apertures, that each chamber may be placed in direct connection with either of those adjacent to it, and may discharge its products of combustion into the smoke flue; or when cooling may communicate its waste heat to any other chamber or to the tunnel between the two sets of chambers. A chimney shaft at one end provides draught. The roof of each chamber is formed of iron or steel joists 14 to 18 inches apart, with perforated bricks or lumps between, the bricks being grooved on opposite sides, to slide over the lower flanges of the joists; above them is a layer of cement concrete. The freshly-moulded bricks are placed on pallet boards, supported on a trolley, which is then run into the tunnel, where they are gradually dried at a gentle heat, and are afterwards stacked in a firing chamber; here they are first dried out by the aid of heat from a chamber which is cooling down, then fired, and finally, in cooling, serve to dry fresh batches of unbaked bricks. The trolley is specially constructed, having four corner uprights with stays; each upright has a rack with notches 7 inches apart, serving to carry longitudinal bars on which rest the pallet boards, kept in place by ledges fastened on their under sides.—W. G. M.

\* Any of these specifications may be obtained by post by remitting 8d.—the price now fixed for all specifications, postage included—to Sir Henry Reader Luck, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

*Imp. Means for Superheating Steam.* J. Musgrave and G. Dixon, both of Bolton. Eng. Pat. 20,359, October 28, 1893.

THE apparatus has reference to means for superheating a portion only of the steam produced in a boiler, and afterwards injecting it into the main body of the steam after it leaves the boiler. The superheater is constructed of a series of U-tubes secured to a steam chest provided with a division plate to separate the ends of the tubes; the apparatus is in Lancashire and similar boilers placed directly behind the boiler, so that the hot gases issuing from the flues may impinge on the superheater and heat the steam passing through it. The steam is taken from the main pipe by means of a branch, and the amount taken is controlled by a thermostat, which latter consists of a flat coil of piping filled with mercury, the outer end of which is connected by means of linkwork to a throttle valve placed in the branch pipe, and thereby controlling the amount of steam passing to the superheater. Modified arrangements are shown, applied to boilers of the marine and multitubular types, and in cases where it is desired to superheat the whole of the steam, some parts of the apparatus may be dispensed with and the superheater connected directly with the main steam pipe.—E. G. C.

*Improvements in Apparatus for Cooling Liquids or Gases.* J. Price, Liverpool. Eng. Pat. 20,944, November 4, 1893.

WITHIN a narrow vertical tank an endless travelling band is carried over a series of rollers arranged at the top and bottom of the tank so that the band exposes a large number of vertical folds with the lower ends passing over the rollers, dipping into the liquid to be cooled, whereby a large surface is exposed to a current of cold air blown through the tank. In this manner the temperature of the liquid becomes much reduced, and it is finally drawn off from the outlet in a cooled state. Gases are cooled by forcing them through the tank and exposing them to the action of a cold liquid such as water, which saturates the band as it passes under the lower guide rollers.—E. G. C.

*Improvements in the Manufacture of Finely-pulverised Substances for Use in the Production of Portland Cement, Pigments, and the like.* A. Patrick, Glasgow. Eng. Pat. 21,292, November 9, 1893.

THE raw materials are fed from a stone-breaker to an edge-runner, and thence by an elevator to a pair of stones, whence they pass by another elevator to a hopper provided with a rotating distributor, which carries the mixture into a current of air supplied by a Root's blower. The finely-divided portion is thus blown through a sieve, while the coarser particles fall to the bottom of the blowing chamber, and are returned to the stones. The material passing through the sieve falls into a large hopper, whence it is withdrawn by a rotating feeder. The dusty air is passed through a screen and fed into the intake of the blower, so that the same air circulates repeatedly. It is claimed that by this system the raw materials of cement-making are obtained in a finely-powdered state, free from gritty unground particles.—B. B.

*Improvements in Ovens for Baking or Firing Ceramic Ware and other Refractory Products.* J. B. Hanquiaux, Charleroi, Belgium. Eng. Pat. 22,637, November 25, 1893.

THE oven is of the continuous type, consisting of a series of compartments communicating by passages and arranged above generators, which can be fed through a space provided by the double wall separating the compartments of the kiln. The air before reaching the fuel is heated by passing through compartments the contents of which have already been burnt, and is at such a temperature that it ignites the fuel in the generators intermediate between the compartments. Each generator is provided with a movable bottom, which can be lowered at will, so that accumulation of ashes thereupon does not block the passage connecting

one compartment with the next. Thus the working of the kiln can be continued until the compartments have to be emptied in due order of the ware which has been burnt in them. Temporary connections can be made between adjacent chambers if required, and an auxiliary supply of producer gas may be fed in if necessary through the internal passages normally used for the introduction of solid fuel to the generators.—B. B.

*Improvements in and connected with Oil and other like Presses.* J. A. Johnson, London. From C. Gounelle, Marseilles. Eng. Pat. 23,595, December 7, 1893.

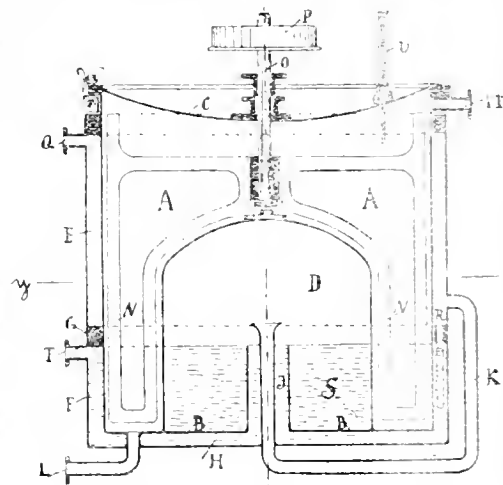
THE improvement claimed, facilitates the operation of clearing out the openings between the bars forming the sides of the cage of an oil-press, without taking the bars apart. The cage is made open in order to render the bars visible and accessible, and is preferably octagonal in shape, and constructed from a number of metal rings or hoops, provided internally and externally with projections and recesses. The rings are connected together at the required distance by vertical bars engaging with recesses on the exterior of the rings; this combination forms the skeleton of the cage, which is supported on legs resting upon hydraulic rams in the lower head. The materials are introduced into the cage and the charges are separated by metallic plates or filtering cloths (preferably woven wire cloths) until the cage is filled.—J. J. K.

*Improved Isolating or Non-conducting Material.* C. A. Peters, London. Eng. Pat. 11,560, June 14, 1894.

85-90 PARTS of kieselguhr are mixed with 15-10 parts of lime or cement, and the dry mixture thoroughly incorporated; moulds are filled with it, and placed in a vessel wherein they and their contents are subjected to the action of steam and water at high pressure. Pieces of wire netting, &c., may be inserted in the mixture as a core, for the purpose of stiffening it. The process is used for manufacturing steam-pipe coverings.—B. B.

*Improvements in the Process of and Apparatus for Heating and Concentrating Liquids.* F. Kleemann, Berlin. Eng. Pat. 14,184, July 24, 1894.

THE apparatus consists of a vertical cylindrical casing provided with jackets E and F, stirrers A driven from the



HEATING AND CONCENTRATING LIQUIDS.

pulley *p*, and a bell casing *D* fastened to the base *B*, in which the condensed water collects. Steam is allowed to enter the upper jacket *E* by the pipe *Q*, and, after circulating in this jacket, it escapes by the pipe *K* into the bell

D, where it is condensed, while at the same time the water of condensation from the jacket E passes into F by the pipe R. The water in D passes into the lower jacket F and away by the pipe T. The liquid enters by the pipe L, and, after being heated and concentrated in the casing, is discharged by the outlet M.—E. G. C.

*Improvements in Filtering Apparatus.* W. Oliphant, City of Paterson, U.S.A. Eng. Pat. 15,881, August 21, 1894.

WITHIN a closed casing are formed three separate filter beds, through which the liquid passes in succession, beginning with the one containing the coarse material and finally passing through the one containing the finest material. The liquid passes downwards through the first chamber, upwards through the second, and downwards through the third, after which it is led away by an outlet pipe. A sediment chamber connects the first and second filtering beds, having a discharge valve for the removal of sediment, while each bed is provided with a cleansing pipe in communication with the supply for cleaning out the chamber.—E. G. C.

## II.—FUEL, GAS, AND LIGHT.

*Observations on the Investigations of V. B. Lewes as to a Cause of Loss of Heat in Boilers.* M. Scheurer-Kestner. Bull. Soc. Chim. 1894, 11—12, 723—726

ACCORDING to Professor V. B. Lewes (*Engineering*, April 6, 1894), the essential factors representative of the waste as between the heat of combustion of a combustible and the heat practically utilised, are expressions of the radiation from surfaces, irregular admission of air, incrustations, and cooling of the flame on coming into contact with comparatively cold surfaces. He states the total loss as 40 per cent. of the heat of combustion. The author contends that this loss is over-stated, and refers to his experiments of 1885 with a triple-effect Alsatian boiler and a Green's reheater, the fuel being Altendorf coal, in which the steam produced involved the expenditure of 67.3 per cent. of the calories produced by the combustion of the coal; and later, in 1888, with English coal, the proportion reached 72.5 per cent. The loss is stated to be more nearly reckoned at 30 per cent. Several important factors are stated to be omitted in Professor Lewes's items of loss, namely: (1.) The calories carried away by the combustion gases as sensible heat (temperature of these gases), reckoned as between 5 and 10 per cent. as a maximum; (2.) The heat carried away by the same gases as latent heat (water vapour either produced in the combustion or arising from moisture in the fuel), reckoned as  $2\frac{1}{2}$  to  $3\frac{1}{2}$  per cent.; (3.) Calories represented by the smoke, produced even on the fire-grate; and (4.) The calories lost from incomplete combustion.

The author cites a memoir published by him in 1868, giving experiments showing that fire-gases deposit soot on a tube placed a little distance from the fire-bars of a steam boiler fireplace, through which cold water is run; and that the deposit disappears when the cooling is stopped, and explains that in such case the gases dissociated by the intense heat of the furnace on meeting a cold surface deposit carbon, which is afterwards transformed into carbonic acid by oxygen coming from the fire-grate as soon as the temperature admits of its combustion. The hydrogen remains free in the combustion products together with some hydrocarbons re-formed by what M. Sainte Claire Deville terms "partial recombination." The author discusses the bearing of these facts on the views of Professor Lewes.

—E. S.

*Difficulties in working Parallel Series of Gas Apparatus.* W. Leybold. J. fur Gasbeleuchtung, 1894, 301; Proc. Inst. Civil Eng. 1894, 118, 26—28.

THERE are certain limits to the dimensions of the various apparatus in gasworks; it is not advantageous for them to be too large, on account of the fluctuating demands of the different seasons of the year, and on account of the difficulty of erecting and handling. In large works it has been found advantageous to adopt small apparatus and to divide the works into separate sections, each dealing with a half or a third of the total production, or to have parallel series of apparatus dealing with portions of the make. The whole should be equal to one third or one-fourth more than the actual maximum daily production, as better results are obtained when the various appliances are not worked to their full capacity. Atmospheric condensers should have 4.6 to 7.6 sq. ft. of cooling surface per 1,000 cub. ft. of gas per 24 hours, and water condensers in the open air, 1.8 to 2.1 sq. ft. of surface for the same duty. In the open air, with the horizontal pipe condensers used in English works, or with the vertical condensers as used in Germany, the temperature of the gas in winter may fall below zero, while in summer it may rise instead of being reduced, with the result that naphthalene gets into the street mains. The dimensions of dry purifiers should be regulated so that the maximum velocity of the gas does not exceed 12 ins. per minute with the maximum make, assuming the purifiers to be empty. The actual flow of the gas is naturally greater than this when the purifiers are charged.

The gas generally passes from the condensers to the exhausters and then through the tar-separators, washers, purifiers, and station meters into the gas-holders. In order to work these in parallel sets, the pipe from the hydraulic main is divided into two before the condensers, and again united into one at the outlets of the same; from the exhauster, or exhausters, it passes into one main, which is again divided before the tar-separators, washers, and purifiers, and also for the meters and gas-holders. Condensers give very little back pressure, and equal quantities of gas will pass through two adjoining ones, except when one branch of the divided main is longer than the other, in which case the gas takes the nearest course; obstructions in the mains may also cause the gas to go principally in one direction; but such obstructions may be easily discovered by means of pressure-gauges, and also by the rise of temperature at the outlets. The gas is then driven through the two divided systems of tar-separators and washers. In the tar-separators a difference of pressures of  $2\frac{1}{4}$  to  $2\frac{3}{4}$  ins. should be maintained between the inlets and outlets.

The unequal division of the gas is less injurious in the tar-extractors than in the washers. The required quantity of water is regulated by taking tests at the tops and bottoms of the washers. With a single series this succeeds very well, but, with the parallel system, sometimes much, and sometimes little gas passes through one of the tar-extractors, and consequently through the washer attached to the same, with the result that the wash-water becomes too strong on the one side and too weak on the other. Pressure-gauges should be fixed on all the purifiers to indicate where increased back-pressure occurs. If each series contains four purifiers, one in each being kept in reserve, the difficulty of the unequal division of the gas does not occur when a box is being recharged, there being always three boxes in action on each side; but the purifying material may still become saturated with moisture. New material also causes less back-pressure than old, and a series with one or more purifiers containing new material allows more gas to pass than where there is only old material.

On reaching the gas-holders one of these is being filled while another sends out the supply; at times, when all the holders are full, gas has to be put into and sent out from one, at the same time, and this tends to produce stratification. When two or more holders are being used at the same time for the supply, attention must be paid to the varying pressures, or the heaviest one will drive gas into the lighter one.

The disadvantages that have been referred to, of working two parallel sets of apparatus, are only of importance as regards the washers, and these disadvantages may be avoided by a connecting pipe between the two systems. With four purifiers in each series the difficulty is not great, but only when series of three purifiers are used.

*On the use of the Auer Incandescent Mantle cause Partial Poisoning.* N. Gréhan. Comptes rend. 119, 1891, 342—350.

In a previous paper the author showed that the products of combustion from an Auer mantle contained a minute proportion of carbon monoxide—about  $\frac{1}{10000}$ th part. With a view to finding whether an animal breathing the air of a closed room, in which an Auer gas light was burning, could begin to be poisoned by the carbon monoxide—in other words, whether the quantity of this gas which is mixed with the other products of combustion, is in sufficient proportion in the air of a room, to be fixed by the blood—he repeated the classical experiment of Felix Leblanc. He operated with a dog, by first determining the volume of combustible gas contained in its normal blood, and afterwards, that contained in its blood after it had been placed a distance of one metre from a burning Auer light, in a room of 51 cubic metres capacity, where it remained seven hours. He found that only 0.15 cc. of carbon monoxide per 100 cc. of blood had been absorbed, corresponding to  $\frac{1}{66666}$ th part of carbon monoxide in the air of the room. He concludes therefore that the use of the Auer mantle is unattended with any danger of poisoning by carbonic oxide.—H. S. P.

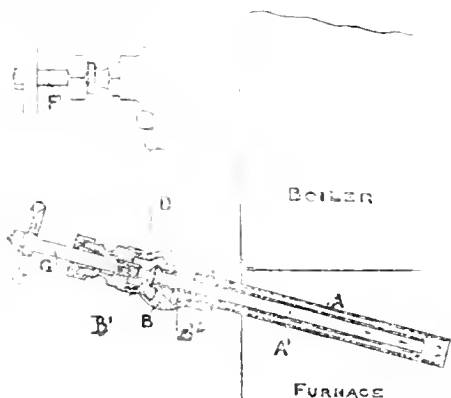
#### PATENTS.

*An Apparatus for Notifying the Presence in the Air of Gases such as Fire Damp.* E. Hardy, Dreux, France. Eng. Pat. 15,329, August 11, 1893.

See under XXIII., page 1091.

*A New or Improved Apparatus for Burning Creosote and Similar Oil for Steam Generating and Heating Purposes.* W. Allen, Manchester. Eng. Pat. 18,656, October 5, 1893.

This liquid-fuel burner is one of those where the oil is atomised by steam alone and by a single supply of this substance, and where there is no admixture of the fuel with air by the burner.



Oil, controlled by F, flows through D into B, where its volume is again specially regulated by G. It then enters the tube A, and at the further end of this tube, is atomised by the high-pressure steam which is escaping through the surrounding tube A from the chamber B.

The chamber B, receives steam by an arrangement similar to that provided for the oil (but not shown in the figure).

The special features of the invention claim: (1) the valve body B, with its two chambers B<sup>1</sup> and B<sup>2</sup>; and (2) the provision G for accurate regulation of the oil supply; when once this regulation is effected it need be only very occasionally disturbed, as the oil supply may be shut off by the main valve.

The inventor inclines the burner, as shown, so that its jet may play upon the layer of fire brick or slag, with which he covers the fire bars, and upon the small coke fire, which he uses at starting. It will also be noticed that he leads the burner some distance into the fire-box.

—E. R. B.

*Improvements in Gas-making Apparatus.* J. W. Hartley, Kilmarnock. Eng. Pat. 19,161, October 12, 1893.

The improvements relate to gas generators, gas washers, and gas purifiers. The gas generator is of the ordinary vertical cylindrical type, lined with fire brick, in combination with which the inventor claims the use of one or more poker bars for removing clinkers, and an arrangement for indicating the level of the fuel in the generator. The pokers or bars, secured at the lower end to a cross bar, when operated are caused to rise vertically into the incandescent fuel, through slots in the circular frame surrounding the fire-grate, and cut away the clinker which forms upon the brick lining. The bars are next lowered to their normal position below the upper surface of the grate. The frame and fire-grate are now caused to rotate (as they are constructed to do) to a certain small extent, and the poker bars are again driven upwards to their full extent, and so on, until clinker is cleared away from the whole of the lower interior of the brick lining. For ascertaining the level of the fuel, a rod passes through the top of the generator, and to the inner end of which is fixed a cone-shaped weight, while the upper end is connected with a wire rope made to pass over suitable pulleys and ending in a counterpoise and indicator. The cone weight is lowered until it reaches the fuel, the counterpoise indicator showing the level. The improved gas washer or dust box consists of one or more concentric cylinders, the lower rims of which are slotted, suspended in a suitable cylinder to a short distance below the water level. The gas passes into the top of the inner cylinder, through the water, into the annular space between the cylinders, and so on to the outlet of the outer casing. A horizontal wheel in the floor of the apparatus and rotated by hand, is provided upon the upper side with scrapers to remove any deposit from the cylinders, which deposit, falling to the bottom of the apparatus, is guided by a scraper upon the under side of the revolving wheel, to an opening in the side of the washer, through which the deposit is removed and the wheel operated. The improved gas purifier consists of a suitable tank with horizontal partitions or shelves carrying the purifying material, and separated by another series of horizontal partitions constructed so that the spaces above the filtering material communicate with a common inlet, and the spaces below with a common outlet.—S. P. E.

*Improvements in Apparatus for Mixing Gas and Air for Gas Furnaces.* G. Axdorfer and C. F. H. Sass, both of Hamburg, Germany. Eng. Pat. 22,681, November 25, 1893.

The patentees provide a mixing chamber between the combustion chamber and the gas- and air-inlet nozzles of furnaces fired by gas. The lateral walls of the mixing chamber are provided with slots corresponding with the orifices of the said nozzles, which have also the form of slots. In the mixing chamber there is also fitted an adjustable grate slide, whose bars cover the slots in the two walls, more or less, according to the adjustment of the slide, the quantity of gas and air to be used in combustion being thus regulated.

Upon leaving the nozzles, the gas and air impinge against the bars of the slide and against the opposite wall of the mixing chamber; the "strong whirling movements" so produced, insuring a uniform mixing of the gas and air.



A considerable number of forms of apparatus, all embodying the above principles, are described: one of the simplest is shown in Fig. 1 where 1 is the central retort, with closed reservoir 1', and grate and ash-pit 2. Air enters at A and passes through the fuel in the central retort to arrive at 3, then to 4, finally leaving the apparatus as fuel gas at B. At 5, 6, 7, 8 any desired additional air or air and steam may be admitted.

The central retort may be vertical, horizontal, or inclined, and may be single or double.

The patentee has also applied his improved method of fuel-burning to the case of steam-raising. In this case his gas-making apparatus becomes an adjunct to the furnace of various types of boilers, and in most instances is combined with water-tube grates, all or part of the floor of which is fitted with mechanism capable of raking the fuel from the floor towards the back of the furnaces.

Most of the many modifications described are illustrated in considerable detail, one of the more elaborate arrangements being shown in Fig. 2. Here the fuel chamber 1 is formed by two series of curved tubular grate bars, 17 and 17', with air spaces between the tubes. Tubes 18 and 19 connect 17 and 17' with the water space of the boiler 20, so that there is efficient circulation in them. Between the front portions of 17' is the raking arrangement 21, consisting of segments 22, supported on rock shafts 23, which are operated by the lever 25 through the link 24. The fuel is progressively shifted on by the raker into the lower part of the grate. The partition wall 31 separates the chamber 32 from chambers 26 and 39, while the "water-wall or curtain" 34 and the wall 35 encloses the upper and lower parts of chamber 33.

The course of the air is as follows. From chamber 26, through the grate and fuel, into chamber 1; thence, through the more strongly incandescent fuel lying between the bent parts of 17 and 17', into chamber 32. In passing from 32 to chamber 33 the fuel gases again pass through incandescent fuel, and combustion is finally completed in the boiler tubes 37, which open from chamber 33. The toothed or grated cylinder 38 allows of the disintegration and removal into the ash-pit 39 of clinker and cinder. Any fuel which may chance to fall through the grate 21 is caught by the grate 29 and is burnt by the air entering by doors 27 or 28.

—E. R. B.

*Improved Incandescent Vapour Lamp with Vapour Generator.* C. Schinz, St. Petersburg. Eng. Pat. 13,419, July 11, 1894.

This incandescent vapour lamp is provided with a retort or generator, in which the hydrocarbon is converted into vapour, this vapour then passing out as a fine jet into the lower opening of the chimney, drawing air with it, the mixture burning at the upper end with an intense blue flame. The lamp has the usual form of reservoir for the hydrocarbon, vertically fixed in the centre of which is a tube containing the wick. Surrounding this tube, and fitting closely around it at the bottom end, is a second one, open to the air above, thus forming an air space around the inner tube and keeping it cool. The tube containing the wick has at the top a lateral enlargement which constitutes the retort in which the hydrocarbon that is carried up by the wick, is vaporised by the heat from a second smaller wick burning beneath it. This second wick and the retort are enclosed by a suitably-shaped chimney or cover made to slide outwards horizontally for uncovering and igniting the wick. A small nozzle leads from the top of the retort, through which the vapour issues, into the bottom of the chimney, carrying the air necessary for combustion with it. The vaporisation is regulated and the lamp extinguished by raising or lowering the smaller wick, suitable means for which are provided —S. P. E.

*An Improved Method of and Apparatus for Detecting and Measuring the Presence and Amount of Inflammable Gases or Vapours Mixed with Air.* B. C. Tilghman, jr., Philadelphia, U.S.A. Eng. Pat. 15,153, August 14, 1894.

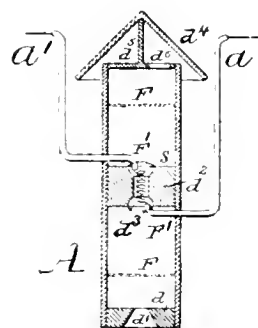
If a metal wire be raised to a suitable temperature—for example, by the passage through it of an electric current—and if the surrounding atmosphere contain inflammable gases, these latter will burn on the surface of the metal, with the consequence that the temperature of the wire will rise. The greater the amount of gas so burned, the higher will be the temperature of the wire.

Now certain metallic conductors—for example platinum—have their electrical resistance increased by increase of temperature, according to a definite ratio; it is therefore obvious that by measuring the increased resistance of the platinum wire part of the circuit of suitably constructed instrument, the amount of inflammable gas present in a given atmosphere can be ascertained.

In his patent, the inventor describes the structures and uses of such instruments, including a special form of Galvanometer.

In Fig. 1 is represented a vertical section of the detecting instrument. The metal casing A carries a block of asbestos or fire-clay  $d^2$ , in the axial passage  $d^3$ , of which is disposed

Fig. 1.

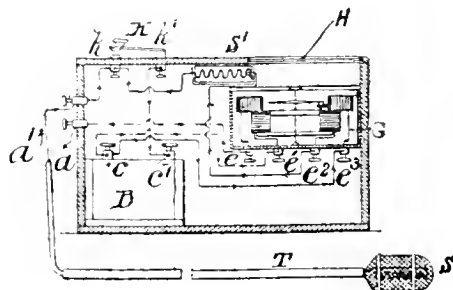


the platinum spiral S; this, in the absence of inflammable gas, is kept at a just visible heat when the current passes through  $a a'$ . The heated spiral causes a current of air to pass through the instrument, traversing  $d^1$ ,  $d^3$ , and  $d^5$ , successively. The sloping roof  $d^1$  and the gauze baffles FF and F' F' minimise draughts, the gauzes also preventing the ignition of inflammable atmospheres.

For the quantitative estimation of the inflammable gas indicated by the foregoing instrument, "any proper means for measuring resistance may, of course, be used"; but the patentee, while not limiting himself to any particular means, specifically claims, in combination with his instrument, the Wheatstone bridge, the rheostat, and the fixed resistance methods. Where quickness of manipulation is desired, it is preferable to use a balancing resistance, and for this purpose he employs a second platinum coil, like to the testing-coil, and under like conditions, except that it is surrounded by an atmosphere free from inflammable gas.

It is this arrangement also which he uses in his portable instrument, represented in Fig. 2, where S is the testing-

Fig. 2.





spiral, carried at the end of the pole T, S<sup>1</sup> the balancing spiral, G the patentee's special galvanometer (for further description of which, reference must be made to the specification of this patent), B a small battery, and H a window through which the indications of the galvanometer may be noted. From the battery, the first circuit (indicated by the dot and dash lines) is via c, c<sup>1</sup>, G, c, a, S, a<sup>1</sup>, and (when the key K is depressed) through h<sup>1</sup> to c<sup>1</sup>; the second circuit (indicated by full lines) being via c, c<sup>1</sup>, G, c<sup>2</sup>, S<sup>1</sup>, and, when K is depressed, through h<sup>1</sup> to c<sup>1</sup>.

There are thirteen claims covering the various combinations of the pieces of apparatus proposed (including the modified galvanometer) among themselves, and the electrical currents manipulated, while two further claims cover the methods employed. These are respectively, that, where the varying resistance of the testing spiral is measured, and that, where the resistance of the testing-spiral under the influence of inflammable gas is compared with that of another not so influenced.

Special advantages of the method are, according to the inventor, that "the indications given are positive, and do not in any way depend upon the judgment of the person making the test," and that there is no necessity for the collection of samples, nor for the observer to go to the, perhaps, dangerous place where the gas itself is being tested; indeed, in the case of mines, there may be permanently fixed in the levels any number of testing apparatus, which all record at the office at the surface.

It is evident that with an instrument of this kind, as, indeed, is pointed out by the inventor, *any change* in the temperature of the gas circulating about the testing-spiral, and also with a given air temperature, the rate of flow of the current of air, would affect the indications and that thus, with, for example, a colder current, an amount of hydrocarbonous less than really existed at the time might be indicated. So far as such a state of things as this might be brought about by draughts, the inventor considers the ganze baffles, &c. of the instrument a sufficient preventive.

With regard to the temperature at which the spiral is maintained during testing, though the patentee prefers to keep it "barely luminous in diffused daylight," yet he states that if the spiral be caused to "glow brightly, the increase in resistance under the same proportions of gas or vapour in the air will be practically the same as when less strength of current is used."—E. R. B.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*The Crude Petroleum occurring in the Trenton-Limestone.*  
O. Mühlhäuser. Dingl. Polyt. Jour. 292, 1894, 116—119.

THE name "Trenton-limestone oil" (from its occurrence in the Lower Silurian Trenton limestone) is proposed for the crude petroleum, hitherto known under the name "Ohio oil" or "Linna oil," and distinguished by the large proportion of sulphur it contains. Owing to the sulphur compounds present, the oil was practically useless for illuminating purposes and was only employed for heating. The quantities raised, up to 1884, from the Berea sand (compare table) had been comparatively so small that processes for the elimination of the sulphur were not seriously considered until the large quantities, tapped from the Trenton-limestone, drew the attention of technologists to this important problem, viz., to so refine the oil as to render it useful for illuminating purposes. A successful process has been invented and carried out in practice by Frasch, consisting in the treatment of the oil with metallic oxides, whereby the sulphur compounds (termed "skuunks," on account of their nauseous smell) are destroyed, and almost the total quantity of sulphur (from 0.4 to 1.0 per cent. of the oil) recovered in the form of metallic sulphides.

#### TRENTON (OHIO, LINNA), OIL PRODUCTION.

Year.	Barrels.	Year.	Barrels.
1876	3,754	1883	47,632
1877	29,888	1884	30,984
1878	38,479	1885	60,000
1879	29,442	1886	478,370
1880	38,940	1887	701,844
1881	34,867	1888	1,001,658
1882	50,760	1889	1,247,136

According to Lange, at present 23,725,000 barrels are refined per annum by Frasch's process, yielding 28,105 tons of sulphur. This enormous quantity of sulphur might yield about 90,000 tons of sulphuric acid of 66° B, but hitherto the sulphur has been allowed to escape into the air as sulphurous acid.

The author, referring to Mahary and Smith's paper (this Journal, 1891, 628), discusses the several series of sulphides—adipathic sulphides and thiophen compounds—that may be assumed to exist in the Trenton oil; and from the fact that the sulphides can be extracted by means of concentrated sulphuric acid, he draws the conclusion that the sulphur cannot occur in the form of sulphides of the series  $(C_nH_{2n+1})_2S$ . For his speculations as to the manner in which the sulphur may be linked with the carbon atoms, the reader must be referred to the original paper, as no experiments are given to substantiate them.—J. L.

*Products of the Distillation of Petroleum Tar.* W. Tistchenko. Jour. Soc. Ph. Ch. russe de St. Pétersbourg, 1893, No. 2.

THE distillation of this tar was carried out with superheated steam. The gaseous products of the distillation are incompletely absorbed by bromine. The brominated hydrocarbons which were obtained belong to the ethylene series. Two series of hydrocarbons were obtained, boiling from 90° C. upwards, which differ clearly from each other in the range of their specific gravities:—

LOWER.	
94—96	0.7100
120—122	0.7275
116—118	0.7468
170—172	0.7995
190—192	0.7823
HIGHER.	
106—108	0.7726
131—136	0.7759
160—162	0.7786
183—184	0.7943

The elementary analysis of these fractions showed that most of them contain oxygenated compounds, and, further, that the fractions of that series distinguished by the higher specific gravity, contain more hydrogen and carbon than the other fractions. Besides these hydrocarbons, the crude distillate contains some products which are formed during distillation. These are (1) sulphides (mercaptans) capable of combining with mercuric oxide; (2) nitrogenous compounds, which can be extracted from the crude distillate either by hydrochloric acid or mercuric chloride, and which in the free state form an oily liquid possessing a very penetrating smell, and boiling at from 170° to 265° C.; (3) crude phenols were obtained by extracting the distillate with a solution of caustic potash, and treating the latter with carbonic acid. Treating this solution after the removal of the phenols with sulphuric acid, some liquid acids, possessing a strong, rancid smell, were liberated.

—C. O. W.



*The Qualitative Composition of Official Beech- and Oak-Wood Cresols.* A. Behal and E. Choay. *Comptes rend.* **118**, 1894, 1379.

See under XX, page 1087.

*Distinction between Coal-Tar and Blast-Furnace Pitch.* D. G. Buchanan. A paper read before the Glasgow City Analysts' Soc. Abstracts, No. 4, 1893—94, 21.

See under XXIII., page 1098.

*Volumetric Estimation of the Three Isomeric Cresols, and also of Xylol.* F. Keppler. *Archiv. f. Hygiene. Zeits. Anal. Chem.* **33**, 473.

See under XXIII., page 1099.

*Some Blue Lakes derived from Dibromogallanilide and Some Blue Reactions of the Polyphenols.* P. Cazeneuve. *Comptes rend.* **118**, 1046.

See under XXIII., page 1098.

#### PATENTS.

*An Improved Process for Recovering Alkali from a Waste Product.* J. B. Murray and M. B. Baird, both of Glasgow. Eng. Pat. 18,786, October 7, 1893.

THE waste product of shale oil works, known as soda-coke, is crushed and heated in a crucible, with stirring, as long as flames are produced. The residue, reduced to about three-fourths its original weight, is boiled with water in a suitable pan, and, after settling, the clear liquid is transferred to a copper pan to be concentrated until fit for running off to crystallise, soda crystals being obtained. The coke is again boiled with more water, and the process repeated as long as alkali is extracted. The coke that remains is about 37½ per cent. of the original soda-coke, and after thorough drying may be ground up with oil for making black paint or blacking.

The soda solution, instead of being crystallised, may be causticised by lime in wrought-iron tanks, the contents being boiled up by steam. The lye and the stronger washings obtained are concentrated in cast-iron pans, steam-jacketed or heated direct. Foreign salts fall at a certain point of concentration.—E. S.

*Improvements in or connected with Retorts for Distilling Shale or Oil-yielding Minerals.* J. Gray, Loanhead, North Britain. Eng. Pat. 21,485, November 11, 1893.

THE improvements are in connection with the present vertical type of coal retorts, in which gases and vapours are formed for heating the retorts employed for distilling shale or other oil-yielding minerals. One or more communications are made between the top of the retort and the bottom near the outlet for the gases, formed either of pipes outside the brickwork or flues constructed within, and provided with suitable regulating valves and doors for removing any deposit that may accumulate. The main object of this arrangement is to allow any gases formed at the upper part of the retort, to escape into the flue at the bottom without having to force its way through the column of fuel.—S. P. E.

*An Improved Process for Purifying Petroleum.* J. Schrader, Stuttgart, Germany. Eng. Pat. 15,323, August 11, 1894.

THE process consists in distilling petroleum with phosphoric or boric acid, or with the salts of these acids, together with sulphuric acid, for the purpose of destroying any resin or other impurities that the hydrocarbon may contain.

—S. P. E.

#### IV.—COLOURING MATTERS AND DYES.

*Colouring and other Principles contained in Mang-Kouda.*

A. G. Perkin and J. J. Hummel. (Trans. 1894, 851.) *Proc. Chem. Soc.* 1894 [142], 175.

MANG-KOUDOU is the root bark of *Morinda umbellata*, largely used in Java for producing fast reds in the native calico prints. The authors correct their previous statement (this Journal, 1894, 940) that its colouring matter was alizarin, and now show it to be morindone. The have isolated 11 distinct substances from the root, which also contains free acid, the nature of which has not yet been determined. No cane sugar was found, a distinction from chay root (*loc. cit.*) and from madder. Full details of the methods employed in extraction and separation are given, and the behaviour of the substance as a dyestuff is described.

#### PATENTS.

*The Manufacture or Production of a Trihydroxynaphthalene Monosulphonic Acid.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 19,624, October 18, 1893.

THE compound described is obtained from naphthalene tetrasulphonic acid (Senhofer, Ber. **8**, 1486) by fusing it with alkalis above 240° C. The naphthalene tetrasulphonic acid is produced either by sulphonating naphthalene or 2,3'-naphthalene disulphonic acid. The new trihydroxynaphthalene monosulphonic acid may also be obtained from the naphthol trisulphonic acids or the dihydroxynaphthalene disulphonic acids which are formed as intermediate products in the fusion of naphthalene tetrasulphonic acid with alkalis at temperatures below 200° and 250° respectively, since Senhofer's acid is a mixture of two tetrasulphonic acids. The fusion may be conducted in open or closed vessels, the proportion of potash or soda to acid being 2:1. When complete, the melt is dissolved in water and the solution made acid with hydrochloric acid, when, if potash has been used, the potassium salt of the trihydroxynaphthalene sulphonic acid separates out. The neutral salts of the new acid dissolve in water to red-coloured solutions with a slight bluish fluorescence. Bleaching powder gives a wine-yellow coloration, which is destroyed by an excess. In solutions of the acid salts, ferric chloride gives a brown colour.

—T. A. L.

*Process for Producing Insoluble Azo Colours on Fibre.* O. Luray, London. From "The Farbwerke vormals Meister, Lucius, and Brüning," Hoechst-on-the-Maine, Germany. Eng. Pat. 21,087, November 6, 1893.

THE specification relates to the production of an insoluble blue azo dyestuff on the fibre by padding the latter with β-naphthol and passing the prepared fibre through a bath containing diazotised dianisidine together with cupric chloride. The following quantities are given. The padding solution made up to 10 litres, contains 150 grms. of β-naphthol in 250 grms. of soda lye of 22° B., together with 400 to 500 grms. of Turkey-red oil. The goods, having been padded, are dried and printed with or passed through the diazo solution prepared from 61 grms. of dianisidine, 106 grms. of hydrochloric acid of 22° B., 600 cc. of water, 300 grms. of ice, and 265 grms. of sodium nitrite. For the printing solution 15 grms. of the diazo solution, 6 grms. of cupric chloride of 40° B., 50 grms. of gum water, and 35 cc. of water are mixed together. Other phenols may be used in place of β-naphthol, and the effect of adding cupric chloride to the diazo compounds of other bases than dianisidine, is to confer greater fastness on the azo colours produced by this process on the fibre.—T. A. L.

*Improvements in the Manufacture and Production of New Azo Dyes.* J. Y. Johnson, London. From "The Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 21,753, November 14, 1893.

IN Eng. Pat. 14,678 of 1893 (this Journal, 1894, 724) the preparation of a *m*-phenylene diamine disulphonic acid is

described. According to the present patent, one molecular proportion of this compound is capable of combining with two molecular proportions of a diazo compound, either of the same or of different ones. Valuable products result from combining one molecular proportion of *m*-phenylene diamine disulphonic acid with one molecular proportion of diazotised primuline and subsequently with one molecular proportion of a diazotised amine or its sulphonic acid, such as aniline, *o*- or *p*-toluidine, *m*-xylydine, *a*- or *β*-naphthylamine, *m*- or *p*-sulphanilic acid, naphthionic acid, and the *β*-naphthylamine sulphonic acids 2.1', 2.3', and 2.4', &c. About 13 kilos. of aniline hydrochloride are diazotised with 7 kilos. of sodium nitrite and 15 kilos. of 30 per cent. hydrochloric acid and diluted to 500 litres. This solution is stirred into 27 kilos. of *m*-phenylene diamine disulphonic acid and 45 kilos. of crystallised sodium acetate in 500 kilos. of water. After stirring for from 5 to 8 days, diazotised primuline from about 56 kilos. of primuline (corresponding in any case to 7 kilos. of sodium nitrite), 7 kilos. of sodium nitrite, 500 litres of water, and 49 kilos. of hydrochloric acid, is added and the whole is stirred for a few days. Hydrochloric acid is then added to precipitate the dyestuff, which is filtered off and mixed and ground with sufficient soda to form the soluble sodium salt. The colouring matter so obtained is a brown powder soluble in water giving reddish-orange shades on unmoordanted cotton. A bright orange for cotton is obtained by diazotising the amount of primuline corresponding to 7 kilos. of sodium nitrite as described above, and adding this to an ice-cold solution of 27 kilos. of *m*-phenylene diamine disulphonic acid and 28 kilos. of calcined soda in 400 litres of water. After stirring 24 hours, 30 kilos. of crystallised sodium acetate are added, and the diazo compound from 19.5 kilos. of sodium sulphanilate, 7 kilos. of sodium nitrite, and 39 kilos. of hydrochloric acid is run in. The whole is then stirred for from 5 to 8 days, when the liquid is heated, acidified with hydrochloric acid, and the dyestuff precipitated with salt.

—T. A. L.

*Manufacture of Blue Acid Colouring Matters of the Triphenylmethane Series.* O. Imray, London. From "The Society of Chemical Industry in Basle," Basle, Switzerland. Eng. Pat. 22,741, November 27, 1893.

By heating together equimolecular proportions of dimethyl- or diethyl-aniline, methyl- or ethyl-*o*-toluidine and *m*-oxybenzaldehyde in presence of a condensing agent there result trialkylated derivatives of diamido-phenyl-tolyl-*m*-oxyphenylmethane which after sulphonation and oxidation give useful blue acid colouring matters. These dyestuffs differ from the analogous ones from tetra-alkyl-diamido-*m*-oxytriphenylmethane in giving bright blue shades on wool and silk which are not changed by artificial light. The following details are given as an example:—12.2 kilos. of *m*-oxybenzaldehyde, 12 kilos. of dimethylaniline, 12 kilos. of methyl-*o*-toluidine, 50 kilos. of hydrochloric acid, and 16 kilos. of sulphuric acid of 66° B., are heated in an enamelled vessel on the water-bath for 24 hours. After careful neutralisation with an alkali, the product is steamed and the leuco base remaining behind is washed two or three times with warm water and dried at 100°. 10 kilos. of this leuco base are dissolved in 40 kilos. of sulphuric acid of 66° B. at a temperature of 60° C. and sulphonated by adding 10 kilos. of fuming sulphuric acid containing 24 per cent. of anhydride. The sulphonation is complete when a test dissolves completely in sodium acetate. The melt is then poured into 200 litres of water and the solution of the lime salt is mixed with 6 kilos. of sulphuric acid and oxidised with 2.8 kilos. of sodium bichromate. The chromium oxide is precipitated with ammonia, filtered off, and the solution of the colouring matter is evaporated, giving a dyestuff in the form of a powder with a metallic lustre easily soluble in water with a blue colour. It dyes wool or silk blue from an acid bath. The order of the sulphonation and oxidation may be reversed, proceeding in the usual manner for the formation of colouring matters of the malachite green series and sulphonating the basic colouring matters obtained with 4–8 kilos. of fuming sulphuric acid at a temperature below 30° C.—T. A. L.

*Manufacture of New Bases and their Sulpho Acids.* C. D. Abel, London. From "The Actien-Gesellschaft für Anilin-Fabrikation," Berlin, Germany. Eng. Pat. 23,310, December 1, 1893.

By reacting with aldehydes on the chrysoidines, there result so-called amido-triazines, which are very stable bodies, not even decomposed by heating with mineral acids under pressure to 170–180° C. Since they contain an amido group they may be diazotised and combined with amines and phenols and their sulphonic and carboxylic acids. The following formula represents the constitution of the simplest of the bodies derived from benzaldehyde and the chrysoidine from aniline and *m*-phenylene diamine—



On sulphonation, these products may be converted into mono- or disulphonic acids according to the quantity of fuming sulphuric acid used and the temperature. Certain of these sulphonic acids have a sweet taste. A solution of 25 kilos. of chrysoidine and 11 kilos. of benzaldehyde in 11 kilos. of concentrated hydrochloric acid and 50 kilos. of ordinary acetic acid is heated for 6 hours on the water-bath until the colouring matter has disappeared. The melt is then poured into 1,000 litres of water, filtered, and the base precipitated by adding sodium carbonate. The sulphate crystallises from water in white needles. The base obtained therefrom is colourless and insoluble in water, but easily soluble in hot alcohol and benzene. It forms a stable diazo compound which is sparingly soluble in water. In order to convert the base into a monosulphonic acid, one kilo. is dissolved in 3.6 kilos. of fuming sulphuric acid containing 20 per cent. of anhydride, and the temperature of the melt is slowly raised. On pouring it into water, the monosulphonic acid separates in white crystalline flakes, and gives a tolerably easily soluble diazo compound. By continuing the action of the sulphuric acid at the temperature of the water-bath, a disulphonic acid is obtained which may be separated in the form of the calcium or sodium salt.

—T. A. L.

*Manufacture of New Condensation Products and Colouring Matters for Dyeing and Printing.* J. C. L. Durand, G. E. Huguenin, and A. J. J. d'Andiran, St. Fons, Rhone, France. Eng. Pat. 15,064, August 7, 1894.

This is an extension of Eng. Pat. 24,802 of 1893 (this Journal, 1894 247), and relates to the condensations of phenols and their sulphonated derivatives, and more especially the *β*-naphthol sulphonic acid of Schaeffer with the gallocyanines which are obtained by condensing the salts of nitroso-diethylaniline or of dialkylamidoazobenzene with gallic acid and its derivatives, such as gallamic acid, gallanilide, the gallonaphthylamides, and methyl gallic ether. The patentees further claim the condensation of pyrogallol or dimethyl-*m*-amidophenol with the gallocyanine from nitrosodimethylaniline hydrochloride and gallic acid, and also the condensation of resorcinol with the gallocyanines from nitrosodimethylaniline hydrochloride and gallamic acid or methyl gallic ether. All the leuco products thus obtained are subsequently converted into colouring matters on oxidation, and if necessary are sulphonated as described in Eng. Pat. 24,802 of 1894. About 33 kilos. of the colouring matter obtained by the action of gallamic acid on diethyl-amidoazobenzene or nitrosodiethylaniline hydrochloride, is added to a solution of 40 kilos. of 2.3'-naphthol sulphonic acid in 160 kilos. of sulphuric acid of 66° B., and the melt is heated on the water-bath until the reaction is complete. The melt is then poured into water, when the leuco product precipitates and may be used directly for printing if it be oxidised on the fibre. This oxidation, which takes place preferably in an alkaline solution, produces an acid blue colouring matter which dyes wool and silk blue from an acid bath and will also dye on mordants. The shades obtained on chrome mordants are fast to light and washing. Two tables are given in the specification, giving the reactions

of the dye compounds and of the colouring matters with various reagents.—T. A. L.

*Method of Finishing Metal in Light-resisting Colours by means of Alizarine Colours.* J. Perl and P. Herrmann. *Verh. d. Ing. Pat.* 15,827, August 11, 1894.

See under XIII. B., page 1072.

## 7.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Souple Silk.* H. Silbermann. *Färb. Zeit.* 5, 352—355 and 365—366.

*Souple silk* is raw silk treated in such a manner that a portion only of the silk-gum is removed, while at the same time it acquires the feel and lustre of boiled-off silk. It is used only for weft. During the soupling process the fibre increases in thickness, owing probably to swelling of the silk-gum. It has these advantages over boiled-off silk, that there is less loss of weight, an important consideration with an expensive fibre, and that it possesses a greater capacity for taking up vegetable and mineral weighting materials. The latter property it possesses even in a greater degree than raw silk, probably owing to the swelling up of the silk-gum. Soupling may be regarded as a morphological alteration of the raw silk fibre, whereby the sericin, without being removed, becomes soft, pliant, and lustrous.

For coloured silks the soupling is carried out in boiling baths containing an acid salt, such as cream of tartar or sodium bisulphate, the completion of the process being indicated by the requisite softness of feel, &c. having been attained.

For black silk, soupling is combined with the weighting and dyeing processes, and the method here described is for the production of a moderately weighted black on souple silk, noting especially the influence of each operation on the tenacity and elasticity of the fibre.

The raw silk, after scouring, is mordanted with basic ferric sulphate. The bath is much weaker and the basicity less than in the dyeing of boiled-off silk, for, owing to the non removal of the sericin, the fibre has a greater power of dissociating iron mordants. In consequence of this rapid dissociation, frequent additions of soda are made to keep up the original basicity. Sodium sulphate accumulates in the bath, and from time to time a fresh bath must be brought into use. To obtain a black weighted to about 190 per cent., four passages through the iron bath are given, the silk being passed after each dip through a lukewarm sodium carbonate solution to complete the fixation of the iron. Serimetric measurements show that these operations decrease the tenacity by 4.5 and the elasticity by 2.5 per cent.

The silk is now dyed Prussian blue by working in a bath of potassium ferrocyanide. The temperature of the bath is lower, and the amount of acid used less, than for boiled-off silk. The Prussian blue is much more superficially fixed than on boiled-off silk, and its injurious action on the fibre is much greater. The tenacity is decreased by 11.7 and the elasticity by 1.9 per cent.

The next operation, the soupling proper, is effected by means of a tannin material, usually catechu. The silk is worked for  $1\frac{1}{2}$ —2 hours in a boiling catechu decoction at 7° B., the bath cooled to 70° C., and 10 per cent. of stannous chloride added; the silk is then worked some time longer, or even allowed to steep overnight; the weight is greatly increased in this operation, after which the tenacity is less by 0.2 and the elasticity by 12.4 per cent. Very great care must be exercised to prevent exposure to air during the treatment with stannous chloride, the neglect of this precaution having sometimes resulted in the total destruction of the fibre.

A passage through weak soap solution follows, and then another ferric sulphate bath, not followed by treatment with soda. Like all metallic salts, this mordant very injuriously affects the tenacity (decrease of 15.1 per cent.) and elasticity (37.3 per cent.). These tests, however, were not made until a few months after the operation, in order to ascertain the practical effect of the compounds fixed on the fibre. The silk has now a reddish-black tone. After a thorough washing it is worked in a sumach decoction at 12—14° B., entered at 50°—60° C., the temperature raised to 90—95° C. in three-quarters of an hour, and worked half an hour longer. The effect is favourable; the tenacity increases by 5.8, the elasticity by 16.7 per cent. The silk has now a deep bluish-black colour.

The next bath contains pyrolignite of iron at 20° B., and has an injurious effect on the fibre (—13.4 tenacity, —12.3 elasticity); the black is now redder in tone and contains much uncombined iron. It is changed to a greenish-black by working in a bath of potassium ferro- and ferriyanides (10 per cent. of each) with 15 per cent. HCl. Enter cold, raise to 60° C. in half an hour, continue for some time. The effect is to increase the tenacity by 10.2 and the elasticity by 23.3 per cent. A second passage through pyrolignite of iron decreases these properties by 3.1 and 3.8 per cent. respectively.

The treatment which follows, aims at increasing the weight and preventing direct contact of the iron with the logwood, as this leads to undesirable lustrous precipitates on the fibre. It consists in a passage through a weak sumach bath, and the silk is then dyed in a bath containing 50—60 per cent. of logwood and 40 per cent. of soap at a temperature not exceeding 50° C. for some hours, or it is steeped overnight. The sumach and logwood baths increase the tenacity by 11.8, the elasticity by 9.8 per cent.

Brightening completes the process. An emulsion consisting of 8 per cent. of olive oil and 8 per cent. potassium carbonate (on the weight of silk) is poured into a bath containing 12 per cent. of crude acetic acid. The silk is worked a quarter of an hour in this bath. The tenacity is thus decreased by 8.1, the elasticity by 3.6 per cent.

During the whole process the silk loses 28.2 per cent. of its tenacity and 32.5 per cent. of its elasticity.—R. B. B.

*The Crystallisation of Cellulose and the Chemical Composition of the Vegetable Cellular Membrane.* E. Gilson. *Bull. Soc. Chim.* 11—12, 1894, 590.

See under Analyt. and Scient. Notes, page 1106.

## PATENTS.

*Improvements in Machinery for Cleaning Cotton Seed.* J. Bibby, Liverpool. Eng. Pat. 21,372, November 10, 1893.

See under XII., page 1070.

*An Improved Process for the Retting, Cleansing, and like Treatment of Textile Fibres.* A. Gruschwitz, Neusalz-on-Oder, Germany. Eng. Pat. 15,140, August 8, 1894.

This relates to an improvement upon the invention described in Eng. Pat. 3506 of 1893, in which the boiling operation, in the retting, cleansing, and deoxidising of textile fibres, is described as being performed at a temperature not exceeding or under 100° C. The present improvement consists in working at a temperature as high as 115° C., which is found necessary for treating certain materials.—S. P. E.

## VI.—DYEING, CALICO PRINTING, PAPER-STAINING, AND BLEACHING.

*The Chlorinating of Wool.* A. Balard. *Monit. Scient.* 42, 1891, 333—336.

Most previous writers on this subject have insisted on the fact that a prolonged action of chlorine on wool is to be avoided, as it imparts to the fibre a yellowish colour and a harsh, unpleasant feel. These authors generally state also that a once-used chlorine bath can be strengthened for further use by the addition of an amount of hypochlorite less than the original quantity. These two statements the present author denies. The chlorinating of cotton is a gradual and progressive action; not so, however, with wool, where the action is very rapid, the whole of the chlorine being absorbed by the wool in at most two minutes, and strengthening of old liquors is quite useless. The experiments on which these conclusions are based were made with a piece of woollen fabric weighing 20 grms., which had previously undergone the operations of soaping, stoving, washing, &c. A solution was prepared containing 5 grms. of sulphuric acid and 12 cc. of hypochlorite of soda (corresponding to 0.6 gm. of dry bleaching powder of good quality) in one litre of water. One volume of such a solution immediately decolorises one volume of a solution of indigo in sulphuric acid, so diluted that its colour is just visible. The wool is steeped in the chlorine bath for one minute, and, after removing it, the bath no longer decolorises indigo solution, showing that all chlorine has been removed by the wool. Sometimes, indeed, half a minute suffices for the removal of the chlorine. Another 12 cc. of hypochlorite are added to the bath and the wool re-entered for a minute; all chlorine has again disappeared from the bath, and this may be several times repeated, always with care that an excess of acid be present. After three or four operations the wool acquires a yellowish tint and a harsh feel. Even when the hypochlorite bath is four times as strong as the above (eq. to 12 per cent. of bleaching powder on the weight of wool), in two minutes every trace of chlorine will have been removed by the wool. The essential point for consideration is evidently the relative proportions of chlorine and wool, rather than the time of action. The best proportion is 2–5 per cent. of bleaching powder, or its equivalent. If calcium hypochlorite be used the acid must be hydrochloric, with sodium hypochlorite either hydrochloric or sulphuric; an excess of acid must always be present. As hydrochloric acid tends to render the wool yellow when used for this purpose, the employment of sodium hypochlorite with sulphuric acid is to be preferred. Chlorates are not here available as the source of chlorine. The acid bath may precede, or follow, the chlorine bath; preferably the former.

A mechanical difficulty to be overcome is that of rendering as even as possible the absorption of chlorine by the wool. If dyed in chain form, those portions reaching the liquor first absorb too much chlorine, while the latter portions receive little or none. It is better therefore to dye in the open width, making use of a frame similar to that employed in vat-indigo dyeing; the parts of the frame must be constructed of some material capable of resisting the prolonged action of chlorine.

The rapid removal of chlorine from the bath might have been attributed to the action of the sulphurous acid present in the stoved wool, but for the striking result of an experiment carried out with a piece which had been stoved but not subsequently washed. This piece was steeped in the acid bath, then in sodium hypochlorite, and finally in a second bath containing sulphuric acid. In the last bath a considerable evolution of sulphur dioxide occurred. On washing, the wool was found to be satisfactorily chlorinated. Evidently the sulphuric acid and hypochlorite reacted to produce chlorine, and a certain amount of the liberated soda combined with the sulphurous acid to form sodium sulphite, this being decomposed in the second acid bath with liberation of sulphur dioxide. The satisfactory result of the chlorination indicates that, in the presence of wool and sulphurous acid, chlorine is more readily absorbed by the former than rendered inactive by the latter.—R. B. B.

*The Cleaning and Dyeing of Cattle- and Horse-Hair, and of Pigs' Bristles.* M. Ganghofer. *Farb. Zeit.* 5, 366—368.

Before making use of cattle- and horse-hair, they must be thoroughly washed. Cattle-hair should be steeped in cold water for 1–2 days, the treatment repeated with fresh water, and the hair then washed with hot water. Horse-hair, if from the tail, is generally less dirty, and washing in hot water suffices; but if from the mane, the steeping is also necessary. In many places a police order is in force, according to which horse-hair must be disinfected by warm hydrochloric acid, as the grease upon the hair, in a state of decomposition, has caused blood poisoning among the workmen. Pigs' bristles are steeped in cold water, and then washed in cold running water. The hair from the sides of pigs (pigs' wool) can be put directly into the dye-bath without washing. For stuffing purposes these various kinds of hair are generally dyed black by the process to be described. After washing, they should be at once dyed, or ammonia may be formed, which acts injuriously on the colour and on the other properties of the hair. Pigs' wool and bristles are the most easily dyed and require the least dyestuff; cattle-hair and that of the horse's tail require more dye and longer boiling; hair from the mane must be boiled long and carefully, since it is easily matted. Goats' hair, camels' hair, and other similar products may also be dyed according to the following method:—

*The Black-dyeing of Hair.*—The hair is packed very closely in the dye-bath. For every 100 lb. of hair, 3 lb. of solid logwood extract and  $\frac{3}{4}$  lb. solid fustic extract are dissolved in water and the solution poured into the bath. 3 lb. ferrous sulphate are dissolved and this solution also poured in. The bath is then filled up with water, and the hair pressed tightly down by means of boards weighted with stones, so that it cannot rise during boiling. The bath is raised to the boil; pigs' wool and bristles are boiled for 2–2½ hours, cattle- and horse-hair (tail) 3 hours, and horse-hair from the mane, at least 4 hours. At the end of this time allow the goods to remain 1–2 hours in the cooling dye-bath. The liquor is then run out, and, except for pigs' wool and bristles, the shade is made darker by working in a weak solution of potassium bichromate at about 80° C. for  $\frac{3}{4}$  hour. Allow to cool, and wash. It is most economical to use as large dye-vessels as possible, holding, for instance, 1,000–1,200 lb. If not more than 300 lb. are dyed in one lot, 2½ hours boiling is sufficient for any variety of hair. In every case the material should be allowed to remain some time in the dye-bath after boiling.—R. B. B.

*The Practical Extraction of Tannin from Myrabolams.* Von Schroeder. *Dingl. Polyt. J.* 292, 213.

See under XIV., page 1073.

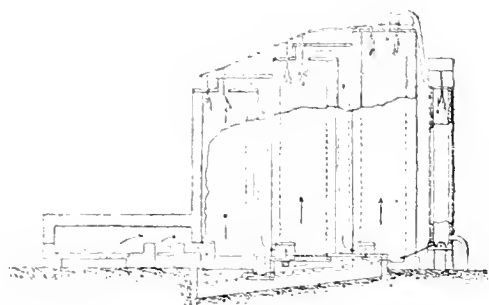
## VII.—ACIDS, ALKALIS, AND SALTS.

*Improvements in the Sulphuric Acid Industry.* P. de Boissieu. *Bull. Soc. Chim.* 1894, 11—12, 726—729.

THE large capital involved in providing sulphuric acid plant, with extensive lead chambers, is commented on, and it is stated that the smallest chambers in use, producing 1,500 kilos. of acid daily, cost 70,000 francs; while plants turning out 5,000 to 10,000 kilos. cost from 100,000 to 200,000 francs. The problem of manufacturing sulphuric acid with a much greater economy of space and capital, appears, it is said, to have been solved by E. J. Barbier, while the produce is not lessened, nor the cost of production increased.

The three reaction towers shown (Fig. 1) in the smallest apparatus (for production of 800 to 1,000 kilos. of acid per day) are respectively 5, 6, and 7 metres high, and  $1\frac{1}{2}$  m.

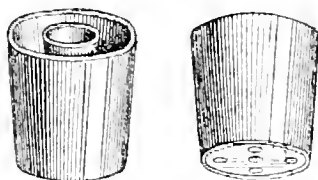
Fig. 1.



THE BARBIER APPARATUS.

square in the base. They are lined externally with sheet lead 3 mm. thick, to prevent leakage of gases. The interiors are filled with special cells (Fig. 2), made, like the towers,

Fig. 2.



of acid-resisting material, which give a surface of about 70 superficial metres for each cubic metre occupied. The oven at the base of the towers carries the concentrating basins, arranged *en cascade*, in which the sulphuric acid is concentrated as it forms, and flows continuously from the lowest basin at  $58^{\circ}$ — $60^{\circ}$  B. The sulphurous acid gas from the pyrites burner, after having heated the space below the basins, enters the upper part of a special tower, where it is washed by strong sulphuric acid, free from nitrous products, and is thereby freed from dust. The gas then circulates through the three towers, and lastly enters the Gay-Lussac column terminating the apparatus. The first tower is washed with water and dilute nitric acid; the other towers by concentrated acid. Thus, in each part of the system sulphuric acid is continuously produced and flows to the base of each tower, whence it overflows into the concentrating basins, in which denitration simultaneously goes on, so that water vapour and nitrous products return to each

tower to aid the reactions. The acid produced does not come into contact with lead, and is not soiled, like chamber acid.

It is a feature of the Barbier apparatus that the temperature of the tail part is always over  $50^{\circ}$  C., and that of the first tower over  $90^{\circ}$ , thus having a wider and higher range than the ordinary chambers, the temperature in which varies between  $40^{\circ}$  and  $60^{\circ}$ . It follows that the new apparatus may be established in warm countries without any loss of advantage. As dilute nitric acid does not react on sulphurous acid at ordinary temperatures, but only at between  $60^{\circ}$  and  $80^{\circ}$ , which latter temperature is never attained in the ordinary chambers, it is considered probable that the reactions which produce sulphuric acid in the Barbier apparatus, differ in certain respects from those occurring in the chambers.—E. S.

*Vinegar: its Manufacture and Analysis.* R. T. Thomson.  
A Paper read before the Glasgow City Analysts' Society.  
Abstracts. No. 1, 1893-94, 3.

THE author's special object is to indicate how and to what extent chemical analysis can show from which of the various sources a sample of vinegar has been derived. For this purpose, the percentages of acetic acid, organic matter non-volatile at  $100^{\circ}$  C., ash, potash, and phosphoric acid are determined. The "original solids" are then calculated by Hehner's method, viz., multiplying the acetic acid by 1.5 and adding the result to the total solids.

The first column in Table II. shows the composition of undistilled vinegar of average strength prepared from pure malt, the figures being based upon analyses of malt extracts prepared by the author, as well as of genuine commercial extracts. To produce a vinegar of this composition, a malt infusion of sp. gr. 1.035.5, containing 9.2 per cent. of total solids, would have to be fermented to a specific gravity of about 1.000, which is the usual practice. During acetification, the specific gravity would gradually rise to about 1.014, if no loss took place, and the total solids would decrease to about 1.7 per cent. The relations of the ash, potash, and phosphoric acid to the original solids, and especially the relative proportions of potash and phosphoric acid, would be practically unaltered by the fermentation; these are therefore the most reliable data, as they do not vary with the strength of the vinegar. Table I. gives these data for vinegars prepared from various materials, and it will be observed that in the sugar and molasses vinegars the phosphoric acid forms a very small proportion of the ash. As the percentage of ash in sugar and molasses varies largely, the comparison with malt must be made on the composition of the ash. To distinguish wine vinegar, the presence or absence of tartaric acid is a valuable criterion; and the percentage of nitrogen is useful in discriminating between starch-glucose vinegar and other vinegars.

The value of the phosphoric acid estimation has been impugned on the ground that if a hard water were to be used in washing the malt, the phosphoric acid would be precipitated as phosphate of lime. The author has, however, calculated that in mashing, conducted on the

TABLE I.

Source of Vinegar.	Pure Malt.	Mixed Grain	Cane Sugar.	Commercial Beet Sugar.		Cane Molasses.	Beet Molasses.	
				1st Quality.	2nd Quality.			
Per Cent.								
In original solids:—								
Mineral matter.....	1.93	2.00	1.40	0.89	2.08	6.90	13.17	15.26
Potash .....	0.70	0.71	0.40	0.26	0.80	4.06	6.91	8.74
Phosphoric acid.....	0.75	0.54	0.08	Trace	Trace	0.06	0.06	0.07
In mineral matter:—								
Potash.....	36.27	35.59	28.57	29.21	38.15	57.98	52.46	57.27
Phosphoric acid .....	38.86	27.00	5.71	Trace	Trace	0.87	0.45	0.45

strength of wort mentioned above, a water having at least 120 of hardness would be theoretically required to remove all the phosphoric acid. It is unlikely that water with more than 30 of hardness would be used, involving the possible removal of one fourth of the phosphoric acid; but even against this must be set the appreciable acidity of the wort, which would probably be sufficient to more than neutralise the alkalinity of the water. But rather than set up too strict a standard, the figures given for mixed grain might reasonably be adopted for undistilled malt vinegar.

TABLE II.

	Malt Vinegar.	1.	2.
		Per Cent.	
Acetic acid .....	5.00	6.12	5.35
Non-volatile organic matter.	1.52	2.30	0.009
Mineral matter .....	0.18	0.27	0.004
Total solids .....	1.70	2.66	0.013
Potash .....	0.066	0.054	Nil
Phosphoric acid .....	0.050	0.084	Nil
Original solids .....	9.20	11.84	8.05
Mineral matter in ditto .....	1.93	2.28	0.95
Potash in ditto .....	0.70	0.60	Nil
Phosphoric acid in ditto .....	0.75	0.68	Nil
Potash in mineral matter .....	33.27	21.93	Nil
Phosphoric acid in ditto .....	38.86	29.00	Nil
Colour .....	..	Brown	Colourless

Referring to the typical analyses in Table II., although No. 1, from the percentages of potash and phosphoric acid in the ash, is of doubtful source, there is no warrant for saying it is not a pure malt vinegar, as there is not a wide enough range of facts to establish an essential difference between it and genuine malt vinegar. No. 2, however, cannot possibly be undistilled malt vinegar, nor can it be fully fermented undistilled sugar vinegar, as in that case it would yield at least 0.3 per cent. of "total solids" consisting of succinic acid and glycerin. The conclusion is inevitable that it is a distilled vinegar, but from what source it has been distilled is not known. Some chemists have regarded the absence of any trace of alcohol in a distilled vinegar as a proof that the sample cannot have been made by fermentation, but must consist of wood vinegar; this is, however, founded on the mere assumption that alcohol cannot be perfectly fermented. According to a decision of the Glasgow Sheriff, distilled malt vinegar is as truly "malt vinegar" as the original article.—L. A.

*The Estimation for Industrial Purposes of Boric Acid in Boronatrocalcites.* G. A. Le Roy. *Monit. Scient.* 42, 1894, 522.

See under XXIII., page 1094.

#### PATENTS.

*Improvements in Apparatus for Concentrating Sulphuric and other Acids.* E. Dyson, Manchester. Eng. Pat. 17,699, September 20, 1893.

THE concentrating vessels are constructed of cast-iron lined internally with an acid-resisting enamel, and are set in an inclined chamber and arranged generally in the fashion now well known in connection with "cascade apparatus." The fire-gases pass from the ascending chamber containing the stills or concentrating vessels, into the upper flue in communication with the chimney, thereby heating the

supply tanks for maintaining the supply of acid to the stills below. To connect the concentrating vessels one with the other, the use of siphons constructed "with a double curve or double U form to deliver acid from the bottom of one vessel to the top of the next," is claimed, as also a suitable float valve with hollow stem extending up the inlet-pipe, the bottom end of which corresponds with the shape of the valve described in the specification.

There is also claimed the combination of the cooler and cooling-vessels described, with the furnace and concentrating vessels.—E. S.

*Improvements relating to the Manufacture of Alkaline Carbonates and Silicates.* J. Reich, Vienna. Eng. Pat. 18,139, September 27, 1893.

SODIUM or potassium chloride solution (or other soluble alkali salt) is treated with fluosilicic acid, and the dried precipitate is heated with silica in a clay retort, to obtain the alkali metal as a silicate, and silicon fluoride gas, which is absorbed by water, to form fluosilicic acid and silica for re-use. If the alkaline silicate is not intended to be used as such, it is dissolved in water and dosed with carbonic acid, to form a carbonate; the silica which precipitates is collected for use in the described process.

The process may be modified by using "boron hydrofluoric acid or hydrofluoric acid for effecting the double decomposition into an alkaline boron fluoride or an alkaline fluoride," the fixed product being, as before, an alkaline silicate, and the volatile products boron fluoride and silicon fluoride, or the latter only, as the case may be. Details are given. In this process, after forming an alkali fluoride, this may be retorted with alumina, instead of with silica, the reaction being thus represented, " $AX$ " standing for the alkali metal:— $12AX + 2Al_2O_3 = Al_2F_6 + Al_2(OH)_6$ , the products being an artificial cryolite and an aluminat. By lixiviation, an alkaline aluminat is dissolved, and the solution, treated with carbonic acid, yields an alkaline carbonate in solution, and a precipitate of aluminium hydroxide.—E. S.

*Improvements in Lime Kilns.* B. Dawson, Malvern Link. From Quarez Frères, Villeneuve-sur-Verberie, France. Eng. Pat. 20,791, November 2, 1893.

See under IX., page 1062.

*Improvements connected with Apparatus for the Manufacture of Bleaching Powder (Chloride of Lime) or similar Commodities.* J. M. Milnes, St. Helens, and A. Milnes, London. Eng. Pat. 21,356, November 9, 1893.

THE inventors employ spear heads or conical tips to the rakes used for stirring the material to be impregnated with chlorine gas, in order to prevent adherence of the pasty substance, and that every particle of lime may have similar exposure. These improved rakes are to be used in combination with other appliances described in Eng. Pat. 15,833, 1891 (this Journal, 1892, 967), and in Eng. Pat. 19,180, 1892 (this Journal, 1893, 263).—E. S.

*Improvements in the Utilisation of the B. An Bricks used in the Deacon Chlorine Process, and the Recovery of Copper therefrom.* J. R. Wyde, Pres., and D. Williams, St. Helens. Eng. Pat. 21,412, November 10, 1893.

THESE bricks become useless after a time, owing to formation in them of cupric sulphate, which is inactive for the decomposition of hydrochloric acid. According to the present invention, the spent bricks are lixiviated, preferably by water containing about 5 per cent. of hydrochloric acid. The copper solution thus obtained is precipitated with iron in the usual way, and the washed bricks are dried, and are then again available for use.—E. S.

*Paper on the Preparation and Manufacture of Hydrosulphurous Acid and Hydrosulphites.* J. Grossmann, Manchester. Eng. Pat. 16,967, August 23, 1894.

SOLUTION of sodium mono-, bi-, or metasulphite, or a mixture of these, of such strength as to contain from 15 to 25 per cent. of total sulphurous acid, is placed in a closed vessel fitted with an agitator, and having a pipe reaching nearly to the bottom. About a tenth of the bulk of the liquid, of 10 per cent. sulphuric acid, is introduced by the pipe, and, after agitation, an excess of zinc dust or other suitable reducing agent is quickly added, followed by agitation, with the usual precautions as regards temperature and exclusion of air. When the reagents are exhausted, fresh portions are added in like manner successively, until about half the bulk of the original liquid in dilute sulphuric acid has been introduced. The reagents are then added in smaller portions, until suitable tests show that the maximum of hydrosulphurous acid has been formed. The proportions thus once determined may be adhered to afterwards. To ascertain the point when the maximum quantity of hydrosulphurous acid has been formed, Berthsen's test as modified by Rawson is recommended. The process may be applied in the case of other than sodium sulphites, and even with difficultly soluble sulphites, as of calcium or zinc, which are held in suspension in water, instead of in solution. Also, dilute sulphuric acid may be replaced in the process by solution of an acid salt, as of sodium bisulphate.—E. S.

## VIII.—GLASS, POTTERY, AND ENAMELS.

*The Weathering of Glass and its Decomposition by Water.* F. Foerster. Zeits. anal. Chem. **33**, 1894, 322—335.

From a long series of experiments of his own on these subjects, and from the work of others, the author draws the following conclusions:—

1. The weathering of glass is caused by the decomposing action of the atmospheric moisture. The carbonic anhydride of the air does not act directly on the glass, but only on the alkaline products of the aqueous decomposition.

2. Dry carbonic anhydride is without action on dry glass.

3. There is no proof that water can be retained by glass, except when it enters into chemical combination therewith.

4. The weathering of glass and the decomposition of glass by water are similar processes, and are both preceded by the taking up of water into the glass molecule.

5. The surface changes caused by weathering are comparatively slight with good glass.

6. The action of water on weathered glass is only temporarily more rapid than it is on new glass.

7. Glasses (lime glasses) are more hygroscopic and weather more easily, the more easily they are attacked by water.

8. Even after long action of water, glass is still capable of becoming weathered.

The following table gives the results obtained. The glass used had the composition  $\text{SiO}_2 = 69.4$ ,  $\text{CaO} = 7.0$ ,  $\text{Al}_2\text{O}_3 = 3.0$ ,  $\text{MnO} = 0.4$ ,  $\text{K}_2\text{O} = 7.1$ ,  $\text{Na}_2\text{O} = 13.1$ . The glass was in each case treated with water at  $20^\circ$  for one minute, and the amount of alkali removed estimated. The glass was then put in fresh water and left therein for 24 hours, when the water was again renewed, and this was repeated each day:—

Alkali removed by Water at  $20^\circ \text{C}$ .  
In Thousandths of a Milligram per 100 sq. cm. exposed.

Length of Time of Treatment with Water.	From the New Glass.	From Glass subjected to Moisture at $30^\circ \text{C}$ . for Six Weeks.	From Glass subjected to Moist Carbonic Acid at $20^\circ$ for Six Weeks.	From Glass subjected to Weathering for a Year in an Ordinary Room.	From Glass subjected to Weathering for a Year in the Open Air.	From Glass subjected to Weathering in a Room for One Year, and subsequently to Moist Carbonic Acid at $30^\circ \text{C}$ . for Six Weeks.
In one minute.....	34	65	79	48	66	109
During first day.....	63	62	10	50	45	13
" second day.....	19	..	7	17	18	6
" third day.....	14	17	6	15	15	7
" fifth day.....	14	14	..	12	12	6
" seventh day.....	11	9	..	9	8	..
" tenth day.....	8	8	..	8	8	7

—L. T. T.

*The Action of Acids on Glass.* F. Foerster. Zeits. anal. Chem. **33**, 1894, 299—322.

In order to complete his own and Mylius' work on the action of water, of alkalis, and of saline solutions on glass, and to control the work of others on similar lines, the author has carried out a long series of investigations on the action at different temperatures of acids of different strengths on glasses of different composition. From his own results and those of others, he draws the following conclusions:—

1. With alkali-lime glass as generally employed for chemical apparatus, and with lead glasses not richer in

lead than is crystal glass, the action of aqueous acids is less than that of pure water, is independent of the nature and (within certain limits) the concentration of the aqueous acid, and is due to the water contained therein; and the action of the acid consists almost solely in neutralising the alkali dissolved out by the water.

2. With glass rich in lime and with flint glass very rich in lead, aqueous acids exert a strong action, which is dependent on the nature and the concentration of the acid.

3. On ordinary lime glass boiling strong sulphuric acid has less action than boiling water.

4. At high temperatures sulphuric acid vapour attacks glass strongly, forming a coating of alkali sulphates which produce a deep-seated change in the surface of the glass.

The following tables give the principal numerical results with the various kinds of glass experimented on.

## ANALYSES OF GLASSES TESTED

Designation of Glass.	K <sub>2</sub> O.	Na O.	CaO.	MnO.	PbO.	Al <sub>2</sub> O <sub>3</sub> 100	SiO <sub>2</sub>	B <sub>2</sub> O <sub>3</sub> CaO. Soda
A	1.3	10.0	7.8	Trace	..	0.3	77.6	1.18 : 1 : 9.28.
B	2.7	9.0	6.8	Trace	..	0.1	74.1	2.04 : 1 : 10.17.
C	6.7	13.7	7.2	0.3	..	3.2	68.9	2.27 : 1 : 8.79.
D	5.5	13.3	5.8	0.1	..	2.5	72.5	2.64 : 1 : 11.96.
E	11.7	4.1	8.1	..	..	0.5	75.6	1.72 : 1 : 8.71.
F	1.8	12.9	11.0	..	..	1.3	73.0	1.16 : 1 : 6.2.
G	0.7	12.3	11.2	..	2.2	1.7	69.4	.. ..
H	12.7	..	..	..	3.0	..	57.3	.. ..
I	7.5	0.5	..	0.1	33.8	..	53.9	BaO = 1.0; As <sub>2</sub> O <sub>3</sub> = 0.2.
16 III.	..	14.0	7.0	..	..	2.5	67.5	B <sub>2</sub> O <sub>3</sub> 2.0 ZnO 7.0.
59 III.	..	11.0	..	0.05	..	5.0	71.95	B <sub>2</sub> O <sub>3</sub> 12.0.

## Action of Acids at 100°.

Nature of Acid.	Concentration.	Loss of Weight in Milligrams per 100 Sq. Cm. Surface exposed.					
	(Gram equivalents per litre.)	Glass A.	Glass B.	Glass C.	Glass G.	Glass H.	Glass I.
Sulphuric acid.....	0.001	0.2	1.1	1.7	..	..	..
" .....	0.1	0.1	1.0	1.5	..	..	..
" .....	1.0	0.2	1.2	1.7	..	1.2	..
" .....	5.0	0.2	1.0	..	..	..	..
" .....	10.0	0.3	0.9	1.7	..	..	..
" .....	25.0	0.1	0.2	0.5	..	..	..
" .....	Pure acid of sp. gr. 1.84	0.1	0.1	0.2	..	..	..
Nitric acid .....	0.1	0.2	1.2	1.7	..	..	..
" .....	1.0	0.	0.7	1.8	..	..	..
" .....	5.0	0.1	1.0	1.5	..	..	..
" .....	10.0	0.1	1.2	1.5	..	..	..
" .....	16.5	0.2	..	1.0	..	..	..
Hydrochloric acid .....	0.001	..	..	1.5	..	..	..
" .....	0.01	..	..	1.5	..	..	..
" .....	0.1	0.2	0.9	1.6	..	..	..
" .....	0.5	..	..	1.8	..	..	..
" .....	1.0	0.3	1.1	1.9	0.3	1.2	1.4
" .....	2.0	0.3	..	1.8	..	..	..
" .....	1.0	..	..	1.7	..	..	..
" .....	5.0	..	..	..	..	1.3	..
Acetic acid .....	0.1	0.2	0.8	1.6	..	..	..
" .....	1.0	0.2	0.8	1.7	..	0.9	..
" .....	5.0	0.1	..	1.4	..	..	..
Pure water .....	.. ..	1.5	4.9	6.0	1.0	2.0	1.9



*Action of Acids of Varying Strengths and at Different Temperatures.*

Loss of Weight in Milligrams per 100 Sq. Cm. exposed.

Glass.	Temperature.	Decca-normal.			Normal.			Penta-normal.			Decca-normal.			Water.
		HCl.	H <sub>2</sub> SO <sub>4</sub> .	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> .	HCl.	H <sub>2</sub> SO <sub>4</sub> .	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> .	HCl.	H <sub>2</sub> SO <sub>4</sub> .	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> .	HCl.	H <sub>2</sub> SO <sub>4</sub> .	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> .	
D	160	14	12	13.7	14.6	14.9	14.5	9.2	11.6	11.2	5.0	8.7	8.0	41
D	180	..	..	..	90.3	94.6	94.1	..	..	..	10.0	17.0	20.0	428
E	180	..	..	..	4.8	8.8	8.4	..	..	..	4.6	6.0	5.8	50
F	180	..	..	..	2.6	2.1	2.3	..	..	..	..	2.4	1.4	43
16 Ltl.	..	..	..	..	16.0	10.0	3.0	21.0	8.0	..	24.0	4.0	2.0	34
59 Ltl.	..	..	..	..	15.0	15.0	3.0	..	..	..	3.0	4.0	..	39

Experiments with Wollastonite, Labradorite, and other silicates (and even some varieties of glass) showed that when the proportion of base to silica is much larger than in the usual varieties of glass the action of acids is much greater than that of water.—L. T. T.

## PATENTS.

*Improved Apparatus for Manufacturing Pottery.* J. Slack, Nottingham. Eng. Pat. 18,023, September 26, 1893.

A THROWING-WHEEL with a hollow vertical shaft is provided with a disc, to which are attached jointed vertical arms controlling the movement of the plates used in moulding the ware. A second disc is attached to a second vertical shaft working within the main shaft, and carries the joints on which the plates are hinged. Similar internal plates serve to mould the inner side of the ware. A lifting-rod is provided for removing the article from the mould, after its ornamentation by means of the plates, has been effected.—B. B.

*Improvements in Ovens for Baking or Firing Ceramic Ware and other Refractory Products.* J. B. Hanquiaux, Charleroi, Belgium. Eng. Pat. 22,637, November 25, 1893.

See under I., page 1048.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

## PATENTS.

*Improvements in Cement Kilns.* E. de Pass, London. From L. E. A. Lavocat and E. L. Candlot, Paris, France. Eng. Pat. 19,259, October 13, 1893.

THE patent relates to the utilisation of the waste heat from cement kilns, particularly those of the continuous type, for drying the raw materials, whether in the form of slurry or bricks. Two drying chambers are connected with the exit flue of the kiln in such a manner that the waste gases can be sent through either at will. The tops of the drying chambers may also be used for drying a further portion of slurry. The arrangement, although primarily intended for continuous kilns, may be utilised for intermittent kilns of the ordinary English pattern.—B. B.

*An Improved Hoffman or Continuous Kiln for Drying and Burning Bricks and other Goods.* R. Cook, Kensal Rise, Middlesex. Eng. Pat. 19,848, October 21, 1893.

See under I., page 1047.

*Improvements in Lime Kilns.* B. Dawson, Malvern Link. From Quarez Frères, Villeneuve-sur-Verberie, France. Eng. Pat. 20,791, November 2, 1893.

GAS from a separate producer, preferably with water bottom, is introduced under pressure through suitable channels into peripheral openings in the lining of a kiln of circular section, and through other ports in a smaller central chamber, below, and concentric and continuous with, the first. Air is supplied below every gas port by natural draught, and both gas and air flues are provided with dampers, so that the supply of each to any point is under control. Thus combustion is complete, and heat distribution uniform. The discharging doors for the burnt lime are hermetically sealed when not in use.—W. G. M.

*Improvements in the Manufacture of Finely-pulverised Substances for Use in the Production of Portland Cement, Pigments, and the like.* A. Patrick, Glasgow. Eng. Pat. 21,292, November 9, 1893.

See under I., page 1048.

*Improvements in the Manufacture of Cement Mortar.* M. Williams, Liverpool. Eng. Pat. 22,227, November 21, 1893.

ABOUT eight parts of furnace ashes, slag, or coke, four parts of slaked lime, and one of clay, are taken and mixed dry so as to form a cement, which, on mixing with water, sets in the ordinary way. The proportions of the materials may be varied so as to produce either an aerial or hydraulic cement.—B. B.

*Improved Process for Manufacturing Hydraulic Cement.* G. W. A. Stein, Wetzlar, Germany. Eng. Pat. 5971, March 10, 1894.

BRICKS or blocks of raw materials for cement-making are usually mechanically weak, and have to be protected from weather and be artificially dried before being burnt to clinker. The present patent prescribes the addition of 5–10 per cent. of a slag cement of the usual composition (e.g., 75 per cent. of granulated slag and 25 of slaked lime) to the raw materials, in order that the bricks may be strong enough for convenient handling, prior to burning.—B. B.

**Improvements in Kilns.** H. J. Haddan, London. From The Columbian Pottery and Brick Kiln Company, Chicago, U.S.A. Eng. Pat. 2516, May 15, 1894.

THE improved kiln is annular in shape and surrounds a central smoke-stack. The floor is divided into five (or more) equal radial sections, and being mounted on rollers is capable of being rotated bodily, but independently of the walls and roof. On one side of the kiln three movable partitions serve to separate two of the sections from the remainder of the kiln and from one another, and each of the two sections thus separated has a door for charging or discharging; the outer partitions are formed of two iron sheets with asbestos between them; the other is of wood. On the opposite side of the kiln a vaulted fire-brick combustion chamber is built over the roof of the central section, and apertures conduct the heated products of combustion downwards into the kiln; over the sections on either side of this, similar chambers of ordinary brick are erected, one with a perforated roof the other plain, but only the central section is fired. Below the floor level are two apertures connected with the smoke-stack, one corresponding with the firing section, and one with the adjoining section, which has the plain roof. The whole of the annular floor is perforated, the perforations communicating with interlaced flues beneath, which flues are arranged in five equal sections independent of one another, and corresponding to the sections of the kiln. The flues independently converge at five apertures symmetrically arranged on the inner side of the ring, and two only of the systems are therefore in communication with the smoke-stack by means of the apertures above alluded to. In use, while one section is being charged, the next to it is being discharged through the two openings provided, and air-holes in the roof at this side of the kiln allow free ventilation. The partitions are then raised and the floor is turned through one-fifth of a revolution, so that each section is moved on, and the partitions are again lowered, charging of the section emptied the previous day, and discharging of the newly-exposed section taking place as before. The chamber, previously being heated by radiation, has now passed into the firing section, and that which was fired is now cooling ready to be discharged at the next shift. The air used for combustion is preheated by radiation from the cooling section of the kiln. The floor of the kiln is shifted once in 24 hours.—W. G. M.

**Improved Method of Vulcanising and Drying Wood.** C. Howard, New York City, U.S.A. Eng. Pat. 15,231, August 9, 1894.

THE wood is enclosed in an air-tight cylindrical chamber, from which the air is then exhausted; a gentle heat is employed, which, aided by the reduced pressure, causes vapourisation of the moisture in the wood and expansion of the air within its pores. Air pressure is next applied and the heating continued; and finally the wood is cooled, air being meanwhile circulated through the cylinders and through a condenser, for the removal of moisture.

—W. G. M.

**Improvements in Plastering for Ceilings and Partitions.** E. H. Jung, Daggens Road Station. Eng. Pat. 15,589, August 16, 1894.

THE patent relates to the production of sheets of plastering material, about two square yards in size, from various mixtures of water, slaked lime, tar, shavings, sawdust, "sifted kiln refuse," cow-hair, heather, cocoa-nut fibre, and plaster of Paris, made into a paste with water and cast into slabs in suitable moulds. An india-rubber sheet is placed on the table used for casting, so that the mixture is spread thereupon and can be subsequently removed. The mixture sets in about 20 minutes, and is ready for use in two or three weeks. It can be nailed to the joists of ceilings and be coated with ordinary plaster to make good the joints between the slabs.—B. B.

**Improvements in and relating to Damp-resisting Materials.** R. J. Goddard, London. Eng. Pat. 15,756, August 18, 1894.

A MIXTURE of asphaltic or bituminous materials is prepared and cut into strips or sheets on a slab or table, so as to be in a form suitable for use as a damp-proof course, in building construction. A lining or backing of canvas or other fabric may be applied to the strip of composition so as to form a portable material which may be laid in place to form a damp-proof course.—B. B.

## X.—METALLURGY.

**Desulphurisation. The Action of Iron Sulphide on Manganese, Aluminium, Nickel, Chromium, &c.** Bull. Soc. Chim. 11 12, 1894, 583—587.

THE Soc. Chim. de Paris, through its *Bulletin*, has previously promoted a discussion relative to the desulphurisation of the products formed during the Saniter process, with the object of determining whether the reaction—



is possible in the absence of a reducer such as carbon. The facts collected do not justify any absolute conclusion. Ledebur in *Stahl und Eisen* gives the arguments on both sides and suggests that a purification by means of a basic calcareous slag is not indispensable to the verification of the said reaction. Another explanation is possible, namely, that the iron sulphide dissolves in the basic slag. There appears no reason why this substance should not dissolve without decomposition. Further, the dross of puddlings, even if poor in manganese and without lime, gives off sulphur in the free state. Lime slags can remove sulphur from iron under certain conditions; but, on the other hand, slags containing sulphur, even if basic, give it up to the metal. Thus, an iron rich in sulphur can be purified at high temperatures by a fluid basic slag containing little sulphur, but, on the contrary, an iron poor in sulphur would absorb that element in contact with a slag containing much sulphur and but slightly fluid. The Saniter process has, then, a certain theoretical value if one regards the addition of calcium chloride (its special feature) as merely a means of making the slag more fluid.

The desulphurising influence of manganese can be explained by the fact that manganese sulphide, which is less soluble in metal than iron sulphide, will rise to the surface and is more easily absorbed by basic slags.

The employment of lime slags with manganese is advantageous because the slag becomes more fluid and a more powerful solvent. Up to the present we depend almost entirely on manganese to desulphurise iron. In the Thomas converter the higher the initial percentage of sulphur the greater the proportion eliminated. Experiments made in Pennsylvania show that in remelting in the cupola for the Thomas process the sulphur increases in proportion as the manganese is low, and with less than 0.7 per cent. of manganese one gets a metal containing more sulphur than in the original metal.

Hüfenstock has tried to find out the reciprocal action of sulphur on iron and certain metals, and also the action of sulphurous gases on iron, manganese, and certain metallic oxides. He found that on mixing and heating together iron sulphide and a rich ferromanganese (sufficient to make manganese sulphide) a mottled mass was obtained infusible at a white heat. If, however, the two were melted apart and then mixed, the manganese sulphide separated in the solid form, leaving the metal liquid. By analysis the iron was found practically desulphurised.

By melting separately and then mixing iron sulphide and aluminium, an energetic reaction took place, with partial combustion of the iron and aluminium. A dross rose to the surface, quickly separating from the refined metal. The iron was of fine grain. The dross, at first black and liquid, became a grey powder and gave off sulphuretted hydrogen.

Treated in the same way iron sulphide and nickel, the mass did not separate, a complex sulphide of iron and nickel being formed. Copper acted in a similar way. For chromium a chrome iron containing 38 per cent. of chromium and 3 per cent. of manganese was used. A refined metal containing 10.56 per cent. of chromium and 0.037 per cent. of sulphur, and a heterogeneous dross, chiefly iron sulphide, was obtained. Probably chromium acts less energetically than manganese.

A ferro-silicon containing 15 per cent. of silicon was poured, when melted, into the molten iron sulphide. The mixture separated completely into metal and dross. The ferro-silicon changed but little, and absorbed very little sulphur, which confirms Turner's observation that silicon hinders the absorption of sulphur.

A current of carbon dioxide containing in turn small quantities of (1) hydrogen sulphide, (2) sulphur dioxide, (3) carbon disulphide, was passed over ferromanganese containing 80 per cent. of manganese, and over finely-powdered metal containing 1.5 per cent. of manganese, and 0.06 per cent. of sulphur, heated to a red heat. Coal-gas *per se* was also employed. The following figures show the percentage of sulphur absorbed in each case:—

	H <sub>2</sub> S.	SO <sub>2</sub>	CS <sub>2</sub>	Coal-Gas.
	S per Cent.	S per Cent.	S per Cent.	S per Cent.
Ferromanganese.....	16.04	1.18	2.69	0.96
Metal.....	12.62	1.95	1.09	0.18

Using sulphur dioxide with steel, the absorption was 1.36 per cent. of sulphur, and with nickel 2.08 per cent.

Finally, the gases previously dried by calcium chloride and equally diluted with carbon dioxide, were passed through metallic oxides placed in glass tubes; the percentages of sulphur taken up were:—

	H <sub>2</sub> S.		SO <sub>2</sub>		CS <sub>2</sub>	
	Low Red.	Bright Red.	Low Red.	Bright Red.	Low Red.	Bright Red.
Fe <sub>2</sub> O <sub>3</sub> .....	0.292	0.377	0.566	0.100	11.020	26.350
MnO.....	1.762	5.043	1.270	4.880	7.065	23.370
CaO.....	3.528	0.990	5.050	5.080	0.650	3.960
MgO.....	1.394	0.960	2.244	0.660	0.718	0.530

All the oxides take up sulphur according to the temperature and the gas employed. What the influence of the carbon dioxide is, and in what state the sulphur is retained, afford field for useful research.—A. W.

*The Dressing of Spathic Iron Ore at Allevard.*  
G. Gromier. Bull. de la Soc. de l'Ind. Min. 1893, 465.  
Proc. Inst. Civil Eng. 1894, 118, 62—64.

In the neighbourhood of Allevard in the Dauphiny Alps numerous workings have been opened by Messrs. Schneider and Co. upon veins containing spathic iron ore in sandstones and crystalline schists, the former being of Permian or triassic and the latter of pre-Cambrian age.

The mineral, a magnaniferous spathic ore very much mixed with waste, including particles of the enclosing sandstones and schists, and quartz often very closely aggregated.

The calcination of the coarse material is effected in tall kilns heated by gas, from producers worked with closed ash-pits and water injection. The point of combustion is at about one-third of the height of the stack, the space below forming a hopper for cooling the burnt ore, which gives off its heat to the air employed for combustion. Four kilns, each of the capacity of 25 tons in 24 hours, are used; the consumption of coal is 3 per cent. of the weight of the ore treated. The loss of weight is 28 per cent., the

burnt ore retaining about 2 per cent. of carbon dioxide, care being taken to prevent clinkering by the formation of silicate when the heat is raised too high.

The calcined ore discharged from the kilns is raised by a direct-acting hydraulic lift, worked by a head of 1,309 ft. of water, to a second picking-house, where it is divided by a 130-mm. sieve, and passed over balanced picking-tables. The object of this second hand-picking is to remove the closely intermixed quartz, which can be readily done in the larger pieces, as the ore is rendered friable by the calcination. The smaller-sized stuff is cleaned by crushing and sizing through a 9 mm. wire sieve, and passing the fine powder over a series of wooden rollers, each carrying 72 horse-shoe magnets, which take out the magnetic dust from the ore as it falls, the deposit being continuously removed by a rotating brush behind each row of magnets.

The smaller stuff separated by the first picking-house contains from 25 to 30 per cent. of waste material which can only be removed by washing, but on account of the small difference in density between spathic ore and quartz, 3.76 and 2.60, tolerably close sizing is necessary as a preliminary to jigging. With a consumption of about 120 gallons of water per minute about 120 tons of material can be passed through the dressing machinery in 10 hours, with a loss of weight of 10 to 23 per cent., the proportion of waste being reduced from 25 to 30 per cent. in the rough ore, to 12 per cent. in the clean ore. The washed ore is received in hutches, where the adherent water drains off, and is afterwards loaded into waggons for removal to the calcining furnaces. These are of two different patters, one being vertical and the other inclined, but both are heated by gas made from coal in a separate producer, with forced draught and steam injection. The vertical furnaces are arranged in groups of four in a tower 46 ft. high and 18 ft. in diameter, the fine ore fed in at the top falling in an interrupted stream down a narrow zig-zag passage between inclined walls, in a similar manner to that adopted in the Hasenclever Heibig furnace, where it is subjected to the flame of gas burnt with hot air, together with a free supply of air for oxidation. The slope of the inclined walls varies from 45° above to 30° below, as the ore is found to run down more easily as it becomes drier and hotter. The inclined furnaces resemble those used in Styria for treating small spathic ore, and in Sardinia for calamine. They are in narrow reverberatory furnaces with beds about 65 ft. long and 5 ft. broad, with a strong upward inclination, so that the feeding hopper is about 45 ft. above the fireplace. The output of both classes of furnace is about the same, namely, 25 tons in 24 hours, but the consumption of coal is 6 per cent. of the weight of the calcined ore, or double that required in the kilns. The inclined furnace is considered to be the best, being simpler in construction and more easily managed than the vertical form.

The calcined ore, when drawn from the furnace, is quenched with a jet of water, in order to slake the caustic lime and magnesia formed by the calcination of the dolomitic constituent of the vein stuff. It is then carried by a travelling band to an elevator, which supplies the agglomerating machine. Finely-ground hydraulic lime is then added to the quenched ore, which contains about 12 per cent. of water, in the proportion of five parts of the former and 95 of the latter. The mixture is then moulded in a double compression Couffignal briquette machine, into blocks 140 mm. square and 120 mm. long, weighing 15½ lb. each, under a pressure of about 4 tons per square inch. The briquettes, after drying for a week on a platform over the ash-pits of the gas-producer, are sufficiently coherent to bear a pressure of 200 lb. per sq. in., and are removed by railway.

The motive power required for the different operations is as follows:—

	H.P.
1. First-sizing and hand-separator.....	12
2. Magnetic separator.....	8
3. Sizing and washing small ore.....	45
4. Briquette machines.....	40
5. Hoists for kilns and washed ore.....	5
	110

*The Manufacture of Open-Hearth Steel in Sweden.*  
Erik G. Östeltjärna. Trans. of the Amer. Inst. of Mining  
Eng. 1894; Proc. Inst. Civil Eng. 1894, **118**, 64-67.

Only three kinds of open-hearth steel are made in Sweden:—

1. The softest steel, containing 0.15 per cent. or less of carbon;
2. Tool steel, containing 0.45 per cent. or more of carbon; and
3. Steel castings.

Steel containing between 0.20 and 0.40 per cent. of carbon is seldom made, except at works which manufacture heavy plates. Of these, however, a considerable quantity is exported to England and other European countries.

*Soft Steel.*—In making the softest steel, pig iron as free as possible from sulphur is selected, this impurity being under no circumstances allowed to exceed 0.015 per cent. if the best quality of steel be required. The scrap used is as free as possible from rust, which in most cases contains a considerable amount of sulphur taken up from the coal smoke of factory chimneys. The finer scrap is often cleaned in a rotating apparatus. The author describes the process of manufacture as such that, in the case of the toughest Swedish soft open-hearth steel, the percentage of sulphur and oxygen in the metal at the end of the melt shall be so small that no ferro-manganese, or at most only a few tenths of 1 per cent., need be mixed with it. One fourth of 1 per cent. of manganese decreases the toughness of the steel more than an increase of phosphorus from 0.020 up to 0.045 per cent., which latter is the highest phosphorus percentage in first-class soft Swedish open-hearth steel.

*Tool Steel.*—For the manufacture of hard, first-class tool-steel, pig-iron made from Swedish ores as free as possible from phosphorus is used, with charcoal also nearly free from phosphorus. In hard steel the amount of phosphorus is kept under 0.03 per cent., and no steel is called first-class that contains 0.035 per cent. or more. The best sorts contain 0.022 per cent. and less. The pig-iron and steel are not allowed to contain more than 0.02 per cent. of sulphur; and, with the careful roasting of the ores always performed at the blast furnaces and the use of fluxes free from sulphur, it very seldom happens that the percentage of this impurity in the pig-iron is higher than that figure. If the steel is coming from the furnace too hot, so that piping is to be feared, it is either kept long enough in the ladle to sink to the proper temperature, or the De Laval heat-rings—iron rings lined with fire-proof material heated to a very high temperature—are used. These rings are placed on top of the moulds, and, in casting, are filled with steel, which remains melted in them, and sinks until the ingot below the ring is entirely solidified.

*Steel Castings.*—To make steel castings of perfect quality, a temperature has to be maintained in the furnace during the melting so high that the metal never becomes free from silicon, whereby the absorption of gas is resisted. For this reason the furnaces are much more severely strained in this manufacture than in making steel to be rolled, and do not last half so long as in the latter case.

The main point in the production of steel castings is that the moulds shall be made of proper materials. In Sweden a coarse-grained red sandstone and the purest quartzite (preferably amorphous), containing 99 per cent. or more of silica, are used for this purpose, both being ground to a suitable degree of fineness. The material is bound together with molasses, a solution of carpenter's glue, beer, wheat, flour, or Dutch clay. The facing material is either the finest quartz dust, or, more generally and preferably, a pure siliceous earth (*Kieselguhr*) obtained from Germany and stirred into a solution of carpenter's glue.

At a number of basic furnaces, magnesite imported from Germany is used as a lining and is considered the best material; but in many places dolomite is preferred because of its abundance in Sweden and its consequent cheapness. The operation of the basic process is the same as in America, except that, owing to the low percentage of phosphorus in the material used, no tapping of phosphoric slag towards

the end of the process is necessary. But, since as much pig iron and as little scrap as possible is used, about as much lime is added to counteract the silicon of the pig-iron. The greatest difficulty to be overcome in the basic method as practised in Sweden, has been to make solid hard steel and steel castings without adding aluminium; but this has been met by the addition of ferro-silicon and ferro-manganese, previously melted in a crucible, to the steel in the ladle.

A peculiarity in both the acid and the basic open-hearth process of Sweden is that it is preferred not to recatharise.

According to the reports of the Royal Department of Commerce, the production of open-hearth steel in Sweden was, in 1886, at 3 works, 22,460 tons, and in 1892, at 22 works, 76,556 tons.

*Contribution to the Study of the Structure of Steel.* F. Osmond. Comptes rend. **119**, 1894, 329-331.

By employing the method of polishing sections of steel in bas-relief described by the author in a previous communication (séance of the Académie of 9th April), he has obtained further insight into the structure of steel.

Working with a steel of medium hardness containing C = 0.45 per cent.; Si = 0.07 per cent.; S = 0.016 per cent.; P = 0.036 per cent.; Mn = 0.35 per cent., he heated it to a constant temperature of 825° C., a temperature sufficiently high to ensure all the transformations of the carbon and iron during heating, and then quenched it at decreasing points of temperature in water at 15° C., and examined its structure microscopically. He describes the microscopical appearance when quenched at 720° (before the beginning of the transformations of the iron); at 690° (during the transformations of the iron); at 670° (after the maximum disengagement of heat which accompanies the transformations of the iron); at 650° (during the transformation of the carbon); at 640° (after the transformation of the carbon). Analogous results were also obtained by varying the rapidity of cooling, starting from the steel at a temperature of 720° and quenching it in water at 15°, in boiling water, and in melted lead.

In the case of a hard steel containing C = 1.24 per cent.; Si = 0.35 per cent.; S = 0.012 per cent.; P = 0.017 per cent.; Mn, strong traces; all the points of transformation unite in one change at a temperature of 675° whilst cooling, and at 705° whilst heating, and the diffusion of the tempering carbon takes place more uniformly than in steels of medium hardness, and within a more restricted range of temperature. For the same reasons the structure of the tempered metal is more homogeneous and the variations on tempering at different temperatures are less sharp. We pass more suddenly from the point of no temper to the point of maximum temper. By tempering the steel heated to 735° in water at 15°, fissures were even produced in a disc of 12 mm. At this temperature also, the heating having been effected at about 1° per second, the decomposition of the carbide of iron was not yet complete; it would seem as if the dissolved carbon hindered the dissociation of the carbide by what might be called a certain osmotic pressure.

*Manganese steel containing 12 per cent. of manganese.*—As is known, without decreasing in hardness, this steel becomes much more malleable after tempering at a bright red or white heat. By polishing in bas-relief it was seen that the particles of the alloy were surrounded, after cooling slowly, by a hard envelope, and after cooling suddenly, by a soft envelope or by none. Hence the above-mentioned difference in mechanical properties: the tempering in this case acts by preventing the liquation of a hard and brittle carbide.

The author admits that all the facts he enumerates have not yet been explained, and points out that the impossibility of isolating all the different constituents of steel prevents us from defining them sufficiently by analysis, but he nevertheless concludes that the study of the structure of steel, especially of the medium hard steels, which are the most important, often furnishes very precise information upon—

1. The maximum temperature of heating.

2. The temperature at which the steel has been tempered.

3. The speed at which it has been cooled.

The observer notes that it is always useful to record the observations by photography, and that an enlargement of 800 times is sufficient, necessary and is generally sufficient.

—H. S. P.

*Temperature of Fusion of Tin.* P. Bogodanoff, Journal Soc. Phys. Chim. Russe de St. Petersbourg, 1893, No. 1.

The tin which has been used in these recherches was purified by a double transformation into metastannic acid, and regenerated by reducing with charcoal. In a series of experiments the author plunged the thermometer into the tin, and marked the mark which corresponded to the point of fusion. The surfaces of the tin and of the alloy which was employed as a bath, were maintained at the same level.

The mean of a number of experiments gave the melting point as  $231.14^{\circ}\text{C}$ . In another series of experiments the monometal method has given a mean result of  $231.17^{\circ}\text{C}$ . With English tin, as ordinarily purified, containing traces of antimony, arsenic, and phosphorus, by the method of immediate immersion, the point of fusion was found to be  $230.93^{\circ}\text{C}$ , and the point of solidification  $230.97^{\circ}\text{C}$ .—A. W.

*Methods of Estimating Carbon in Iron.* Götting, Verhandl. Ver. zur Beförd. des Gewerbell., October 1893.

See under XXIII., page 1096.

*Quantitative Estimation of the Impurities most commonly found in Commercially Pure or Rolled Nickel.* T. Fleitmann. Zeits. anal. Chem. 33, 1894, 335.

See under X., page 1096.

*The Part played by the Transformations of Iron and of Carbon in Tempering.* G. Charpy. Comptes rend. 1894, 113, 1258—1260.

From his experiments already described (see this Journal, 1894, 40 and 737), the author has concluded that an allotropic change is produced in iron by hardening, and that it is indicated by the disappearance of the characteristic halt in the stress-strain curve. It has long been known that the intensity of the Eggertz carbon coloration is also reduced by hardening; and in this paper the author seeks to define the conditions under which the two changes occur. Bars of good open-hearth steel, containing 0.71 per cent. of carbon, were cut from the same ingot for the purposes of the test: they were heated, for periods ranging from five minutes to an hour, to different temperatures between  $700^{\circ}$  and  $1,050^{\circ}\text{C}$ , and were then quenched in boiling water or in cold oil, and in one case in cold water. No change in the carbon colour is indicated in samples quenched at temperatures below  $740^{\circ}$ , although in some of the samples there are signs of iron alteration. Above  $740^{\circ}\text{C}$  the iron is completely changed, while the carbon change proceeds more gradually, and is accompanied by an increase in tenacity and decrease in extensibility. It is noteworthy that the allotropic iron change exerts but slight influence upon the tenacity of the metal, whilst the carbon transformation appears to be correlative with the increase in hardness.

The following table gives the details of the experiments upon 24 bars:—

#### IRON AND CARBON TRANSFORMATION IN TEMPERING.

Table of Results of Experiments.

No. of Expts.	Treatment of Metal.			Percentage of Carbon by Eggertz Process.	Tensile Strength.		Extension Per Cent. after Rupture.	Length of Halt in the Curve.
	Heated to.	For Minutes.	Quenched in.		Kilos. per Sq. mm.	Tons per Sq. in.		
2	700	5	Cold oil	0.71	74.3	46.5	20.9	1.0—1.7
2	"	5	Boiling water	0.71	76.7	48.7	18.1	0.7—2.0
3	"	30	Cold oil	0.71	72.2	45.8	19.3	1.9—2.0
1	"	30	Boiling water	0.71	76.0	48.2	17.5	2.0
1	"	60	Cold oil	0.71	70.6	44.8	17.8	2.2
1	720	5	"	0.71	70.0	48.2	17.7	1.7
1	"	5	Boiling water	0.71	70.2	44.6	17.0	2.2
1	"	10	Cold oil	0.58	90.3	57.3	11.5	0.5
1	740	5	"	0.56	86.4	54.9	8.3	0
1	750	5	"	0.55	108.1	68.6	8.9	0
1	"	20	"	0.55	108.1	68.6	8.0	0
1	"	5	Boiling water	0.67	81.3	51.6	13.0	1.2
1	"	30	"	0.67	81.3	51.6	13.3	1.3
1	780	5	Cold oil	0.45	114.3	72.6	9.8	0
1	800	5	"	0.46	98.4	62.5	9.7	0
1	"	5	Boiling water	0.55	96.7	61.4	8.5	0
1	825	5	Cold oil	0.45	117.2	74.4	7.6	0
1	929	5	"	0.45	108.9	69.1	7.5	0
1	1059	5	"	0.45	118.0	74.9	2.8	0
1	850	5	Cold water	0.49	136.1	80.1	0.4	0

\* The records of water quenched steel are printed in italics.

\* An abnormal result.

† A flawed bar.  
—W. G. M.

## PATENTS.

*Improvements in Metallurgical Furnaces for the Reduction and Refining of Ores and Metals.* J. B. Torres, London. Eng. Pat. 13,696, July 11, 1893.

A COAL or crucible of refractory material is fixed within a heating chamber, and this is heated by means of gas from a two-step grate gas-producer. The grate-bars are hollow, and water is made to flow through them, so producing steam, which, passing through the burning fuel, yields water-gas by dissociation.—J. H. C.

*Improvements in the Treatment of Zinc Produced by Electrolysis.* W. Wright, Bettws-y-Coed, and J. B. Hamoud, Bromley. Eng. Pat. 17,166, September 16, 1893.

THE spongy metal at first produced is pressed into thin sheets between rolls, which is found to be better than pressing it into blocks.—J. H. C.

*Improvements in the Extraction of Gold and Silver from Ores or Matte.* B. Mierisch, Niagara. Eng. Pat. 22,397, November 22, 1893.

THIS is a modification of the chlorination process, in which hypochlorites and chlorates of alkalis and alkaline earths are employed to render innocuous the sulphur, arsenic, and low oxides existing in roasted pyrites or arsenious ores. The ores are mixed with sodium hydrate and then treated with chlorine, the admixture of sodium sulphate with the concentrated salt solution serving for lixiviation being prevented, if necessary, by replacing a portion of the sodium hydrate with a corresponding quantity of calcium hydrate. The precious metals, and also lead and zinc, are separated from the iron and copper contained in the solution by saturating it with sodium hydrate. The solution so obtained is passed through granulated zinc to separate the gold, silver, platinum, mercury, and lead, and the zinc solution is afterwards electrolysed in order to recover the zinc in a metallic condition, chlorine and soda solution being re-produced and again used in the process.—J. H. C.

*Improved Method of and Apparatus for Hardening or Tempering Steel Plates and other Articles.* H. W. C. Fairfax, London, and W. Beardmore, Glasgow. Eng. Pat. 4199, February 27, 1894.

THE tempering medium is a liquid which has been artificially cooled below the temperature of melting ice. The chilling process is in some cases applied to the metals while in a molten or semi-molten state, in a mould, and under pressure. Devices are described by which special parts may be cooled, such as the apices of conical projectiles.—J. H. C.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

## PATENTS.

*Improvements in Carbons for Electrodes and other Purposes.* H. Y. Castner, London. Eng. Pat. 19,809, October 20, 1893.

THE present invention relates to the preparation of electrodes for use in electrolytic processes, such as that set out in the inventor's prior Eng. Pat. 16,046, 1892 (this Journal, 1893, 768), to prevent their rapid disintegration under the combined action of oxygen and chlorine. This he proposes to effect by heating gas-retort carbon to a high temperature by an electric current, while the carbon is surrounded by charcoal powder or otherwise protected

from any oxidising action. A current of 350 to 500 amperes passed through a carbon rod one inch square raises it in a few minutes to dazzling whiteness. Ordinary gas-retort carbon under similar treatment gives off an inflammable gas, decreases in weight by from 3 to 7 per cent., swells slightly, and increases in electric conductivity.—J. C. R.

*Improvements relating to the Deposition of Metals and Compounds.* S. O. Cowper-Coles and Captain Sir B. W. Walker, London. Eng. Pat. 21,760, November 14, 1893.

THE authors claim the addition to the liquid, in electrically depositing metals or compounds, of the finely-divided metal or compound, which is kept in suspension during the passage of the current by mechanical or other means.—J. T. D.

*Improvements relating to the Coating of Plates and other Metal Articles to Protect the same from Corrosion, and Apparatus therefor.* S. O. Cowper-Coles and Captain Sir B. W. Walker, London. Eng. Pat. 22,203, November 20, 1893.

THE authors claim the electro-deposition, on iron and other articles, of a thin coating of zinc, enough to protect them from oxidation till they can be painted, then the application of a coat of oil or oily compound over the zinc, by dipping in an appropriate bath, and finally the coating of the whole with paint. They also claim the mode of arranging the depositing tanks and connections for altering the disposition of the current, the mode of supporting the anodes and their connections with the source of current, and an apparatus for rotating the anodes when necessary to face articles of irregular shape.—J. T. D.

*Improvements in or connected with the Electro-Deposition of Zinc or Zinc Compounds, and the Treatment of the Deposited Metal or Compounds.* S. O. Cowper-Coles and Captain Sir B. W. Walker, London. Eng. Pat. 7145, April 10, 1894.

HERE is claimed the use of an alkaline electrolyte in which zinc or a zinc compound, finely divided, is suspended; also the alternation of brief currents of high density with longer currents of lower density to brighten the deposited metal. The use, too, of a sort of sand blast to brighten the metal after deposition is included, and the plan of coating iron with a film of magnetic oxide on which to deposit the zinc.—J. T. D.

*Improvements in Portable Primary Batteries.* W. Morison, Montclair, New Jersey, U.S.A. Eng. Pat. 9862, May 21, 1894.

THESE improvements relate to a portable battery produced by moulding some insulating material to form a liquid and gas-tight box or case to contain the electrolyte, at the same time embedding connections to join up the electrodes. The case shown is divided into three vertical compartments, each containing a pair of electrodes, zinc and carbon, communicating with each other by an open chamber at the bottom. A safety valve is provided. The electrodes not extending to more than half the depth of the box, and the electrolyte not half-filling the box, the battery is brought into or thrown out of action by being inverted or tilted, the mechanism for which, suitable for application to bicycle and miners' lamps, is provided.—J. C. R.

*Improvements in Process and Apparatus for Electrolytically Treating Salts in Solution or in Molten State.* H. C. F. Störmer, Christiania. Eng. Pat. 13,377, July 10, 1894.

A DOUBLE perforated partition is used in the electrolytic cell, into which is led from above a slow stream of conducting liquid, usually the electrolyte itself. In this way

the wandering of the ions and consequent recombination is prevented. Any excess of liquid, should the rate of entry exceed the rate of decomposition, is led off by overflow pipes and collected again.—J. T. D.

*Gravity Battery.* C. E. Baell, North Plainfield, New Jersey, U.S.A. Eng. Pat. 14,634, July 31, 1894.

The inventor claims "a gravity battery cell," having "the combination of an iron upper electrode, a copper lower electrode, and an electrolyte consisting of solid or crystalline copper sulphate with water."—J. C. R.

*Electrical Battery.* A. I. Feeny, London. From F. Dubers and P. Mohrdieck, San Francisco, U.S.A. Eng. Pat. 14,612, July 31, 1894.

This is a battery having sea water for the electrolyte. For the negative electrode, blocks of carbon are employed surrounded by "manganese or other suitable or desired material" in coarse particles, which are contained in a cylinder having perforated sides. This cylinder, or cylinders, is caused by any suitable means to rotate on a shaft journaled at a tank through which the sea-water circulates, and "being rotated at a very slow rate, a portion of it will always be submerged in the liquid and another portion will be constantly exposed to the air, where it, and the contents will be partially dried and will absorb a certain amount of oxygen from the atmosphere, which remains therein when it is again plunged beneath the surface of the exciting liquid."—J. C. R.

*Improvements in or connected with Diaphragms for Electrolytic Purposes.* M. Kiliani, W. Rathenau, and C. Suter, Neuhausen, Switzerland, and the Corporation. "Elektrochemische Werke," Berlin, Germany. Eng. Pat. 15,276, August 10, 1894.

THESE improvements relate to the construction of "an acid proof and alkali proof diaphragm" in the form of a vessel built up of a permeable membrane ("parliament, glass-wool, asbestos, card, or like") held between frames of lattice-work. This lattice is composed of parallel laths, bars, or rods of acid proof material, such as glass, porcelain, or like, secured to end frames which are indented or notched to receive them. The frames thus determine the shape of the diaphragm or receptacle, as rectangular frames for rectangular vessels, oval, or round frames for corresponding shaped diaphragms. The permeable or porous material is tightly stretched around this edge, and surrounded by another outer casing of similar lattice work, retained in position by india-rubber rings, or by wire wound round them.—J. C. R.

*Improvements in Apparatus for Refining Metal by Electrolysis.* H. A. House, sen. and jun., East Cowes, and R. R. Symon, London. Eng. Pat. 16,002, August 22, 1894.

A TANK containing the solution is fitted above the level of the liquid, with a horizontal rotating shaft, on which are wheels of insulating material provided with metal poles near the rim, connected with a segmental commutator near the axle. These wheels are alternately anodes (the crude metal being attached to the pole) and cathodes. Contact pieces make and break with each segment as it comes into and out of the solution. Above the solution are arranged brushes to clean the anodes, and blades or scrapers to remove the deposited metal from the cathodes, at each revolution. The new metal falls into appropriate chutes or receptacles, while any which falls into the trough, or any material falling from the anodes, is caught in a series of canvas bags arranged under the wheels.—J. T. D.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*The Bleaching and Purifying of Refuse Greases from Slaughter-houses.* L. Virolleaud. Bull. Soc. Chim. 11—12, 1894, 526—528.

THE animal refuse coming from slaughter-houses is placed in tanks of 100 galls. capacity. 50 galls. of water and about 2 per cent. of sulphuric acid (53° B.) are added, and the whole is then boiled by means of a perforated steam coil, laid in the tank. As soon as the mass begins to boil, the admission of steam is so regulated that the temperature remains at between 80° and 90° C. After two hours' treatment the steam is shut off, the tank covered and left standing until the following morning. The grease which has collected on the surface of the water is removed. The insoluble residue which has settled to the bottom of the tank, when dried, forms good nitrogenous material for fertilisers. The grease thus obtained, is freed from the disagreeable odour and the objectionable green colour which are peculiar to it after treatment with potassium permanganate. This operation is carried out by melting the grease with direct steam, then adding for each 100 kilos. of grease under treatment a previously-prepared mixture of 1 kilo. potassium permanganate with 300 grms. of sulphuric acid, and energetically agitating the whole mass. After this treatment, the whole is again left over night, and on the following morning the now odourless and colourless grease is separated from the acid solution upon which it is floating.—C. O. W.

*The Examination of Wool-fat.* W. Herbig. Dingl. Polyt. J. 292, 1894, 42—47 and 66—69.

PREVIOUS investigators of the chemistry of wool-fat (with the exception of some of the earliest workers) have taken as a starting point the material obtained by adding the fatty acids precipitated by acidifying the potash soaps, which can be extracted from raw wool by simple washing with water, to the true wool-fat consisting of cholesterol and the esters derived from it. The author has adopted the preferable plan of extracting raw wool with ether, and thus obtaining the wool-fat free from any educt, such as a fatty acid arising from the decomposition of the potash soap naturally present in the fleece. Complete separation of the wool-fat and the potash soap is not practicable in one operation, as the former, when extracted from the wool by dry ether, carries with it a certain quantity of the latter, and the latter, if removed by washing with water, emulsifies a portion of the former. The present investigation was confined to the examination of the behaviour of wool-fat on saponification, particularly in respect to the action upon the fat of sodium ethylate,—and to the separation of the fat into constituents differentiated by the greater or less ease of the saponification. Four samples of wool were used, their places of origin being New Zealand, Australia, South America, and Russia. Large differences in quality were observed.

The samples of wool-fat examined by the author were obtained by extracting portions of 200 grms. of very finely shredded wool for about 16 hours with dry ether, filtering the resulting solution, distilling off the ether, and spreading out the fat in large shallow dishes, which were heated on the water-bath until all ether had been driven off, special care being necessary on account of the persistence with which wool-fat retains the last portion of a solvent. Prolonged exposure to 100° C. is inadvisable, although the author dissents from the supposition of Lewkowitsch (Chem. Centr. 1892, 1, 652; this Journal, 1892, 136 and 137), that the formation of anhydrides or lactones is likely to occur when fatty acids are treated in this manner. The character of each sample obtained from the wool extracted in the manner described above, is appended.

(1.) *Wool-fat from New Zealand Wool.*—The raw wool felt very greasy, contained many clots of fatty matter, some red-yellow and others black in colour. The fat obtained was fairly soft when fresh, but became harder on keeping.





(2.) That the saponification proceeds farther with  $\frac{n}{2}$  potash in a copper tube under pressure than in a flask with a condenser, and that the point thus reached varies with the character of the fat.

(3.) That the limit of saponification of all wool-fats is reached by one hour's heating, under pressure, with  $2n$  potash. (This Journal, 1892, 137.)

On account of the value claimed for the use of sodium ethylate as a saponifying agent, the author made a series of experiments with this reagent (10 grms. of sodium in 150 cc. of alcohol), adding it in regulated amount (15 cc.) to the wool-fat either suspended in alcohol or dissolved in ether. The results are recorded in tables, and may be verbally summarised as follows:—Saponification of wool-fat with sodium ethylate in alcoholic solution (or suspension) never reaches the limit arrived at by the method of heating under pressure (see this Journal, 1892, 137). In ethereal solution it proceeds to the same point as that arrived at by saponification with  $\frac{n}{2}$  alcoholic potash under pressure, but fails to saponify that portion of the fat which requires the use of  $2n$  alcoholic potash under pressure for its complete attack. The author concludes that the use of sodium ethylate presents no tangible advantage in the treatment of fats difficult of saponification.—B. B.

*Studies in Oil Analysis.* H. Ballantyne. A paper read before the Glasgow City Analysts' Soc. Abstracts No. 4, 1893-94, 6.

See under XXIII., page 1100.

*Evaluation of Wool Fat.* E. v. Cochenhausen. Dingl. Polyt. J. 292, 1894, 91-96; 112-116.

See under XXIII., page 1100.

## PATENTS.

*Improvements in Recovering Grease from Waste Suds which have been used in Washing Wool, Hair, or other Fibre.* A. J. Inston, Kidderminster. Eng. Pat. 17,309, September 14, 1893.

THIS patent is for a method of recovering the grease contained in waste suds from the washing of wool, hair, or other fibre. The ordinary method consists in treating the suds with sulphuric acid, and collecting the "recovered grease" which rises to the surface. The patentee seeks to avoid the use of any acid. He first strains or filters the suds to remove suspended impurities, then adds "an alkali such as caustic soda, caustic potash, or the like," and boils for about two hours. "On the liquor cooling, the grease will be found in a solid or viscous state at the top," and can be separated and dried or pressed in the ordinary way, the liquor beneath being run to waste. This is the first method claimed. The second consists in using, instead of the alkali, oil, tallow, or other oleaginous material, the process otherwise being the same. The third method consists in using a mixture of alkali and oil.—L. A.

*Improvements in Machinery for Cleaning Cotton Seed.* J. Bibby, Liverpool. Eng. Pat. 21,372, November 10, 1893.

THIS relates to a machine for removing the adhering fibre or lint of cotton-seed after the bulk of the cotton has been removed, and for cleaning the seed thoroughly without damaging it. It is composed of an outer cylinder and an inner revolving barrel, the axes of which are horizontally disposed. The inner surface of the cylinder and the outer surface of the barrel are lined with emery, which holds the seeds against the rotary action of the barrel. By this means the cotton is removed, and the surface of the seed polished. In some cases wire netting (through the meshes of which the emery in a moist or plastic condition is squeezed) is

attached to the surfaces of the cylinder and barrel. The barrel surface is preferably ribbed or fluted. At the feeding end of the machine a number of saws are placed, which facilitate the introduction of the seeds into the space between the barrel and the cylinder. The same arrangement of saws, together with a brush to remove from the saws the fibre adhering to them, is placed at the exit end of the machine.—J. J. K.

*Improvements in and Connected with Oil and other like Presses.* J. Y. Johnson, London. From C. Gounelle, Morilles. Eng. Pat. 23,595, December 7, 1893.

See under I., page 1048.

*Improvements in the Manufacture of Cleansing, or Soap Powder for Washing Fibrous and other Materials.* Andrew Ross Scott, Glasgow. Eng. Pat. 1963, January 20, 1894.

COCA-NUT or palm-nut oil is saponified with caustic soda in slight excess, the excess being afterwards allowed to remain, or neutralised with any suitable acid or acid salt, preferably boric acid. Water and soda-ash are then added in such proportions that the mixture will contain from 20 to 60 per cent. of crystallised sodium carbonate. The mixture is cooled, pulverised in a disintegrator, and then intimately mixed with ammonium sulphate in proportion equivalent to the sodium carbonate. The ammonium carbonate formed by double decomposition imparts the desired properties to the compound.—L. A.

*Process and Apparatus for Obtaining Glycerin and other Products from Soap-Makers' Spent Lyes.* J. V. Ruyembeke and W. F. Jobbins, New York, U.S.A. Eng. Pat. 12,367, June 26, 1894.

THERE are 49 claims, the gist of which consists, *firstly*, in an improved process for recovering products from the spent lyes by removing the heavy impurities as a sediment, allowing the lye to cool, whereby suspended soap is brought to the surface, and dissolved soap is separated and suspended, and, as such, is brought to the surface and the separated soap skimmed off, the operation being carried on previous to the addition of any chemicals to the lye.

*Secondly*, in an improved dash-plate for use in vacuum evaporators or similar chambers in which liquids or fusible solids are heated, comprising the annular dash-plate within the chamber, and an imperforate dash-plate supported above or below the annular one, and in line with the opening therein.—J. J. K.

*A New or Improved Apparatus for Treating Substances containing Fats and Oils.* W. O. Robbins, New York. Eng. Pat. 14,821, August 2, 1894.

THIS is a complete self-contained apparatus for extracting oil from seeds, wool, and other substances by means of a solvent, and for recovering the solvent for use again. It consists of a cylinder for receiving the material to be treated, a pipe for conveying gasoline from a tank and spraying it through the material within the cylinder, steam-pipes for heating the gasoline after admission to the cylinder, an air-compressor for expelling the solution of the oil in gasoline from the cylinder into a still, where the oil and solvent are separated, a vessel connected to the still to receive the extracted oil, a worm for condensing the gasoline vapour, and a pump for forcing the condensed gasoline back into the vessel from which it started. The whole series of vessels, pumps, and condensing worm are connected by pipes and valves, so that after the material has been placed inside the cylinder, and the latter closed, the complete extraction of the oil, the separation of the oil and solvent, and the drying of the material, can be effected without opening any part of the apparatus. The material to be treated is contained in a cylindrical ear of wire netting, fixed on wheels, which runs on rails within the cylinder. The single claim is for the complete apparatus as described and illustrated in the specification.—L. A.

*Solutions of Sweet Carbamides in Oils, Fats, Waxes, Resins, and Process of Making the same.* A. Sommer, Cambridge, U.S.A. Eng. Pat. 15,168, August 14, 1894.

THE inventor has found that an agreeable sweet taste can be communicated to the saponifiable oils, fats, waxes, and resins by dissolving in them certain sweet carbamides, such as paraphenetol carbamide ("Dulcine"), which he uses almost exclusively. He finds that the solubility of this substance in the neutral oils, fats, &c. is almost nil in the cold, and greatest between 140 and 160 °C., but that when once solution has been effected in the hot oil, and any excess of the carbamide which may have crystallised out on cooling has been filtered off, enough is still retained in solution to impart a sweet flavour to the cold oil, &c., the proportion so retained varying from less than 0.1 per cent. to over 5.0 per cent. of the oil, &c. taken. He has further found that the free acids of the above products dissolve the sweetening agent much more freely than the neutral products themselves, and by taking advantage of this fact he has devised another method of effecting solution. Thus, if the oil, &c. to be sweetened does not already contain sufficient free acid, he adds some, or produces some in the oil itself by partial saponification, followed by decomposition of the soap with acid and washing with water. He then dissolves the carbamide, either in the free acid before adding it to the oil, or in the naturally acid or artificially acidified oil itself, solution being hastened by heating the mixture in the water-bath. Another method of effecting solution is to dissolve the carbamide in a volatile solvent, which is then mixed with the liquid or liquefied oil and afterwards evaporated off.—L. A.

acid, yielding the acetic acid required for the preparation of the lead acetate. This salt may also be obtained electrolytically, a specially designed apparatus being described and figured in the specification. T. H. L.

*Improved Decorative Paper for Walls, Ceilings, Door Panels, and the like.* J. F. H. Vander Hoop, London. Eng. Pat. 20,353, October 27, 1893.

ANY suitable paper is coated with oil paint on one or both sides, and, when dry, any ornamented figure is printed with oil paint upon its surface. To obtain the impression in relief in gold, silver, or bronze, the metal powder is applied while the paint is still in the sticky condition.—S. P. E.

*Improvements in and relating to a Process of Making Green Oxide of Chromium.* F. L. Stoeum, Pittsburgh, and C. Y. Wheeler, Allegheny, U.S.A. Eng. Pat. 15,473, August 14, 1894.

TEN parts of powdered potassium or sodium bichromate are moistened with strong hydrochloric acid, and one part of finely-divided wood-charcoal added, and the whole made into a paste with three parts more of hydrochloric acid. The mixture is then placed in a suitable furnace, and allowed to burn without admission of air, and the oxide finally washed and dried.—F. H. L.

### (B).—RESINS, VARNISHES.

*The Latex of the Lacquer Tree.* G. Bertrand. Comptes rend. 118, 1215—1218.

By making an incision in the bark of several species of *Rhus* (*Anacardium*) which occur in the south-east of Asia, a latex is obtained which the Chinese and Japanese use as a varnish for their furniture under the name of lacquer. This latex, which is known under the name of "So'n-mat-Dai," is a thick cream of a bright yellow, almost white colour; it has a feeble odour recalling butyric acid, and reddens blue litmus paper. It may be kept for a long time without undergoing alteration, in flasks, if the latter are quite filled and well stoppered; but in contact with air, it rapidly oxidises, becomes brown, and in a few minutes there is formed a black pellicle, insoluble in the ordinary solvents. It is on account of this remarkable property that the latex is employed, and it is only necessary to observe some precautions in applying it, in order to obtain a brilliant and permanent coating of black.

When the latex is treated with alcohol this alteration does not take place, and a precipitate is formed of a body which is chemically identical with the gums, and contains an enzyme designated by the author, laccase; whilst the portion dissolved in the alcohol is an oil insoluble in water named laccol, which oxidises very readily when exposed to the air, becoming red—the more readily, however, in presence of dilute alkaline hydroxides. Towards the last-named compounds it behaves like pyrogallol; thus, the liquid becomes warm with the production of first a green coloration and then a deep black, a large absorption of oxygen taking place simultaneously. Ferric chloride in alcoholic solution is immediately reduced, the mixture becomes green, and a black amorphous metallic derivative separates; lead acetate produces a white precipitate, becoming bluish-green and finally black by slow oxidation. In the natural product, the gum and laccase are dissolved by a little water, the laccol being simply emulsified. The characteristic property of furnishing a lacquer is due to the successive actions of oxygen and laccase on the laccol.

—A. R. L.

*Analysis of Sealing Waxes.* C. Mangold. Zeits. anal. Chem. 1894, 33, 477—478.  
See under XXIII., page 1100.

## XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

### (A).—PIGMENTS, PAINTS.

#### PATENTS.

*The Manufacture or Production of Paints.* W. A. and R. H. C. Wilson and W. B. Priest, London. Eng. Pat. 17,159, September 12, 1893.

By the addition of about one part of amorphous lead hydrate to two parts of such substances as barium or calcium carbonate or sulphate, iron oxide, ochre, umber, sienna, &c., it is claimed that these latter become far more valuable pigments for grinding in oil, the lead hydrate increasing their otherwise small body and covering power.

—F. H. L.

*Improvements in the Manufacture of White Lead.* J. H. Noad and S. Z. de Ferranti, London. Eng. Pat. 20,239, October 26, 1893.

A FAINTLY alkaline solution of ammonium acetate, prepared as described in Eng. Pat. 6009, 1892 (this Journal, 1893, 363), is added to litharge and heated by steam in a closed still. So long as the latter is kept in excess, only the lead monoxide is dissolved, all impurities and higher oxides being unattacked. The ammonia liberated is condensed and carbonated by being passed through coke towers through which a stream of carbonic anhydride obtained from lime kilns or washed furnace gases is ascending. The filtered lead acetate solution, still at the boiling point, is run into another vessel provided with an efficient agitator, and the ammonium carbonate sprayed in; the white lead is filter-pressed, and the filtrate is ready to dissolve a fresh amount of litharge. To economise the amount of heat required for this process, the still may be worked under diminished pressure.

As an alternative process, a solution of basic lead acetate is prepared in the usual manner, and mixed with the calculated proportion of ammonium carbonate, prepared as above described, the white lead filter-pressed, and the ammonium acetate distilled with chalk or lime to form fresh carbonate or hydrate (to be afterwards carbonated), the calcium acetate being finally decomposed with sulphuric

## PATENTS.

1 *Process of Manufacture of Films Imitating Mother-of-Pearl.* S. Hahn, Berlin. Eng. Pat. 13,139, July 6, 1894.

WHEN nitrocellulose, dissolved in alcohol and ether, or in soda- or potash-soluble glass, is spread over a surface of wood, paper, glass, porcelain, or metal, and the solvent allowed to evaporate, the film remaining is said to have the appearance of mother-of-pearl. The proportions recommended are:—

1 part of nitrocellulose.

78 parts of alcohol (90 to 100 per cent.).

21 parts of ether.

With soluble glass as solvent 10 parts of this to 90 parts of water are employed.

The nitrocellulose may be pure or crude, or in different stages of nitric acid, as gun-cotton, &c. Ethyl or methyl alcohol and sulphuric or acetic ether are recommended. The degree of concentration of the nitrocellulose may be varied within certain limits, which variations produce different results. The addition of bisulphide of carbon in the proportion of 25 parts to 100 of the above solution, or the addition of benzene, produces a difference in the brilliancy and arrangement of the colours of the iris developed on the mother-of-pearl-like surface.—S. P. E.

*Method of Varnishing Metal in Light-resisting Colours by Means of Alizarine Colours.* J. Perl and P. Herrmann, Berlin. Eng. Pat. 15,327, August 11, 1894.

THE objects are dipped in a colourless pyroxylin varnish and then heated in a current of air at 80° C. until the varnish is thoroughly dry, when they are immersed for a few seconds in a two per mille alcoholic solution of alizarin, followed by a wash in water, to change the colour from the original light yellow to a golden red.—F. H. L.

## (C.)—INDIA-RUBBER, &amp;c.

## PATENTS.

*Improvements in Artificial India-Rubber.* P. A. Newton, London. From A. Nobel, Paris. Eng. Pat. 20,234, October 26, 1893.

By dissolving nitro-cellulose or nitrated hydro- or oxy-cellulose in a suitable non-volatile or slightly volatile "solvent," such as nitronaphthalene, dinitrobenzene, nitro-toluene or its homologues, products may be obtained varying from a gelatinous consistency to the hardness of ebonite. The proportions will vary from about 20 per cent. of nitro-cellulose in the finished product, forming a soft rubber, to 50 per cent., imitating celluloid, and the "solvent" chosen will depend on the use to which the rubber substitute is to be put, the liquids giving a more elastic substance, while mixtures of solids and liquids may be employed when the product is to be used at high temperatures. By means of rollers—steam-heated, if desired—the incorporation may be accomplished without the aid of a volatile liquid, or the nitrocellulose may be employed wet, the water being re-

moved after "solution." It is advisable to use the cellulose nitrated only just enough to render it soluble, in order to reduce the inflammability of the finished product.—F. H. L.

*An Improved Preparation for Cleansing Vulcanite.* L. and S. Levy, London. Eng. Pat. 3,180, February 14, 1894.

THE improved preparation consists of a "chemical compound" of washed and levigated calcium carbonate, coleothar, rectified spirit, methyl alcohol, and water, the addition of a little ammonia solution being an improvement. It is rubbed on with a rag, the vulcanite being afterwards washed with water and polished with chamois leather.

—L. A.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

*The Water-Content of the Common Tanning Materials.* Von Schroeder. Dingl. Polyt. J. 1894, 292, 284—287.

FOR the scientific comparison of tanning materials a knowledge of the percentage of tannin in the anhydrous material is all that can be desired, but for the purposes of the tanner, who deals with the moist bark, it is obviously of little value. The question thus arises—What is the water-content of the sample submitted for analysis? Is the moisture in the sample when it reaches the analyst identical with that contained in the sample when it was drawn from the store? Disputes between buyer and seller may possibly be due to a variation in the water-content of the material.

The author has examined 11 different tanning materials, some ground and some unground, whilst some were partially ground or pulverised. The object of the examination was to ascertain the degrees of variation which occur in their water-content at different seasons. After the samples had been kept in a room—which was chosen so that the conditions might resemble those of the tanner's store as nearly as possible—for three weeks, in order that they might attain an equilibrium of moisture proper to the situation, the percentage of moisture in each was determined, and thereafter the samples were merely weighed from time to time, the variation in weight being calculated into variation of content of water. The samples varied in weight from 1,400 to 4,500 grms., and were contained in linen bags of known tare; the hygrometric variation of these was sufficiently slight to be neglected. In a second series of experiments about 100 grms. of the sample were contained in metal cylinders open at the top except during the weighing process. The percentage of water calculated from the monthly weighings are tabulated in the original paper, and a very close agreement is observable between the results obtained in the two sets of experiments. The state of division of the material has hardly any effect on the water content; the variation which occurs in the different materials will be appreciated from the following table, comprising the mean values for all the experiments:—

Material.	Jan.	Feb.	Mar.	April.	May.	June.	July.	Aug.	Sept.	Oct.	Nov.	Dec.	Mean.
Sumach .....	11.89	12.56	13.27	10.77	10.11	9.72	11.12	11.42	12.27	13.46	14.16	14.80	12.13
Azorebilla .....	12.41	12.78	13.12	12.04	11.60	11.28	11.97	12.23	12.79	13.41	14.00	14.27	12.66
Oak bark .....	13.23	14.14	14.07	11.42	11.13	10.48	11.78	12.04	12.71	13.74	14.52	15.06	12.92
Myrabolans .....	13.72	11.90	14.41	12.35	11.76	11.91	12.13	12.28	12.98	13.77	14.42	14.88	13.14
Divi-divi .....	13.26	13.99	14.16	12.31	11.81	11.38	12.56	12.80	13.55	14.50	15.06	15.53	13.43
Pine bark .....	16.31	16.34	16.13	12.89	12.41	11.48	12.87	13.13	14.09	15.08	16.01	16.64	14.48
Valonia .....	15.74	15.82	16.00	13.47	13.10	12.42	13.52	13.70	14.43	15.12	15.75	16.25	14.61
Mimosa bark .....	14.23	14.75	15.06	13.57	13.16	12.79	13.79	13.90	14.54	15.13	15.85	16.16	14.41
Quebracho wood .....	16.97	16.52	16.05	13.89	13.11	12.26	13.14	13.28	13.64	14.16	14.58	14.88	14.43
Rowe .....	15.66	15.69	15.65	14.59	14.19	13.65	14.57	14.72	15.24	15.54	15.94	16.38	15.16
Knoppers .....	18.11	18.20	18.33	15.30	14.66	13.85	14.96	15.12	15.68	16.39	16.94	17.47	16.26

It is right to add that in 1885, the year of this investigation, the weather suddenly became very warm in April, that the temperature rose during May, and became very high in June.

It will be found by examining this table that the variation in content of water of the common tanning materials amounts to from 3 to 5 per cent.; if the results of analyses are calculated on the mean value for the whole year, there is an error owing to this amount of moisture being about 2 per cent. too low in winter and about 2 per cent. too high in summer. In the author's opinion, however, it is best to adopt the mean value in returning the percentage of tannin in the sample, although it is quite reasonable to make the correction indicated for the time of year when the sample is received.—A. G. B.

*The Amount of Water contained in Air-dried Bark-tanned Leather.* Von Schroeder. *Dingl. Polyt. J.* 1891. **293**, 139—143, 164—167, 187—192.

In the first part of this paper the authors describe the character and tannage of the samples used in the investigation, and the method of exposing them to the air so that they might take up or lose moisture under conditions such as prevail in the trade. Full analyses of samples are given, these differing chiefly, of course, in the content of fat; 12 out of the 24 samples were dressed leather.

The most salient points elucidated by the investigation are the following:—

In the first series of experiments, in which the samples were exposed in a shed resembling in its conditions a normal leather store, it was found that the water-content of air-dried leather is highest in the three winter months (December—February), and generally attains a maximum in February, whilst the minimum occurs in June. The following lines of figures, selected from the author's table, will illustrate the monthly variations for undressed and dressed leather:—

	Sole Leather (lined).	Kip Upper Leather (black).
	Per Cent.	Per Cent.
January .....	20.15	15.00
February .....	19.69	14.96
March .....	17.34	12.60
April .....	16.08	11.41
May .....	16.04	10.90
June .....	15.59	10.76
July .....	16.80	11.61
August .....	16.78	12.03
September .....	17.50	12.65
October .....	17.74	12.86
November .....	20.30	13.57
December .....	19.55	14.28
Mean .....	17.80	12.67

It is generally the case that dressed leather contains less water than undressed leather. The mean water-content of undressed leather for the whole year may be taken as 18 per cent. Untanned hide is much more hygroscopic, and the water in it may vary from 28.20 to 19.14 per cent., the mean for the whole year being 23.13 per cent.

As might be expected, the higher the content of fat in a dressed leather, the lower will be the percentage of water. It is possible to calculate the water ( $w$ ) in a dressed leather when the percentage of fat ( $F$ ) is known and the percentage

of water ( $a$ ) in the corresponding fat free leather is known, by means of the following formula:—

$$w = \frac{100 \times (100 - F)}{100 - 100 \times a + a(100 - F)}$$

The mean value for  $w$  for all classes of dressed leather is 10.35. The value  $a$  may be taken as 18.

Bark-tanned leather which is exposed at a low temperature to an atmosphere saturated with water, will take up about 24 per cent. of water.

The deviation of the percentage of water with the time of year from the adopted mean of 18 per cent. for an undressed leather, may be said to be  $\pm 2.50$ . A leather which suffers loss of water through any period from August to February must be reported as improperly dried. A decrease of 2 per cent. of water in the warmer months of the year does not necessarily indicate that the leather has been badly dried; but a loss amounting to 4 per cent. must certainly be reported as such. It must be remembered, however, that these remarks apply only to the normal conditions of the atmosphere, and due attention must be paid to the hygrometer readings in the room in which the exposure of the leather is effected.

In the second series of experiments the samples were exposed in a room where the temperature throughout the year was far more equable than in the shed. This difference of condition made but little difference in the mean water-content of the leather for the whole year, but, as might be expected, the monthly variation was far smaller. This is illustrated in a table giving the figures.—A. G. B.

*The Practical Extraction of Tannin from Myrabolans.* Von Schroeder. *Dingl. Polyt. J.* **292**, 213—214.

THE use of myrabolans, the fruit of *Terminalia Chebula* (Willd.), is yearly extending in Germany. In 1891 there were imported 62,870 centners (1 centner = 50 kilos, or 100 lb.); and in 1893, 101,394 centners. Myrabolans contain on the average 30 per cent. of tannin, and tannin can be bought at present in this form cheaper than in almost any other. The price of myrabolans of good medium quality is 7 marks per centner (7s. per 100 lb.), which corresponds to 0.24 mark per centner for 1 per cent. of tannin, compared with 0.59 mark for oak-bark tannin and 0.26 mark for fir-bark tannin. Somewhat inferior qualities of myrabolans, containing, however, very little less tannin, can be had for 5.5 marks, or a cost of 0.18 mark for 1 per cent. of tannin. The only material approaching this in cheapness is quebracho wood, 4.25 marks per centner, containing 23 per cent. of tannin, or 0.19 mark for 1 per cent.

Myrabolans alone do not give satisfactory results in tanning, imparting an unpleasant, greenish-yellow colour to the leather. They are largely used in mixtures with more expensive materials, e.g. oak-bark, in order to lower the cost, and in some cases can replace knoppreri and valonia. If the colour is not important, myrabolans and quebracho wood form a very cheap mixture, the addition to which of fir-bark considerably improves the colour.

The fruit is more or less pointed at both ends, and on the average 3 cm. long. The more expensive kinds have a light yellowish-brown colour, the lower qualities being darker in appearance and containing nearly as much tannin. The fruit consists of an outer fleshy rind, of loose, parenchymatous tissue, in which most of the tannin is contained, and within this a hard stone enclosing the seed and bearing very little tannin. While the rind is easily reduced to powder, the stone is extremely difficult to grind. The rind on the average forms 65 and the stone 35 per cent. of the whole fruit. The following analyses show the proportion of tannin in different qualities of myrabolans and in different parts of the same fruit. They are calculated on the average percentage of water = 13 per cent.

## I.

Selected dark myrabolans, inferior in appearance, consisting of 63.5 per cent. rind and 36.5 per cent. stone.

	Rind.	Stone.	Whole Fruit.
	Per Cent.	Per Cent.	Per Cent.
Water .....	13.00	13.00	13.00
Tannin substances .....	35.46	3.52	23.80
Organic substances (not tannin) .....	23.65	3.22	16.19
Ash of extract .....	2.74	0.71	2.00
Insoluble in water .....	25.15	79.55	45.01
	100.00	100.00	100.00
Total extract .....	61.85	7.45	41.20
Sugar (calculated as grape sugar) .....	4.74	0.39	3.15

## II.

Selected light-coloured myrabolans, of good appearance, with 67.58 per cent. rind, and 32.42 per cent. stone.

	Rind.	Stone.	Whole Fruit.
	Per Cent.	Per Cent.	Per Cent.
Water .....	13.00	13.00	13.00
Tannin substances .....	42.34	2.60	29.46
Organic substances (not tannin) .....	20.98	2.58	15.02
Ash of extract .....	3.35	0.58	2.44
Insoluble in water .....	20.33	81.24	40.08
	100.00	100.00	100.00
Total extract .....	66.67	5.76	46.92
Sugar (calculated as grape sugar) .....	7.40	0.50	5.16

Myrabolans are used either in the ground condition or as extract. In the former case, since the stones contain little tannin, and are difficult to grind, the rind and stone are sometimes separated mechanically, and the rind only ground to powder. Such a separation is necessarily very imperfect, while the stones form a by-product difficult to make use of, and, if this product is considered as waste, the expenses of grinding are considerably increased. To illustrate the incompleteness of a mechanical separation, two samples were obtained from a tannery, ground on the spot for their own use, of which one should consist chiefly of ground rind, the other of the residual stones. On analysis the percentages of tannin in the two are found to be very near together. Water is assumed at 13 per cent.

	Tannin Substances.
	Per Cent.
Nominally consisting chiefly of rind .....	35.89
" " " " " stones .....	31.91

It may be concluded, then, that it is better either to grind the whole fruit or to extract it. Extraction of myrabolans is a very rational method, because the greater part of the tannin-containing rind is soluble in water. On boiling with water, the rind takes up water and the swollen fruits give up almost all their tannin if the liquor be changed sufficiently often. The extraction of whole myrabolans

succeeds well in practice. Some time ago, in the Freiberg tanning school, a sample lot of myrabolans was extracted in a closed extraction boiler. The fruits were analysed before extraction and after three- and fourfold boiling, with these results (water reckoned at 13 per cent.) :—

	Before Extraction.	Three Times Extracted.	Four Times Extracted.
	Per Cent.	Per Cent.	Per Cent.
Water .....	13.00	13.00	13.00
Tannin substances .....	32.14	4.65	3.18
Organic substances (not tannin) .....	11.05	3.30	1.48
Ash of extract .....	2.27	0.49	0.29
Insoluble in water .....	41.54	78.65	82.05
	100.00	100.00	100.00

The weight of the myrabolans dried in air after three extractions is 52.8, and after four extractions 50.6 per cent. of their original weight. Hence, of the 32.14 per cent. of tannin in the fruit, after three extractions, 2.46 per cent. remains, and after four, only 1.61 per cent. is still in the residue. Thus, in the last case, of 32.14 parts tannin, 30.53 parts are brought into solution—a highly satisfactory result, and one that points to the extraction of unground myrabolans as a safe and commendable process.—R. B. B.

*The Estimation of Sugar, and on the Sugar-Content of Tanning Materials, Tanning Extracts, Tanning Liquors, and Weighted Leather.* V. Schroeder, A. Bartel, and W. Schmitz-Dumont. Dingl. Polyt. J. 1894, **293**, 229—237, 252—260, 281—287, 297—304.

See under XXIII., page 1101.

## PATENTS.

*Improvements in or connected with Tanning Hides and Skins.* J. Durio, Turin, Italy. Eng. Pat. 16,783, September 6, 1893.

The hides are limed, unhaired, fleshed, and treated to remove the lime, as usual. They are then immersed in a solution of "granular hydrate of soda," containing 3—12 kilos. of hydrate per 100 kilos. of hides and 400 kilos. of water. They are then placed in a centrifugal machine to remove the absorbed hydrate, washed, and pressed. Degreasing by means of a suitable solvent in a centrifugal machine concludes the preparation.

The prepared hides are suspended in a drum into which liquid tannic extract (in the condition in which it is sold in the trade and without dilution) is introduced at intervals in gradually increasing quantity until the tanning process gets slow, whereupon the quantity introduced is gradually decreased. The drum should receive a continuous motion so that the hides may be agitated in the tannic extract; the rapidity of agitation is regulated according to the quality and thickness of the hides, the minimum time of tannage being about two hours and the maximum about 24 hours.

The advantages claimed for the process are that it can be used in any district, and the tanner thus rendered independent of the quality of the water supply, and that the product is better than that obtained by the old methods.

—A. G. B.

*Improvements relating to the Manufacture of Artificial Whalebone and to Apparatus therefor.* M. Levy, A. Rustin, and L. Boyer, Paris. Eng. Pat. 20,144, October 25, 1893.

To unite two pieces of buffalo horn in order to make a long piece of artificial whalebone, the ends of the pieces are immersed for from 5 to 10 minutes in a hot solution of

bicarbonate of soda (10 grms. per litre), the pieces are allowed "to sweat," and then bevelled. The horn is softened by the hot alkaline liquid, so that by wrapping paper round the joint and applying pressure whilst it is hot the union may be made perfect. To prevent fracture at this joint it is again dipped in the solution of bicarbonate of soda.

The above method is the only point of chemical interest in the specification, the remainder of which relates to the machinery necessary for effecting the union and cutting of the horn. Figures of this machinery are given.—A. G. B.

*An Improvement in Softening and Cleansing Hides.*  
W. L. Jackson, London. Eng. Pat. 22,819, November 28, 1893.

Raw hides and skins, before they are unhaired, are softened and cleansed by thoroughly soaking them and then beating them by fulling stocks or equivalent apparatus. The improvement consists in adding a soluble antiseptic during this process; common salt, chloride of magnesium, calcium carbonate, or sodium sulphate is recommended. For a full sized hide 1—1½ lb. of the salt should be added.

—A. G. B.

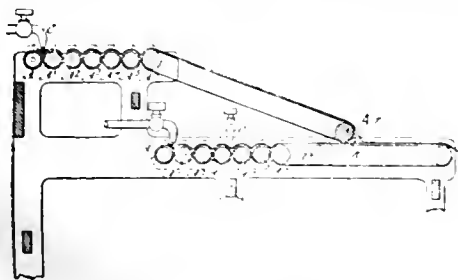
*Improvements in the Manufacture of Animal Charcoal.*  
C. W. Kriens, London. Eng. Pat. 8112, April 24, 1894.

Waste leather or scrap, or the trimmings, or curriers' cuttings of hides or skins, are steeped in water at 100°—110° F. for 48 hours, and then pressed to expel the absorbed liquid. The steeping process is repeated for another 48 hours and the materials are again pressed; they are now tightly packed into an iron cylinder, provided with the usual condensing apparatus for the recovery of by-products, and the whole is introduced into a retort to effect the carbonisation.

—A. G. B.

*Improvements in Processes and Apparatus for Cooling Glue and Forming it into Sheets.* P. C. Hewitt, New York, U.S.A. Eng. Pat. 11,426, June 12, 1894.

The liquid glue is allowed to flow on to a series of cylinders A—A<sup>5</sup> (see figure), cooled internally by a flow of water, and all revolving in the same direction. The solidified sheet of glue is thus caused to travel forward on to an endless band composed of the wire network usually employed for drying glue. When it is desired to make a



sheet of twofold thickness, the endless band is inclined so that the sheet may be deposited upon a second sheet, itself borne on an endless band and formed in the same way; such an arrangement is shown in the figure. A steam-pipe *k* serves to heat the two sheets and to cause their adherence to each other. By flowing liquid glue on to the lower sheet, through the tap *C*, a threefold thickness may be obtained. Various modifications for combining the sheets are depicted. The method serves for the combination of glues of different grades of quality.—A. G. B.

*Improvements in Clarifying or Removing Impurities and other Substances from Glue.* P. C. Hewitt, New York, U.S.A. Eng. Pat. 43,369, July 10, 1894.

CASEIN, separated from skimmed milk by rennet, is dissolved in lime water, and the solution is added to the glue solution. In the case of some glue solutions it is advisable to add albumin (preferably blood albumin) to the casein solution. For some uses it is desirable that the casein should not be alkaline, in which case it may be neutralised, preferably by phosphoric acid.

By the present process of glue manufacture the other constituents of the stock, dissolved by the gelatin during the rendering by heat, are removed by large filters and by the use of clarifying agents, such as albumin and alum; these clarifying agents are expensive, leave the glue foamy, and cause a precipitate which is filtered with difficulty. By this new process about 1 lb. of the dry clarifying agent, mixed with an appropriate quantity of water, is mixed with 1,000 lb. of the hot glue solution (containing 10 per cent. of dry glue); the coagulation is aided by heat, or by addition of acid or of rennet. The precipitate is sufficiently hard to cause but little impediment during filtration. The clarified glue is bleached by sulphurous acid or "hydrosulphurous" acid (zinc and sulphurous acid) in the usual manner.

—A. G. B.

*A New or Improved Process for the Production from Skins or Hides of a Substitute for Whalebone, Horn, or the like.* F. Hartmann, Berlin, Germany. Eng. Pat. 14,697, July 31, 1894.

This new product, to be called "whalebone leather," is made from the unhaired, fleshed, scoured, and cleansed skin or hide, by first submitting it for a short time to the action of wet steam, and then steeping it in a turpentine-bath until it has become perfectly saturated with that material; it is finally dried in the open air, and varnished and glossed to render it weather-proof.—A. G. B.

## XV.—MANURES, Etc.

### PATENTS.

*Improvements in the Manufacture of Mineral Manure.*  
G. F. Redfern, London. From J. Hensel and F. Spiethoff, Soermsdorf, Kynast. Eng. Pat. 17,235, September 13, 1893.

PRIMITIVE rocks, such as porphyry, granite, gneiss, syenite, or basalt, are heated to a cherry-red heat, quickly cooled in water, and then crushed as fine as possible. This artificial stone meal is then intimately mixed with finely-powdered coal, in about the proportions of 10 parts of coal to 100 parts of stone. The manure is then ready.

The rocks supply potash, soda, and phosphoric acid to the crop, whilst the coal co-operates as a result of the presence of "nitrogenous hydrocarbons" in it. To make the manure act more quickly sulphuric acid may be added to it, or it may be mixed with other mineral manures.

—A. G. B.

*An Improved Method of Utilising and Means for Treating Yeast.* E. W. Greening, Thames Ditton, Surrey, and E. O. Greening, Lee, Kent. Eng. Pat. 20,060, October 24, 1893.

See under XVII., page 1082.

## XVI.—SUGAR, STARCH, GUM, Etc.

*The Properties of Caramel.* A. Sabanejeff and A. Antouchévitch. Soc. Ph. Ch. de Saint-Petersbourg, No. 1, 1893.

For authors arrived at the following conclusions:—

1. Caramel, precipitated from a solution of the crude product by alcohol and then dialysed, is not a carbohydrate. The formation of caramel from sugar is accompanied by evolution of carbon dioxide. The formula of that compound is very approximately expressed by  $C_{12}H_{13}O_4$ . The formation of caramel is illustrated by the formula—



2. Dialysed solutions of caramel undergo modification under the influence of temperature and light, their coloration diminishing at the same time. By a process of hydrolysis, substances of a lower molecular weight are formed.

3. Dialysed solutions of caramel are capable of a special (mucilaginous) fermentation. The coloration diminishes thereby, and by a process of dehydration, substances of considerably higher molecular weight are formed.

4. Gélis' caramel is merely dialysed caramel modified by the elevated temperature of the solutions.

5. Graham's caramel, the dialysability of which is only one quarter that of albumin, is a product of the mucilaginous fermentation of dialysed caramel.—C. O. W.

*Composition of Honey-Dew: Influence of a Summer in which Honey-Dew is abundant, on the Character of the Honey.* E. von Raumer. Zeits. anal. Chem. 33, 397—408.

HONEY-DEW forms a thin glutinous sweet-tasting coating on the upper surface of leaves of various trees and shrubs; in some summers, especially if hot, dry weather prevail, it is very abundant, in others it is scarcely to be found. By some chemists honey-dew is looked upon as an exudation from the leaves themselves, but the major portion now regard it as a secretion of the "vine freter."

The author quotes the historical portion of the subject from Büsgen's biological study (Jenaer. Zeits. f. Naturwissenschaft., 1891, 25, 339). It appears that the first analyses of honey-dew were published in Gothe's treatise "Zur Morphologie" (Band I. S. 299, Cotta, 1817). The next to examine honey-dew was Unger in 1837 (Ber. k. Acad. Wissensch. Wien. Math.-nat. Cl., 25, 449), and further analytical values were published by Boussingault in 1872 (Comptes rend. 74, 87). The author then mentions that the most recent analyses of honey-dew were carried out

simultaneously with his own, viz., in the summer of 1893 (Jour. Amer. Chem. Soc., 1893, 350). He has not been able, however, to refer to the original paper.

The honey-dew examined in the present paper was prepared as follows from maple leaves:—The leaves were treated with cold water, and allowed to remain therewith for only a few minutes, after which the liquid was decanted off and evaporated to a syrup, which was finally decolorised by repeated evaporation over animal charcoal.

The author's results may be briefly summarised thus:

1. Almost all kinds of honey contain more or less dextrin of slight fermentability; its presence is ascertained by a comparison of the amount of sugar as found by the Soxhlet-Allihn method with that calculated from the alcohol obtained on fermentation with pressed yeast.

2. Hable's dialysis method (Zeits. anal. Chem. 33, 99), should be modified, in that the honey is not subjected directly to dialysis, but only after fermentation; the reason being that by direct dialysis the sugar, even after a protracted period, cannot be completely removed.

3. The dextrans of honey-dew probably undergo an alteration in the organism of the bee, which enhances the dialysing power of the sample.—A. R. L.

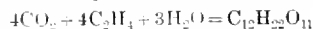
*Estimation of Saccharose.* Zeits. anal. Chem. 1894, 33, 474.

See under XXIII., page 1101.

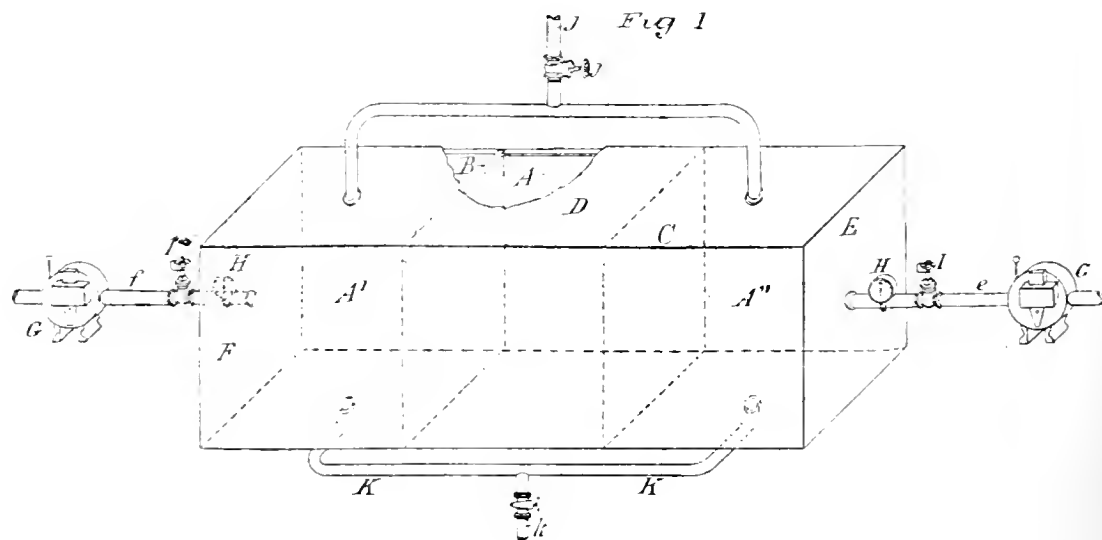
### PATENTS.

*Improvements in the Manufacture of Sugar and in Apparatus Employed therein.* J. Y. Johnson, London. From J. E. Pellegrini, Barcelona, Spain. Eng. Pat. 14,686, July 20, 1893.

THE patentee claims to have discovered a process whereby sugar (saccharose) may be synthesised from carbonic anhydride, ethylene (or other hydrocarbon of the  $C_2H_4$  series), and water; these reagents are said to interact when brought together under the conditions to be described in accordance with the equation—



The apparatus employed for carrying out the process consists of a rectangular box or case (Fig. 1) constructed of cast iron or other suitable material; the interior of this box is nickel plated and planed. It is also hermetically closed to prevent any leakage of gas. Within this box, equidistant from either end, is placed a cube of platinised porous material, the side of which is about one-third the



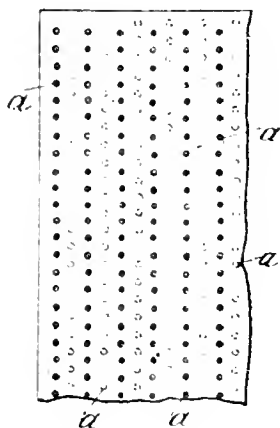
entire length of the box; this constitutes the catalytic agent. A longitudinal section of part of the cube is shown in Fig. 2, and a transverse view in Fig. 3. The best results

Fig. 2.



are obtained with blocks manufactured as follows:—Light coloured dense pumice is washed successively with hydrochloric acid, sulphuric acid, and water; it is then dried, pulverised, and mixed with 10 per cent. of dry clay (as free as possible from iron) and 3 per cent. of cork, both

Fig. 3.



being in a finely powdered condition. This mixture is made into a paste with brine of sp. gr. 1.20; it is then air-dried for about 15 days, placed on a stove heated to about 300°–400° C., where it is allowed to remain for 4–5 days, and subsequently baked in a pottery furnace. The block thus obtained is then drilled with holes or passages *a* which do not pass completely through it, but extend from each side to within say 0.8 in. from the opposite side; the passages are about 0.13 in. in diameter and are parallel to one another. After drilling, the block is immersed in a hot 10 per cent. solution of platinum tetrachloride or other suitable platinum salt, which will, when sufficiently heated, deposit finely-divided platinum within its pores; the platinising will usually have to be repeated two or three times. "The boiling in each case continued for three hours." After sufficiently heating, the block is tested with dry steam, and if it fails to decompose the latter, it is again platinised until it does so.

The block is held in position by two steel plates, one of which is shown at B and the other at C; these are perforated with holes coinciding exactly with those of the block. To the ends E and F are fixed the tubes *e* and *f*, conveying carbonic anhydride and ethylene respectively; each tube is furnished with a meter G, a gauge H, and a regulating valve I. Ethylene is passed, under slight pressure, into the chamber V' and carbonic anhydride at about the same pressure into the chamber V". After the lapse of a few minutes the valve *j* is opened and dry steam at about two or three atmospheres pressure is introduced into the chambers through the pipe J. The apparatus now becomes rapidly heated; the supply of gases and vapor is continued for about half an hour, but not so long that the temperature rises above 150° C.; a lower temperature than 100° C. will be found more suitable. The gases mix through the dry substance of the pumice, and on the expiration of the above-mentioned period, the syrup produced, containing 20–25 per cent. of sugar, is run off through the valve *k*. When the apparatus is cool the operation may be repeated. —A. R. L.

*Apparatus for Separating and Utilising Steam from Cane Juice on Leaving a Juice Heater.* A. Chapman, Liverpool. Eng. Pat. 20,221, October 26, 1893.

THE patentee claims the invention and application of an apparatus consisting of a cane-juice heater in combination with an evaporating apparatus, and of a steam and cane-juice separator comprising two or more vessels, the first connected with the second by an inverted siphon pipe, and the second (where a third is employed) similarly connected with the third. The hot juice from the heater is delivered to the first separating-vessel, whence it passes, by the inverted siphon, to the second, and from thence to the third, or to defecators or filters or elsewhere. The steam which is evolved from the juice in the first and second separating-vessels is conducted to and utilised in the evaporating apparatus or otherwise. —A. R. L.

*Improvements in the Manufacture of Animal Charcoal.* C. W. Kriens, London. Eng. Pat. 8112, April 24, 1894.

See under XIV., page 1075.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*Composition of Hops, and their Influence on Wort.* Aubry. Wochenschr. f. Brauerei, 11, 289–296, from Zeits. Gesamte Brauwesen, 1894. Part I.

THE author examined eight samples of hops for moisture (1.2–7.30 per cent.), alcoholic extract (40.87–44.69 per cent.), and aqueous extract (10.2–14.72 per cent.), and then examined the influence of these various hops on the same wort. He found that the values of these determinations gave no criterion of the values of the hops for brewing. Two hops, one a very fine Saaz hop, the other a coarse but healthy and vigorous Al-sace hop, gave the same alcohol and water extracts.

The author found that in extracting the resin of hops by light petroleum, only very little of the tannic acid is removed, and that the aqueous extract from hops contains but little of the nitrogenous matter. The ripeness of the hops does not influence the fermentability of the wort, but only the flavour of the beer. Hopped wort ferments better and more completely than unhopped wort, and yields clearer beer. The percentage of sugar in wort is not influenced by hopping, but the nitrogenous matter (both albuminoid and total) is increased thereby and to the greatest degree by the finest quality of hop. Beer brewed with inferior hops had a more fiery but coarser flavour than when fine quality hops were employed. —L. T. T.



*Finding the Specific Gravity of Barley by means of the Volumetric Barley-Balance.* Wochenschr. f. Brauerei, 11, 290.

BANZOWSKI has recently patented a "volumetric barley-balance," by means of which the specific gravity of barley may be quickly and accurately determined (to third decimal). Wölky and Heine and others have, however, shown that the specific gravity of barley cannot be taken alone as a criterion of value, but that a complete chemical analysis is also necessary.—L. T. T.

*Respiration of Malt on the Malting Floor.* H. Jaeschke. Wochenschr. f. Brauerei, 11, 473—476.

The author has carried out a series of experiments on the practical scale, in order to determine the variation of moisture and of temperature in germinating barley, and how high the percentage of carbonic acid rises in the air of the heap during the ordinary process of malting.

**Steeping.**—Barley containing 15.15 per cent. of moisture was steeped in water (changed as usual) at 46° F. for 70 hours, samples of 1,000 corns being from time to time removed and weighed. The increase of weight (disregarding the very slight loss by aqueous extract) gave the moisture absorbed by the barley. After 24 hours' steeping, the increase in weight (*i.e.*, increase in moisture) was 32.35 per cent., after 48 hours 41.49 per cent., and at the end of 70 hours 48.52 per cent. The barley was then transferred to the malting floor.

**Malting.**—The malting was carried on for nine days, the temperature of the air of the malting chamber being 43—45° F. For the first five days the heap ("piece") was turned twice a day, then once a day. The tests for temperature and moisture were always made at five different places in the "piece." There was a further absorption of moisture during the first two days to a maximum of 55.57 per cent., after which there was a gradual loss of moisture, so that on the third day the excess of moisture over that originally present in the barley was 51.75 per cent., on the fourth day 41.47 per cent., on the fifth 34.51 per cent., on the sixth 33.69 per cent., on the seventh 31.20 per cent., on the eighth 33.04 per cent., and when the malt was removed from the malting floor 28.38 per cent. The moisture in the green malt was thus 33.95 per cent. of its total weight.

The temperature in the middle of the piece gradually rose from 51° F. on the first day to 79° F. on the fourth day, and then slowly fell to 62° F. on the ninth day. The temperature near the floor was generally two or three degrees lower than in the middle of the piece, and during the most active germination (*i.e.*, from the third to the sixth day) the difference was 9° or 10° F., and in one instance 13° F.

The percentage of carbonic anhydride in the air of the piece was determined by inserting a pointed tube punctured

been drawn into the aspirator, it was considered that that in the burette was a fair sample of the air of the piece. The accompanying sketch will render the method employed clear. The average percentage of carbonic acid found, was 5.40 on the first day, rapidly increasing to 19.4 on the morning of the third day, and then falling to 3.8 at the end of the fourth day, and 0.0 on the sixth day. Estimations of oxygen were made in many of the samples, to determine whether the increase of carbonic anhydride was accompanied by a corresponding decrease in oxygen. At first this was the case, but as the percentage of carbonic anhydride increased, the sum of the carbonic anhydride and oxygen exceeded 20.9 more and more, until on the third day, when the germination was most active, the carbonic acid was 19.1 per cent., and the oxygen 4.4 per cent., or an excess of 2.6 per cent. over the normal 20.9 per cent. This excessive formation of carbonic anhydride was also observed by Schütt, and is probably due to chemical processes going on even in the absence of oxygen, by which carbonic anhydride is liberated—a kind of self-fermentation.

—L. T. T.

*The Action of Oxygen on Yeast.* H. van Laer. Wochenschr. f. Brauerei, 11, 353—359

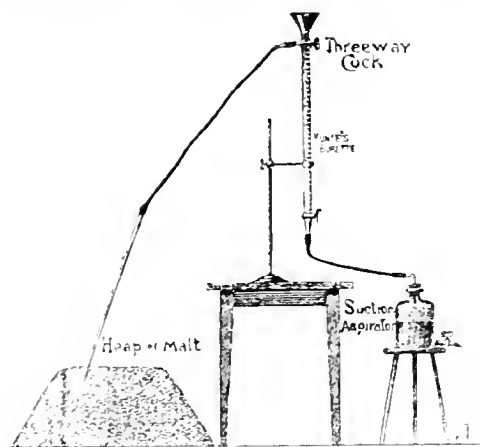
THIS paper consists of a critical discussion of the work published on this subject and especially of that centering round the question of the correctness or the reverse, of Pasteur's two statements, (a) "No alcoholic fermentation can take place without simultaneous formation, development, or increase of cells, or continuous life of already formed cells," and (b) "Fermentation is a consequence of life without air." The author points out that much, at least, of the discrepancy in views of different workers is due to a confusion of the meaning of Pasteur's "fermentative power" (*Fermentativ-vermögen*) with what may be called "fermentative activity" (*Gährthätigkeit*) and with Schützenberger's "fermentative energy." Pasteur gives as the measure of

"fermentative power" the expression  $\frac{S}{Y}$  where *S* represents the total amount of sugar fermented and *Y* the total weight of the yeast obtained. It may in fact be considered as the total fermenting capacity of the yeast-cell, *irrespective of time*, and is quite distinct from the general expression "fermentative activity" which represents the amount of sugar fermented (or alcohol produced) in a *given time*, either per unit weight of yeast or irrespective of the weight of yeast formed, or Schützenberger's more exact expression "fermentative energy" or quantity of sugar fermented in the unit of time by a unit-weight of yeast. Thus, whilst the "fermentative activity"—which is, in a measure, the most important to the brewer—is undoubtedly assisted by the presence, during a part of the fermentation at least, of oxygen, this does not in any way necessarily disprove Pasteur's statements. Thus Pasteur himself says: "Whilst the fermentative power of yeast is greatest when it is growing in the absence of free oxygen, its fermentative activity—that is its capacity of decomposing a given quantity of sugar in a given time—is diminished. The ternary substances will only be very slowly decomposed, because the vital force of the cells is weakened. Yeast produced in the presence of air decomposes more sugar per unit of time than yeast produced under any other conditions."

Another cause of the discrepancy in the conclusions drawn by various investigators, is the fact that often not enough weight is given to the varying conditions under which the different experiments are carried out, and to the influence of factors—sometimes omitted from consideration altogether.

The author draws the following conclusions:—

1. It is definitely proved that alcoholic fermentation can take place, both with and without the formation of new cells.
2. If a yeast is growing in a suitable saccharine liquid its fermentative-power (*i.e.*, the ratio  $\frac{S}{Y}$  of sugar decomposed to yeast formed) is greater the more complete is the aëration.
3. The intensity of the sugar-decomposition in a given time (fermentative energy or activity) is greater when the



with several small holes near the point, well into the piece. The other end was attached to a Bunte or Orsat gas burette, and this to an aspirator. After about a litre of air had

yeast is in contact with air than when it is living in the absence of air.

1. Under otherwise like conditions, yeast grown in the presence of air has a higher fermentative power than that grown in the absence of free oxygen.

5. When a fermenting liquid is aerated, the fermentation is rendered quicker and more complete, and in general more yeast is produced.

6. Yeast growing in a suitable medium cannot increase beyond a certain maximum, even under the most favourable conditions.

7. When a yeast which cannot further increase (*i.e.*, has reached the maximum referred to under 6) is living in a suitable nourishing medium, both the fermentative power and fermentative activity are increased by aeration.

On the other hand the author considers that the following problems still require further experimental investigation.

1. In alcoholic fermentation with formation of new yeast cells is the production of alcohol dependent on this formation of new cells, or is it an effect of the already-formed and non-increasing cells?

During fermentation, with or without new yeast-cell formation, would alcohol be produced if *all* the yeast-cells were exposed to the influence of free or atmospheric oxygen?

3. How do yeast-cells which ordinarily show the greatest reproductive increase, behave in the presence of air?

4. What are the conditions which restrict within certain limits, the increase (reproduction) of yeast in saccharine liquids, especially in beer-worts?

5. Can certain yeast species produce simultaneously more alcohol and more yeast in wort in the presence of air, than under ordinary conditions?—L. T. T.

#### *The Influence of the Compounds of Fluorine on Beer-Yeast.* J. Effront. Comptes rend. **118**, 1894, 1420—1423.

THE author has previously demonstrated (this Journal, 1892, 50) that beer-yeast, when grown in media containing fluorides, may be gradually accustomed to comparatively large doses of the reagent, to quantities in fact large enough to render ordinary non-habituated yeast completely inert. The life-process of such yeast is, however, profoundly modified. Reproduction is greatly restricted, whilst on the other hand, the fermentative power of the cells is largely augmented. Further experiments have shown that the chemical action of the yeast also suffers a change. The fact was demonstrated by making two series of fermentations of sugar solutions, &c., under strictly identical conditions, but using in one series, ordinary yeast, in the other, yeast habituated to fluorides. The results proved in every case that the treated yeast produced more alcohol, and a much smaller proportion of glycerol and succinic acid than ordinary yeast. For instance, in a glucose fermentation, the treated and ordinary yeasts yielded respectively 50.49 per cent. and 48.37 per cent. of alcohol (calculated on the dextrose fermented). The former figure approaches very closely to the theoretical value demanded by Gay-Lussac's simple equation; the latter is almost identical with Pasteur's result. As regards the formation of by-products the following figures are of interest.

	Volume of Dissolved Gases.	CO <sub>2</sub>	O	N	Wort at
I.—	cc.	cc.	cc.	cc.	} 12.5° Balling.*
Aeration by diffusion with a small opening . . . . .	8.7	2.5	1.3	4.2	
Aeration 30 minutes at 16° . . . . .	16.9	6.06	2.5	8.34	} 10° Balling.
II.—					
Aeration 10 minutes in the hot wort . . . . .	7.8	2.4	1.1	4.3	
Complementary aeration, 17 minutes at 18° . . . . .	17.9	5.8	3.66	8.44	

\* See this Journal, 1891, 821.

	Habituated Yeast.		Ordinary Yeast.	
	Strong Wort.	Weak Wort.	Strong Wort.	Weak Wort.

Alcohol formed . . . . .	12.7	10.1	12.5	9.3
Glycerol . . . . .	0.065	0.049	0.75	0.257
Succinic acid . . . . .	0.011	0.0032	0.132	..

—H. T. P.

#### *The Oxidation of Beer Wort.* P. Petit. Comptes rend. **118**, 1055—1057.

ACCORDING to Reinker, beer wort when it reaches the fermentation vats, contains an approximately constant quantity of dissolved oxygen, about 4 cc. per litre, and taking account of the products of oxidation he estimates that 800 cc. of oxygen is the largest quantity one hectolitre can absorb. The author has examined the gases dissolved in the wort and finds that after it has passed into the refrigerator that it contains large quantities of carbonic acid, and also that the proportions of oxygen to nitrogen dissolved in the wort correspond to the solubilities of these two gases in water, that is to say, about 28.5 per cent. of oxygen to 71.5 per cent. of nitrogen. A sample of the wort kept for a week in a sterilised flask closed with a cotton wool plug gave approximately the same numbers for oxygen and nitrogen, but the amount of carbonic acid had largely increased. The same result was obtained by boiling a sample of the same wort for 1½ hours when it no longer contained any gas. It was allowed to cool, air only being admitted through a very small orifice. After five days the gas was extracted from the wort and gave 13.3 cc., containing 5.7 cc. of CO<sub>2</sub>, the rest being oxygen and nitrogen in the proportion of 28.3 to 71.7 as before. The wort thus required 570 cc. of oxygen per hectolitre for chemical action. It is therefore necessary when using an air injection to regulate it so as not to remove the whole of the aroma from the wort since the latter can be oxidised to an almost indefinite extent by means of air, and even in the cold.

—T. A. L.

#### *The Oxidation of Beer Wort.* P. Petit. Comptes rend. **119**, 1894, 312—343.

IN a former note (see preceding abstract) the author examined the conditions of oxidation of beer wort as it takes place in practice; he now describes his observations upon the oxidation of wort by direct injection of air so as to produce the same phenomena and with a view to ascertaining what are the constituents of the wort that become oxidised. He aerates the wort by passing a measured volume of air through it, and then by aid of a mercury pump extracts the dissolved gases, measures them at 17° C. and 740 mm. pressure, and analyses them. The quantity of air necessary to effect complete oxidation of course varies with the concentration of the wort, and in his experiments he used a wort containing from 10 to 12 per cent. of extract. The following are the results obtained:—

It is seen that the aeration in the case of No. 11, is complete, since the quantity of oxygen dissolved is normal and bears the ratio  $\frac{1}{2}$  to the dissolved nitrogen. The volume of air injected was 110 litres per minute under a pressure of 0.6 kilo, through a volume of 400 litres of wort, and it may therefore be deduced that a sufficient oxidation and oxygenative is arrived at by injecting 750 litres of air at a rate of 110 litres per minute into a volume of one hectolitre of wort either hot or cold. The experiment on wort No. 1, indicates that a portion of the air should be injected into the hot wort, since complete saturation was not obtained after aeration for 30 minutes at 16°.

The author also finds that in the case of wort with hops,  $\frac{1}{2}$  of the carbonic acid in the dissolved gases is due to the oxidation of the constituents of the wort and  $\frac{1}{2}$  to those of the hops. He next proposes to ascertain what are the constituents of the wort thus oxidised.—H. S. P.

*The Diastase of Cereals.* J. Egaroff. Jour. Soc. Ph. Ch. Russe de Saint-Petersbourg, 1893, No. 2.

Fair diastase was obtained by extracting cereals with 30 per cent. alcohol. The extract was precipitated with absolute alcohol three times in succession. The precipitates were then suspended in water and left to settle in fractions. It was, however, found impossible to completely remove the albuminoid matters by this treatment. The elementary analysis of the purest fraction gave the following figures:—10.24 per cent. of C, 6.78 per cent. of H, 4.70 per cent. of N, 0.70 per cent. of S, 1.45 per cent. of P, and 4.60 per cent. of ash. The purification of the diastase by fractional settlement appears to have a great influence upon the reduction of the percentages of nitrogen and ash in the substance. This diastase is a white powder with a faint yellow shade. It assumes a dark blue coloration with guaiacum and hydrogen peroxide. Water rather emulsifies than dissolves it, the liquid formed being slightly opalescent. Alcohol of 75 per cent. and a semi-saturated solution of salt do not extract the diastase from gluten. The reaction of diastase is faintly alkaline, that of the ashes faintly acid. The latter contain potassium, magnesium, calcium, and phosphoric acid.—C. O. W.

*Reichler's Artificial Diastase.* J. Egaroff. Jour. Soc. Ph. Ch. Russe de Saint-Petersbourg, 1893, No. 2.

THE solution, possessing a very considerable degree of activity, which, according to Reichler, is obtained by warming gluten with weak acids ( $\text{HCl}$ ,  $\text{C}_2\text{H}_3\text{O}_2$ ,  $\text{CH}_3\text{CO}_2$ ,  $\text{KH}_2\text{PO}_4$ ) represents a solution of diastase pre-existent in the gluten, but mechanically retained by the latter. The solution of gluten in di-acid potassium phosphate decomposes rapidly whilst its saccharifying power is increasing. Under the microscope this solution is found to be full of bacteria.

A series of experiments made with this and other solutions of gluten demonstrated that they contain no diastase but what is pre-existent in the gluten. The more energetic action of the above-mentioned solution is due to the bacteria contained in it, and which, as Wortmann showed, are capable of converting starch into maltose. Jäger's observations on pancreatic appear to admit of a similar interpretation.—C. O. W.

*The Supposed Occurrence of Free Tartaric Acid in Wine.* A. Carpené. Selmi, 1894, 4, 13.

WHEN tartaric acid is added to a solution of potassium malate, succinate, pectinate, or acetate, or to acid potassium sulphate or phosphate, and also to mixtures of these salts, a precipitate of cream of tartar is obtained. This precipitation continues until all the potassium has combined with tartaric acid. Consequently it is impossible for free tartaric acid to exist in wine along with these salts. Experimenting with 100 wines a precipitate was always obtained.

The ash of a wine treated with a solution containing 3.5 grms. of tartaric acid per litre, contains 0.2–0.3 grm.

less of potassium than the natural wine-ash does. When therefore free tartaric acid is proved to be present in a wine in absence of the potassium salts of the other acids, we may rightly suspect the wine of artificial preparation.—T. A. G.

*The Influence of Cane Sugar on the Valuation of Malts and Worts.* E. Prior, Bayer. Brauerjourn. 1894, 4, 49.

THE total amounts of sugars, reducing sugars, and cane sugars were estimated (according to Meissl's method) in 13 malts. The tabulated results show that 100 parts of wort extract contain 5.59 to 8.69 per cent. of cane sugar. Of two malts containing about the same amount of reducing sugars, the one contained about 2 per cent. more cane sugar and this malt, although in all respects a good brewing malt, gave in practice a higher finished fermentation. This is attributed by the author to the greater percentage of cane sugar present.—T. A. G.

*Quantity of Steam and Capacity of Boiler required in Washing and Boiling Wort.* Wochenschr. f. Brauerei, 11, 323.

EXPERIMENTS made at Schönberg when mashing 5 tons of malt showed that 2.9 tons of steam were required for mashing and 2.2 tons for boiling the wort, or, together, 5.1 tons of steam for 14 hours' boiling. Basing calculations on a production of about 3 lb. of steam per hour per square foot of heated boiler surface, these results show that for every cwt. of malt mashed, 7.75 lb. of steam and 2.5 sq. ft. of heated boiler surface are required.—L. T. T.

*The Influence of Ferruginous Water in Malting.* Kukla. Wochenschr. f. Brauerei, 11, 350. From Oesterr. Brau-u.-Hopf-Zeit. 1894, part 5.

AN otherwise good water, but containing 2 parts in 100,000 of iron oxide caused considerable darkening of the malt, and the latter absorbed so much of the iron (23 mgrms. in 100 grms. of malt) that in mashing it gave up some of the iron to the wort, which then became dark coloured on the addition of the hops.—L. T. T.

*Effect of Alkaline Chlorides on the Flavour of Beer.* Wochenschr. f. Brauerei, 11, 289.

IT is generally considered in England that chlorides, and especially salt, exert an important influence on the flavour, apparent maturity, and brightness of beers, and salt is therefore often added to the brewing water. The recent experiments of Zuntz on "The Physiology of Taste" (Naturw. Rundschau, 7, 619) give support to this view. Zuntz finds that if salt or bitter substances are added to sweet substances in quantity insufficient of themselves to produce a decided taste, the sweetness is apparently increased. Thus, if to a 12–13 per cent. sugar solution, 0.1 per cent. of salt or of quinine sulphate be added, the sweetness of the solution appears much increased. It is thus probable that the presence of minute quantities of salt &c. has an important influence in bringing out other flavours.—L. T. T.

*Some of the Micro-Organisms causing the Diseases of Beer.* F. W. Fellowes, London. Trans. North of England Inst. of Brewing, Manchester. May 1894.

THE normal acidity of beer acts as a strong preservative against the attacks of many of the disease-producing organisms.

Wort contamination is usually due either to atmospheric infection or the use of impure pitching yeast.

Hayduck observed that the lactic ferment has strongly marked stages of fermentative activity, for instance, during 11–12 hours, 0.35 per cent. of acid was formed; 13–14,

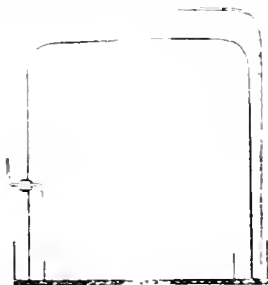
0.60 per cent.; 15—16, 0.1 per cent.; 17—18, 0.22 per cent. The percentage of alcohol in the liquor has a marked effect on the vital activity of the ferment; 2 per cent. has practically no effect, whilst 4 per cent. checks it considerably, and 6 per cent. entirely. Mr. Fellowes attributes the excessive production of lactic acid in many beers to a somewhat high rate of nitrogenous matter in conjunction with an abnormal number of lactic bacteria, which, in all probability, had their origin in a highly infected pitching yeast.

A neutral sterile beer infected with *Saccharo bacillus Pastorianus*, as Van Laer has appropriately named this organism, was found at the end of four weeks growth at 25°—30° C., to have produced volatile acid = 3.5 cc. N. alkali, and fixed acid = 24.0 cc. N. alkali per 100 cc.

Van Laer has investigated "ropy" or excessively viscid Belgian beers, and attributes their production to the action of *Bacillus Viscosus*—of which there are several varieties. The fact of "ropiness" in English beers not always affecting an entire brew appears to point to infection having taken place after the primary fermentation.

Illustrating the rapid deterioration which infected yeast may undergo, Mr. Fellowes mentions a case in which ordinary commercial yeast was used for a series of laboratory experiments. The yeast in its fresh state gave a satisfactory fermentation; with, however, a rather high acidity. It was passed through some three or four generations by successive growths in sterile wort. Each successive generation was very strongly reduced in fermentative power, until a stage was reached when it failed to produce more than a slight alcoholic fermentation. In all cases, however, (about three dozen in number) the beer ultimately became turbid and decidedly ropy; the viscosimeter taking 53 seconds to deliver 25 cc. of distilled water at 20° C., and 98 seconds to deliver an equal volume of the viscid beer at the same temperature.

The bacteriological examination of air and water are dealt with, and a new apparatus, introduced by the author and Mr. A. J. Banks, for the culture of anaerobic organisms, is described. This consists of a glass dish having an inner circular wall of lesser diameter than that of the bell-jar. The bell-jar has a tubulated opening at the side, into which a caoutchouc stopper holding a doubly bent glass tube is fixed; this serves as a gas inlet. The top of the chamber



has a similar opening, and is likewise closed by a stopper through which is passed a bent glass tube of sufficient length to reach well below the inner wall of the dish. After the chamber has received its charge of plates, a layer of light colorless mineral oil is run into the outer space until the end of the gas outlet becomes immersed. The inlet tube is connected with a gas generator and a rapid current of nitrogen, hydrogen, or carbonic acid gas passed through until the air is expelled. The apparatus, after being placed in the incubator, is connected with a gas holder from which an almost imperceptible current of gas is forced by means of a suitably regulated siphon affixed thereto.—A. J. B.

*Rectification of Alcohol.* E. Sorel. *Comptes rend.* 118, 1213—1215.

It has been shown by the author in previous papers (*Comptes rend.* 108, 1128, 1205, 1317) that the volatile impurities contained in a liquid undergoing rectification

ascend to higher levels or descend towards the still, according as the difference  $K - \frac{P}{p}$  is positive or negative. In this expression  $K$  is the ratio of the weight of impurity in 1 kilo. of the vapour and in 1 kilo. of the liquid in contact with a given level;  $P$  is the weight of the reflux liquid, and  $p$  is the weight of vapour which traverses the given level in unit time.  $\frac{P}{p}$  is therefore of necessity less than unity; but it is the nearer unity the more concentrated the alcohol. The author has now determined the value of  $K$  for fermentation isomyl alcohol, ethyl formate, methyl acetate, ethyl acetate, ethyl isobutyrate, ethyl isovalerate, isomyl acetate, and isomyl isovalerate. The results are given in tabular form and the following deductions are drawn from them:—The value of  $K$  decreases with the increase of the molecular weight and the rise of the boiling point; but there is no apparent relation between the three variables.—A. R. L.

*Utilisation of Vintage Dregs.* A. Muntz. *Comptes rend.* 118, 1221—1226.

The dregs of the grapes in wine manufacture are more often a source of little profit, and in some cases are a source of actual loss. The author took up the problem of the utilisation of these dregs during the vintage of 1892. The pressed dregs were found to contain 60 per cent. of their weight of wine; it is possible to recover this wine by displacement with water, and the "piquette" thus obtained is superior in strength to that consumed by the farm labourers. The surplus of these "piquettes" when submitted to distillation gives an "eau de vie" of superior value to that obtained by the direct distillation of the dregs. After having separated the wine from the dregs the latter have lost none of their nutritive value, which was proved by the fact that a flock of sheep were fed on the material during the winter.

The author resumed his studies during the vintage of 1893, the operations being conducted in the following manner:—The dregs as they leave the presses are introduced without delay into cylindrical vats, where they are treated with 4—5 per cent. of water to assist the pressing down. When the vat is full, the surface of the dregs is treated uniformly every quarter of an hour with water, which is distributed uniformly over the surface; in the present case there were used every quarter of an hour, 12 litres of water, this being the requisite amount for a cask of 80 hectolitres capacity. The water thus presses out the wine contained in the dregs, and the first runnings which collect at the bottom of the vat consist of wine of excellent quality without admixture of water. The operation is arrested when the liquid only contains 1 per cent. of alcohol which will be attained at the end of four days, the liquid having then but a faint colour. The more concentrated liquors are reserved for direct consumption or distillation; the more dilute are used for the lixiviation of a fresh quantity of dregs.—A. R. L.

*Estimation of Saccharose.* *Zeits. anal. Chem.* 1894, 33, 474.

See under XXIII., page 1101.

*The Properties of Caramel.* A. Sabanejeff and A. Antouchevitch. *Soc. Pho. Ch. de Saint, Pétersbourg*, No. 1, 1893.

See under XVI., page 1076.

*The Quantitative Separation of Amorphous Nitrogenous Organic Compounds contained in Beer-Wort.* H. Schjörning. *Zeits. anal. Chem.* 33, 1894, 263.

See under XXIII., page 1103.

*The Detection of Abrastol in Wines.* L. Briand. Comptes rend. 118, 1894, 925.

See under XXIII., page 1099.

### PATENTS.

*Improvements in the Manufacture of Yeast.* W. S. Squire, London. Eng. Pat. 19,843, October 21, 1893.

WHEN distiller's spent wash is heated to a temperature corresponding to about two atmospheres above the atmospheric pressure, it becomes perfectly clear and bright after filtration or subsidence, less viscid, and more suitable for the manufacture of yeast. In the ordinary process of brewing in distilleries the grain is treated with water at, or about, the boiling point, and thus most of the albuminous matters are coagulated and lost for yeast manufacture. To avoid this loss, an albuminous solution is made by treating the grain, before boiling, with ordinary water, spent wash or, preferably, spent wash which has been purified as above described, at a temperature not exceeding 130° to 140° F. Albuminous solutions thus prepared are to be used instead of water or spent wash for mashing malt, rye, or other suitable grain before the addition of the boiled maize, as described by the patentee in Eng. Pats. 12,058 and 12,463 of 1885, and 5457 and 8982 of 1886 (this Journal, 1886 542, also 1887, 297 and 514).

For the manufacture of yeast a mash, made in this manner, should, after the addition of the boiled maize, have a temperature of about 145° F., and when saccharification is complete spent wash or one of the solvents named is added so as to obtain a wort of specific gravity 1.030. The wort is then clarified, cooled to 80° F., yeast is added and air blown in until fermentation is complete. The yeast is collected and pressed, and the resulting wash may be distilled to recover the spirit.—T. A. G.

*An Improved Method of Utilizing and Means for Treating Yeast.* E. W. Greening, Thames-Ditton, Surrey, and E. O. Greening, Lee, Kent. Eng. Pat. 20,960, October 24, 1893.

WASTE brewer's yeast is dried, conveniently, by passing through steam-heated rollers, until the moisture is reduced from the normal amount of 75 or 80 per cent. to less than 10 per cent. The thin flakes thus obtained are scraped from the rollers by suitably arranged knives, cooled in trough conveyors by continual turning and stirring, and then ground into powder or meal of any requisite degree of fineness.

Fresh yeast meal, being exceptionally rich in albuminous matters, and the tonic principle of hops, is serviceable for admixture with other substances, to increase their feeding value and flavour when used as feeding-meals, or cakes, for cattle.

Meal from stale or dirty yeast forms a valuable manure since it is rich in phosphates, potash, nitrogenous matters, &c. It may be used by itself, or mixed with other fertilizers.

—T. A. G.

*A Method of Treating Grain and other Materials for Employment in Brewing and for other useful Purposes.* T. Bayley, Dublin. Eng. Pat. 20,930, November 4, 1893.

GRAIN and other starch containing materials are treated with dilute acid at or below a temperature of 40° C. (104° F.) whereby the starch is, for the greater part, converted into the soluble form, and when the grain is subsequently treated with hot water at 190° F. or upwards, a comparatively limpid solution is obtained, and any undue thickening or viscosity avoided. The time required for conversion depends upon the nature and strength of the acid used and upon the temperature. If rice be digested in the cold with sulphuric acid, diluted with nine times its weight of water, modification of the starch results in 21 days, whilst at a temperature of 100° F. about 48 hours suffice for dilute sulphuric acid and seven or eight days for a saturated solution of sulphurous acid. At the end of the necessary

time the acid is removed, and the rice is well washed with water until the washings are no longer acid. It is then ready for use either in the wet state or after being dried.

—T. A. G.

*Improvements in the Manufacture of Compounds suitable for the Production of Ginger Beer, Herb or Botanic Beers, or other Fermented Beverages.* J. Heron, London. Eng. Pat. 21,332, November 9, 1893.

THE object of the invention is the preparation of solid compounds containing all the necessary ingredients, except the actual water, required for producing "home-brewed" ginger beer or other fermented beverages.

For instance, a ginger-beer compound is made thus:—A solution of solid dextro-glucose is concentrated until it contains only 10 to 12 per cent. of water. Whilst still hot, the ingredients are added, the mixture is cooled to about 70° F. and thoroughly incorporated with fresh, pressed yeast (about two parts, by weight, to 100 parts of glucose). The mixture is run into moulds, where it sets into a hard, compact mass. When required for use it is broken up into small pieces and dissolved in water at a temperature of 70° to 80° F. The water should be previously boiled to destroy any germs which it may contain. After standing 12 hours the beer is ready to bottle. In cases where any of the ingredients can only be extracted by boiling water they are not incorporated with the glucose and yeast, but are boiled in the water, which is then cooled, and the prepared compound is added.—T. A. G.

*A New or Improved Apparatus for Fining Beers and other Liquors in Cask, applicable also for Priming and Filling up Casks.* J. G. Crossman, Cork. Eng. Pat. 2152, January 31, 1894.

THE apparatus consists of a portable hand-pump of which the cylinder is fitted with plunger, piston-rod, and handle. The top cover has a packed joint, and the lower cover carries a projecting nozzle and thimble. The rubber-covered thimble is tapered to fit any bung-hole and ensure a tight joint. An adjusting nut on the piston-rod is used to regulate the volume of contents to be delivered.

In operation the finings are drawn into the cylinder by an instroke of the plunger, the nozzle is passed through the bung-hole into a full cask, the rubber securing a tight joint, and the finings are forcibly injected. The displaced beer simultaneously passes up an annular space between the nozzle and the thimble, and through the side tube into the cylinder, and at the next instroke is forced out again to be received into any suitable receptacle.—T. A. G.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY AND WATER PURIFICATION, DISINFECTANTS.

### (A.)—CHEMISTRY OF FOODS.

*Testing Milks with Rennet.* R. Lezé and E. Hilsont. Comptes rend. 118, 1069.

See under XXIII., page 1099.

*Centrifugal Methods of Milk Analysis.* A. Scott. A Paper read before the Glasgow City Analysts' Soc. Abstracts, No. 4, 1893-94, 9.

See under XXIII., page 1104.

*Soxhlet's Arcometric Method of Determining the Fat in Milk.* H. Zimpe. *Chem. Zeit.* 1894, **18**, 392.

See under XXIII., page 1104.

*Composition of "Honey Dew": Influence of a Summer in which "Honey Dew" is abundant, on the Character of the Honey.* E. von Raumer. *Zeits. anal. Chem.* **33**, 397.

See under XVI., page 1076.

#### PATENTS.

*New or Improved Compositions for Preserving Beer, Wine, Milk, and other Liquids.* J. W. Pickles, Leeds. Eng. Pat. 18,736, October 6, 1893.

THE compositions described contain a mixture of boracic acid, sodium salicylate, and potassium bicarbonate, which has been allowed to absorb sulphur dioxide (produced by burning sulphur), with or without addition of capsicum and primrose yellow, and a mixture similar to the above, but without the sodium salicylate.—T. E.

*Improvements in or connected with the Production of Butter from Cream or Milk.* S. H. Wright, Monmouth. Eng. Pat. 18,708, November 25, 1893.

THE object of the invention is to produce butter from milk or cream by passing a rapid current of air through it. The current of air is produced by exhausting the air over the liquid with a water pump, or by forcing the air into the liquid under pressure. The butter can then be washed in the same apparatus by forcing water through it. The apparatus (which is described and illustrated by three sheets of drawings) is stated to be of an inexpensive nature.—T. E.

*Improvements in or relating to the Roasting of Coffee, Cocoa, and similar Materials.* A. J. Boulton, From C. A. Otto, Dresden. Eng. Pat. 22,645, November 25, 1893.

THE roasting is done by passing the material in small quantities through a helically-wound and heated tube. The improvements claimed are the different pieces of machinery necessary for carrying this out. They consist mainly of the helically-wound rotating tube heated in any convenient way and containing heart-shaped scoops for the purpose of more completely mixing the material passing through the tube, and of a hollow rotating ball, of a capacity which can be varied, by means of which small quantities of the material may be fed into the tube at small successive intervals.

—T. E.

*Improvements in Apparatus specially suitable for Use in Estimating the Amount of Fat in Milk and other Substances.* R. W. Woosnam and the Dairy Supply Co., Lim., London. Eng. Pat. 23,039, November 30, 1893.

See under XXIII., page 1091.

*Improvements in the Means of Using Baking Powder.* W. G. Dunn, East Croydon, and J. A. Brown and J. Polson, Paisley. Eng. Pat. 9183, May 9, 1894.

ANY of the well-known chemical raising agents is ground up with five times its weight of a non-nitrogenous flour, such as corn-flour. The injurious action of the raising agent on the nitrogenous constituents of the flour generally employed in making baking powder is thus avoided. The patentees also state that their preparation keeps better, is more easily distributed uniformly through the dough, and that the addition of corn-flour improves many kinds of bread, especially when milk is used.—T. E.

*Improvements in Compound Edible Fats.* A. W. Winter, Chicago, U.S.A. Eng. Pat. 9526, May 15, 1894.

THE object of the patent is the production of fats which may be used for food, containing about 60 per cent of refined mineral oil mixed with varying quantities of animal or vegetable fats and oils. Eleven claims for different mixtures are made; for details of their composition and preparation the specification may be referred to.—T. E.

*Peptonised Extract of Malted and other Cereals.* Peptine Maltine, Lim., and G. Bartlett, Leicester. Eng. Pat. 12,174, June 23, 1894.

THE cereals are extracted with water at 140° F., and 1 part of pepsin and 1 part B.P. hydrochloric acid to 2,000 parts of albumen added; the mixture is then heated to 104° F. for six hours and evaporated *in vacuo*. The peptones formed render it a powerful digestive agent, and it is said to keep for a considerable period.—T. E.

#### (B.)—SANITARY CHEMISTRY.

*Estimation of Nitrates and Nitrites in Waters.* J. Fraser. Communicated to the Glasgow City Analysts' Society. Abstracts, No. 4, 1893-94, 47.

See under XXIII., page 1095.

*The Removal of Iron from Drinking-Water.* H. Schuster. *Zeits. des Ing. und Arch. Ver. zu Hannover*, 1894, 297; *Proc. Inst. Civil Eng.* 1894, **118**, 34-35.

AT Aurich, in East Friesland, a tube-well 36 mm. in diameter was put down to a depth of 84 metres in alluvial ground, for the purpose of supplying the barracks; but the water, although free from organic matter, was unfit for use on account of the large amount of iron in solution, amounting to 19.2 mgrms. per litre, and, when exposed to the air, became brown and turbid from the formation of ferric hydrate when the carbon dioxide which kept the iron in solution, had escaped. As no other supply could be got, it was decided to purify it by the method adopted by C. Piefke, the separation of the iron being facilitated by breaking up the mass of water into numerous fine streams so as to obtain the largest possible contact with the atmosphere. The apparatus consists of a rectangular iron scrubber 3 metres high, 2 metres long, and 1 metre broad, filled with lumps of coke. The water is distributed by a series of perforated plates uniformly over the top of the coke-column, and passes downward into a collecting chamber below, and from thence through a sand-filter bed 640 mm. thick, with about 11 square metres area, that of the scrubber being 2 square metres. The proportion of iron was diminished from 19.2 to 7.21 mgrms. per litre in the scrubber, and to 1.08 mgrms. after passing the filter. The apparatus was intended to treat 3½ cubic metres per hour, under which condition the rate of delivery per square metre was 1,750 litres in the scrubber and 380 litres in the filter.

The removal of the ochre deposit on the coke is effected from time to time by stopping the communication with the filter and allowing the water from the pump to flow for a short time in a single stream through each of the distributing plates in succession. The strong current thus obtained detaches the deposit. A similar coating accumulates on the top of the filter, rendering it nearly, if not quite, impermeable to water, so that about 2 metres of the upper part must be renewed at intervals of three or four weeks.

#### PATENTS.

*An Improved Device for Filtering Water.* S. Wittman, London. Eng. Pat. 20,259, October 27, 1893.

IN the ordinary filter, charcoal is contained in a vessel into which water is poured and from which it percolates into another vessel or compartment. The novelty consists in making the first vessel itself the filtering medium, by fashion-

ing it out of a mixture of animal charcoal, clay, and ground coke or lignite, and firing it in a closed kiln from which air is excluded. The vessel may be of any shape, preferably egg-shape, and in moulding it starch or sawdust may be substituted for the coke or lignite.—L. A.

*Improvements in the Utilisation of Sewage Sludge.*  
T. Twynnam, Egham, Surrey. Eng. Pat. 21,024, November 6, 1893.

THE patentee proposes to utilise well-pressed sewage sludge by charging it into a blast-furnace making phosphoric pig-iron. "The carbonaceous and volatile matter in the pressed sludge (generally amounting to about 30 per cent.) serve as part of the requisite fuel, the lime and alumina go to form a fusible slag, the iron oxide present being reduced to metal, whilst the phosphoric acid is all reduced to phosphorus, which enters into the pig-iron, being eventually recovered as a valuable manure in the form of basic slag when the pig-iron is used in the manufacture of basic steel. All the ammonia of the sludge passes away in the furnace gases, and is recovered by condensation in the usual manner.

—L. A.

*Improvements in Means for Cremating and Destroying Obnoxious Gases and Fumes from Dust Destroyers and the like.* T. W. Baker, London. Eng. Pat. 21,820, November 15, 1893.

THE inventor proposes to destroy or render innocuous the gases given off by the destructors of town refuse, and also other objectionable gases, by conducting them into the combustion space of the furnaces of steam boilers, so mixing them with the furnace gases at their hottest and burning them, thus utilising the heat given off during the operation.

At present, a special furnace, known as a cremator, has to be employed for rendering the destructor-gases innocuous.

The inventor prefers to use the dry-back marine steam boiler, with two or more furnaces and a common combustion chamber connecting the furnaces with the return tubes; in this case he simply connects the destructor-gas flue to the combustion chamber.

With wet-back marine boilers, the destructor-gases pass through openings made through the stayed space at the back of the combustion chamber or chambers. With Lancashire or with Cornish boilers he inserts between, and at a lower level than, the furnaces, another tube, similar to the third flue of a three-flued boiler, but reaching only to the back of the furnaces, which connects up with the two combustion spaces (this modification is specially claimed).

With water-tube boilers the inventor uses a double back to the furnaces, and leads the destructor-gases through the space between the two backs, into the combustion space, so that fuel-gases and destructor-gases mix above the fuel and also in the spaces between the water-tubes.

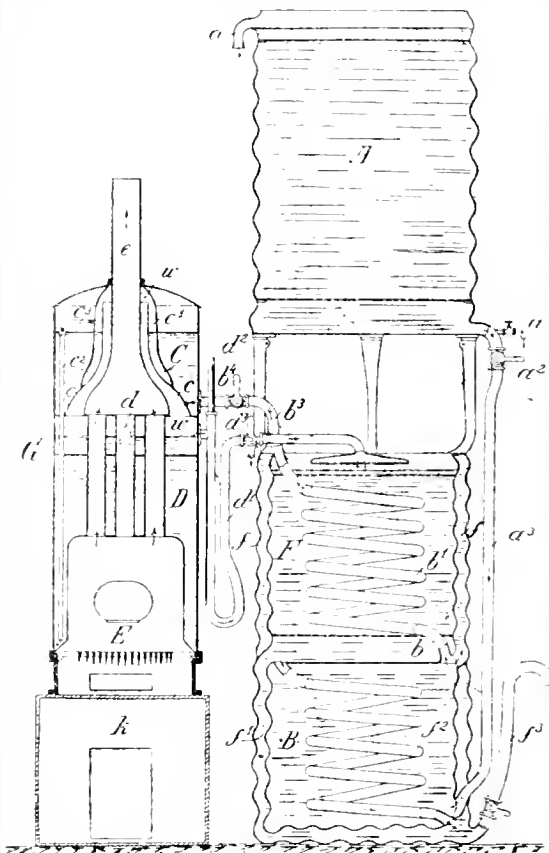
In some cases, however, he prefers to pass the destructor-gases through the burning fuel, and in this case introduces them into the closed ash-pit, claiming the use of fans for this purpose.—E. R. B.

*Improved Apparatus for Filtering and Sterilising Water.*  
H. Kowalski, Vienna. Eng. Pat. 15,487, August 14, 1894.

THIS is an apparatus for providing a continuous supply of sterilised and filtered water.

The water flowing into the vessel A, (see figure) and maintained at a constant level by the overflow *a*, passes downwards, through a layer of filtering material in the bottom of A, into the pipe *a*<sup>1</sup>, which conducts it into the compartment B, from which it rises through the pipe *b* into the coil *b*<sup>1</sup>, where it becomes heated by the hot water in F. A portion of the water from B rises between the double walls of the vessel F, and meeting, in the pipe *b*<sup>2</sup>, the water which has passed up the coil, the mingled stream flows into the heater C, whence it overflows, down the pipe *c*<sup>1</sup>, into the bottom of the

boiler D. The water is here boiled by the heat of the furnace E, and overflows through the U-pipe *d*<sup>1</sup>, which terminates in a rose, into the vessel F, where it is partially cooled by the cold water passing up through the space *f* and the coil *b*<sup>1</sup>. At the bottom of F there is a second filtering layer, and, after passing through this, the sterilised water is further cooled by flowing partly between the double sides of the vessel B and partly through the coil *f*<sup>2</sup>, and is finally drawn off through the cock and pipe *f*<sup>3</sup> for use.



IMPROVED APPARATUS FOR FILTERING AND STERILISING WATER.

The steam generated in the boiler D rises through the space *w*, between the chimney *e* and the dome *c*, and, finding an outlet through the pipe *c*<sup>2</sup>, condenses in the water passing down the pipe *c*<sup>1</sup>: any excess of steam condenses in the water which gets through the perforations in the lower part of the outer dome *c*<sup>1</sup>. The cover of the vessel C is left loose to prevent the steam pressure from accumulating, and also to prevent the formation of a vacuum if the boiling should slacken or cease. *d*<sup>2</sup> is a thermometer. The vessels A, B, and F are made of corrugated sheet iron, and are of such relative dimensions that they may be packed one within the other for transport.—L. A.

## (C).—DISINFECTANTS.

### PATENT.

*Improvements in Disinfecting and Fumigating Apparatus.*  
G. G. Crosby, New York. Eng. Pat. 22,820, November 28, 1893.

THIS specification describes a portable box or case containing a mechanical contrivance for fumigating the air of a room. The mechanical contrivance consists of three parts: 1st, perforated trays containing absorbent material saturated



with a disinfectant or perfume; 2nd, an air propeller driven by an electro- or other motor; 3rd, a battery to work the electro-motor. When the motor is set going, air is drawn through an opening in one end of the box, perfumed by passing through the material contained in the trays, and ejected through an opening in the other end of the box.

—L. A.

## XIX.—PAPER, PASTEBOARD, Etc.

### PATENTS.

*Improvements in Apparatus used in the Manufacture of Paper Pulp.* W. H. Caldwell, Loanhead, Midlothian. Eng. Pat. 15,332, August 11, 1893.

THE improvements relate to the arrangement of the knives or blades of a heating or pulping engine. According to the inventor, the fibres, when flattened by pressure, are capable of holding the water on the machine wire longer than when in the original cylindrical form. The object of the invention is to flatten the fibres by pressure while they are passing between the bars or knives of the grinding surfaces, and this is claimed to be accomplished by an apparatus constructed as follows:—Within a cylindrical shell is a heating roll having knives of the ordinary type fixed upon its circumference. Outside this roll is a fixed cylinder having another set of knives which are movable in radial slots, and corresponding to the bed plate of the "Hollander." Upon the exterior surface of this cylinder is a second cylinder constructed of rubber or similar flexible material, and upon which the distal end of the movable knives rest. The space between this latter cylinder and the outer shell is filled with water under any desired pressure, which pressure, acting upon the flexible cylinder, is transmitted to the pulp through the movable knives. The general construction of the apparatus and the mode of driving is of the usual pulp-refiner type.—S. P. E.

*Improvements in Obtaining Useful Products from the Liquors resulting from the Manufacture of Cellulose or Fibres by the Sulphite Process.* C. D. Ekman, London. Eng. Pat. 20,036, October 24, 1893.

THE improvements relate to a method of recovering some of the organic compounds contained in the spent liquor from the manufacture of cellulose fibre from wood by the sulphite process. The waste liquor is first clarified by allowing it to settle or by passing through filter beds, after which it is concentrated, preferably to about 63° Tw. at 85° C., in suitable vessels having an acid-resisting lining, and heated by steam in a leaden coil or jacket. Before evaporating, the liquor may be made alkaline by the addition of any of the caustic or carbonate compounds of the alkaline earths, in which case an acid-resisting lining in the evaporating vessel is unnecessary. To the concentrated liquor sulphuric, hydrochloric, or oxalic acid or an acid salt is added to improve the colour of the product formed, which is somewhat hygroscopic and contains a compound resembling dextrine and which the author has called "dextrone." To obtain this product, soluble salts, as the sulphate or chloride of sodium, &c., are added to the boiling liquor as long as the dextrone separates out, which, rising to the surface, may be easily removed. When cold this substance is a semi-solid, and may be either dissolved in water or dried and reduced to a powder. Mixed with gelatine or glue it forms a substance similar to the *gela-lignosine* described by Messrs. Cross and Bevan, and, like that compound, when dissolved in a suitable solvent, as sulphite of soda, can be employed as a mordant or size for textile fabrics, paper, &c. The liquor remaining after removing the dextrone may be further employed for the production of the *gela-lignosine*-like substance, by adding a concentrated solution of glue or gelatin as long as the compound separates out.

—S. P. E.

*Improvements in the Treatment of Sulphite Pulp used in the Manufacture of Paper and the like from Wood.* E. Partridge, Glossop. Eng. Pat. 3440, February 17, 1894.

THE improvement consists in the addition of liquid hydrocarbon as petroleum, preferably of a specific gravity of 0.800 to 0.850, for the purpose of dissolving and removing any resinous material that may be present in the boiled sulphite wood-pulp, and thus prevent the formation of the black resinous specks that are usually present in the finished pulp and upon the machinery employed. The proportion of petroleum recommended for this purpose is about six to eight quarts to one ton of air-dry pulp, added gradually while the pulp is being "opened" or beaten up in the rag engine, pulp opener, or washing engine.

—S. P. E.

*Improvements in the Manufacture of Parchment.* H. O. Brand, Brentford, Middlesex. Eng. Pat. 14,384, July 26, 1894.

IN addition to the lime-water washing, claimed in a former specification (this Journal, 1893, 946), the parchment is to be treated with alum, the object being to further harden the skin and to prevent the ink from soaking in. The alum is added, in the proportion of half a pound to 90 galls. of water, to the bath or washing of lime-water already referred to. The finishing process is similar to that previously described (*loc. cit.*), save that before the final rubbing with flannel the parchment is cleaned by rubbing with sand or glass paper.—A. G. B.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*The Essence Ylang-Ylang.* M. A. Reyher, Bull. Soc. Chim. 1894, **11**—12, 576—583.

THIS essence, in which up to the present only benzoic acid has been definitely recognised, contains quite a number of substances. Benzoic and acetic acid (in the form of their esters) appear to be present in the proportion respectively of 9 and 7 per cent. Ylangol constitutes about 30 to 32 per cent. of the essence, and the sesquiterpene present is contained in similar amount. The remaining 20 per cent. comprise the more volatile portions together with the resinoid constituents (polyterpenes, &c.).

With regard to Ylangol, it resembles Licareol (Barbier) in its rotatory power in alcoholic solution ( $[\alpha]_D = -19.3$ ). Oxidation by means of chromic acid mixture furnishes an oil with an odour like citral, and forming a crystalline compound with sodium bisulphite. Ylang-ylang contains, it appears, an alcohol of the geraniol series, with the formula  $C_{10}H_{18}O$ .—W. S.

*The Rhodinol of the Essence of Pelargonium.* Ph. Barbier and L. Bouveault. Comptes rend. **119**, 1894, 334—337.

THE chief part of the essence of pelargonium is a colourless, slightly oily liquid possessing a very strong odour of roses. It boils at 115°—116° under a pressure of 10 mm., its density at 0° is 0.8866, and through a thickness of 20 cm. it gives a deviation of  $-12.28^\circ$ .

That this compound is an alcohol is demonstrated by its easy conversion into its acetic ether by heating to 150°—160° with an excess of acetic anhydride. This acetate boils at 120° under 10 mm. pressure, and has an agreeable odour, and its density at 0° is 0.9458. That it is a primary alcohol is shown by its yielding an aldehyde and an acid on oxidation. The authors have prepared the oximide from the aldehyde, and from this the nitrile, which has the composition  $C_{10}H_{15}N$ ; the aldehyde has therefore the composition  $C_{10}H_{16}O$ . This nitrile fixes one molecule of



bromine. They have also isolated from the oxidation products of the rhodinol of pelargonium, an acid boiling at 149—150°, and have analysed its barium salt. It has the composition  $C_{10}H_{16}O_4$ . A more complete oxidation of the alcohol yields a considerable quantity of dimethylacetone and a crystallised acid which separates in fine needles melting at 100°. The composition of this acid is  $C_{10}H_{16}O_4$ . It distils without alteration above 300° at the ordinary pressure and at 205° under 10 mm. pressure. Its formation is indicated by the formula—



It is a dibasic saturated acid. The authors conclude that it is a methyl adipic acid. The alcohols isomeric with the rhodinol of pelargonium (geraniol, linaldolol), with which it has been confounded until now, readily lose one molecule of water and thus yield terpenes,  $C_{10}H_{16}$ . The alcohol in question, however, behaves differently: acid potassium sulphate converts it into viscous products of very high boiling point, no doubt oxides, and only a very small amount of hydrocarbon is formed. Hydrochloric acid, which etherifies its isomerides in the cold so easily, only attacks the rhodinol of pelargonium when heated, and the hydrochloride then produced, when it is heated with potassium acetate, yields the acetate of rhodinol, whilst the hydrochlorides of the isomerides yield terpenes.—H. S. P.

#### A Natural Unsaturated Ketone in Lemon-Grass Oil.

Ph. Barbier and L. Bouveault. *Comptes rend.* **113**, 983—986.

ACCORDING to Dodge (*Amer. Chem. J.* **12**, 553; this Journal, 1891, 158), oil of lemon-grass contains an aldehyde,  $C_{11}H_{16}O$ , to which he gave the name citridic aldehyde, but which was subsequently found to be identical with geranaldehyde (Semmler, *Ber.* **24**, 201—211; this Journal, 1891, 382). With a view of obtaining it the authors have distilled a large quantity of lemon-grass oil in a vacuum. After four or five distillations three fractions were obtained, one boiling at 65°—75° C. at 12 mm., the second at 110°—115°, mainly consisting of geranaldehyde, and the third a brown viscous fluid, which was not further investigated. The lowest fraction distils under atmospheric pressure at 170°—175° C., and generally consisted of a mixture of a terpene and an unsaturated ketone, the amount of the former varying from 0 to 50 per cent. of the fraction. The ketone was separated by means of its bisulphite compound, and was found to be methyl-hexylene ketone. Although the physical properties of this ketone are very similar to those of an isomeric compound obtained by Wallach (*Annalen*, **28**, 318) by distilling cinoleic aldehyde, the two substances react differently with zinc chloride.

Tieman and Semmler, by oxidising geraniol and geranaldehyde, also obtained an isomeric compound, which they termed citral. This, according to Tieman and Semmler, gives a well-defined crystalline bromine derivative, whereas the new ketone only gives a yellow viscous mass which will not crystallise. The authors have also examined the terpene which occurs together with the ketone in lemon-grass oil. It was purified by distilling over sodium and boiled at 175° C. Its specific rotatory power is  $\alpha_D^{20} = -5.48$ . It is not a limonene, and the authors are of opinion that the very varying quantities of this terpene in samples of lemon-grass oil, point to its presence as an adulterant.—T. A. L.

#### The Aldehyde of Lemon-Grass Oil.

Ph. Barbier and L. Bouveault. *Comptes rend.* **113**, 1059—1062.

THE authors have studied certain of the reactions of geranaldehyde, as the conclusions they have come to with regard to methyl-hexylene ketone are at variance with those of Tieman and Semmler. On oxidation with a sufficient quantity of bichromate to evolve 4 atoms of oxygen, geranaldehyde is decomposed into a mixture of volatile and non-volatile acids, the former consisting chiefly of acetic and formic acids. By boiling geranaldehyde with three times its weight of glacial acetic acid it is readily converted into xymene. The formula proposed by Tieman and Semmler for geranaldehyde explains

this reaction, but the oxidation of a substance constituted according to them ought to give terebic acid on oxidation. This, however, is not formed, neither does their formula explain the formation of methyl-hexylene ketone, carboxylic acid, or terebic acid. T. A. L.

#### Geraniol from Oil of *Andropogon Schenanthus*.

P. Barbier and L. Bouveault. *Comptes rend.* **118**, 1154—1157.

THE Geraniol of *Andropogon Schenanthus* furnished the same products of oxidation as citridic aldehyde, and the authors therefore admit the truth of Tieman and Semmler's observation that the citridic aldehyde is really the aldehyde of the geraniol of *Andropogon Schenanthus*.

The results of experiments with the essence of Pelargonium all point to the conclusion that this essence is absolutely different from the essence of *Andropogon Schenanthus*. Hence the alcohol of the essence of *Andropogon Schenanthus*, must no longer be termed "Geraniol," and the authors propose the name "Lemonol," which indicates its relation to citridic aldehyde, to which the name "Lemonal" might be given, thus indicating its origin (essence of lemon grass, *Andropogon citratus*) as well as its chemical functions.

#### The Physiological and Therapeutic Action of the Homologues of Quinine.

E. Grimaux and Laborde and Bourru. *Comptes rend.* **118**, 1294, 1303—1306.

GRIMAUX and Arnaud have shown (this Journal, 1891, 723, 770; 1892, 631—632) that quinine is the methyl-ether of cupreine. Since then other allyl derivatives of cupreine have been prepared and their relative physiological and therapeutic values have been investigated by Laborde and Bourru respectively. The results in general prove that the activity of these homologous bases increases as the series is ascended.

**Physiological Action.**—Cupreine administered subcutaneously to guinea-pigs, &c., produces local anaesthesia persisting for several days, and an increase in temperature of about 1° C. Its toxic influence is about half that of quinine.

**Ethyl Cupreine** resembles quinine in its action, but has a more marked effect, producing "quinine intoxication," stupor, bilateral tremor, and an average decrease in temperature of 2°—3° C. 0.15 gm. caused death in the case of a guinea-pig weighing 400 gm.

**Propyl Cupreine** is analogous to the above, but its action is still more intense. An injection of 0.025 gm. reduced the temperature of a guinea-pig by 2° in 15 minutes, 5° in 2 hours, and produced stupor, collapse, &c.; whilst 0.05 gm. was fatal, death being due to epileptic convulsions and suffocation.

**Therapeutic Action.** Cupreine administered internally in various fever cases exerted only a very slight and transient antiperiodic action.

**Ethyl cupreine** employed as basic sulphate (0.5 to 0.75 gm.) was found to have an excellent anti-periodic influence. Notably in two instances of fever, complete exemption from further attacks was secured by 3—4 doses of ethyl-cupreine, although repeated use of quinine had failed to afford relief. The toxic action of the drug when employed at the above rate was nil.

**Propyl Cupreine** has a still more powerful antiperiodic function, and exerts a very marked influence on temperature. In a case of typhoid fever, doses of 0.5 gm. administered from the third to the eighth day, gradually reduced the temperature from 103.3 to 37°—4. The base is, however, very toxic, and in every instance caused nausea, dizziness, &c.

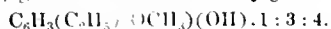
It appears, therefore, that ethyl-cupreine might with advantage be employed where the quinine fails; whilst propyl-cupreine might find application as a potent antithermal remedy in continuous fevers.—H. T. P.

*The Technical Production of Bodies rich in Nicotine.*  
Th. Schloesing, jr. Comptes rend. **118**, 1053—1055.

THE process hitherto employed for preparing these extracts has consisted in evaporating an aqueous extract as far as possible, but due regard has not been paid to the amount of nicotine contained, which is the essential principle. Extracts of tobacco are largely used as a preservative against the ravages of certain plant insects, but by far the largest use is as a sheep-wash in cases of scab. The Argentine Republic imported in one year more than 3,000,000 kilos, for this purpose. With a view of obtaining nicotine extracts of known strength, the aqueous tobacco extracts are distilled with steam in a column apparatus. The resulting solution has approximately the same nicotine strength as the original extract, but is to be preferred to it as it is nearly colourless. Moreover, by passing the steam charged with nicotine from the exit of the column through a tower down which a slow stream of sulphuric acid is allowed to fall, the nicotine is absorbed and passes on to the tower in solution as nicotine sulphate. It is not advisable to work to a higher strength than to 200 grms. of nicotine per litre on account of the poisonous nature of the alkaloid. The liquor thus obtained contains a small quantity of ammonium sulphate, and the solution coming from the tower ought to be neutral or slightly alkaline. If acid it must be neutralised with sodium carbonate and is then coloured with tobacco extract and diluted with water until it contains about 100 grms. of nicotine per litre. This solution diluted with 80 times its volume of water kills the scab mite, or diluted with 50 times its volume of water it kills the eggs of this insect, whilst solutions containing 0.01 per cent. of nicotine are of a convenient strength for horticultural purposes. The method proposed by the author renders it possible to prepare, on a manufacturing scale, colourless nicotine solutions of known strength, which may be used for medicinal purposes. —T. A. L.

*The Qualitative Composition of Official Beech and Oak-wood Creosotes.* A. Richal and E. Choay. Comptes rend. **118**, 1894, 1332—1342.

"OFFICIAL creosote" is designated as that fraction of wood-tar which distils over between 200° and 220° C., this range being adopted because it comprises the extreme limits laid down in the pharmacopœias of various countries. Specimens of creosote answering to this requirement were prepared by the distillation of beech and oak "heavy oils" and were subsequently exhaustively examined. It was found that qualitatively the two creosotes were identical. The following phenolic bodies were definitely recognised:—Phenol, *o*-, *m*-, and *p*-cresol, *o*-cetylphenol, two *m*-xyleneols (1:3:4 and 1:3:5), guaiacol, rosol, and ethylguaiacol,



Besides these, traces of sulphur derivatives, probably thiophenols, were detected, as well as a substance resembling, but not identical with, Reichenbach's "pittacal."—H. T. P.

*The Stability of Aqueous Solutions of Mercuric Chloride.*  
E. Bureker. Comptes rend. **118**, 1894, 1345—1347.

THE following observations were made:—

1. When mercuric chloride is dissolved in ordinary service water, a portion of the salt is immediately decomposed; but under the combined influence of air, light, and the mineral and organic substances present in the water, or derived from the atmosphere, decomposition goes on indefinitely, resulting in the formation of a brownish, crystalline precipitate which contains mercury, chlorine, ammonia, and organic matter.

2. Further decomposition is, however, almost entirely arrested when the solution is completely protected from contact with air and light.

3. Solutions of mercuric chloride in distilled water suffer only an insignificant change, even when freely exposed to the air and light.—H. T. P.

*Stability of Dilute Solutions of Corrosive Sublimate.*  
L. Vignon. Comptes rend. **118**, 1099—1101.

THE alterations which dilute solutions of corrosive sublimate undergo on keeping (see this Journal, 1894, 171) are due principally to the presence of alkaline substances, which may either be derived from the water with which the solutions have been prepared, from the air, or from the glass vessels containing the solutions. It is also to be remembered that dust or organic substances with which the solutions may come into contact bring about a certain amount of reduction.—A. R. L.

*Physical Properties of Pure Nitrous Oxide.* P. Villard. Comptes rend. **118**, 1096—1099.

NITROUS oxide after treatment with appropriate absorbents still contains air and nitrogen. It may be freed from these impurities by the preparation, from the impure gas, of the hydrate of nitrous oxide (Comptes rend. **118**, 646), which, although scarcely decomposed below 0° C. under the ordinary pressure, furnishes about 200 times its volume of gas when warmed above this temperature. A more rapid process of purification consists in rectifying the nitrous oxide contained in a gasometer, or, better still, the liquefied gas in an iron cylinder. To this end the gas is passed through appropriate reagents contained in a long tube of resistance glass or of ordinary glass incased with copper, whence it issues deprived of those impurities which are capable of being absorbed. It is then led into a glass tube, where it is liquefied by cooling. The pressure rises progressively, showing that the superincumbent atmosphere is rich in nitrogen; this is allowed to escape from time to time, and the release of pressure causes the liquid to boil, thereby removing from it the major portion of dissolved gas. After this the tube is reversed, and the liquid is passed into a recipient tube; the first portions, which serve to wash out this tube, are rejected. A prolonged ebullition effects the purification, and the tube is closed. The whole operation is carried out in a gas-tight apparatus without joints; the cocks used were of metal. About 20 grms. of pure nitrous oxide may thus be prepared in one operation.

From experiments described in the paper, the author deduces the critical temperature of nitrous oxide as 38°·8 C., a value notably higher than any hitherto recorded (35°·4 Dewar, 36°·4 Jansen). The volume and density are then 0·00436 and 0·454, and the critical pressure 77·5 atmospheres.—A. R. L.

## PATENTS.

*An Improved Process for the Extraction and Estimation of Eucalyptol.* L. B. Scammell, Adelaide. Eng. Pat. 14,138, July 23, 1894.

See under XXIII., page 1106.

*Investigation of Certain Commercial Forms of Artificial Musk.* Bericht von Schimmel and Co. October 1893. Zeits. anal. Chem. **33**, 487.

See under XXIII., page 1105.

*Estimation of the Total Alkaloids in Cinchona Bark.* C. C. Keller. Apoth. Zeit. **8**, 542; Zeits. anal. Chem. 1894, **33**, 490.

See under XXIII., page 1104.

*Estimation of the Alkaloids in the Strychnos Nux Vomica Seeds.* C. C. Keller. Apoth. Zeits. **8**, 542; Zeits. anal. Chem. 1894, **33**, 491.

See under XXIII., page 1105.

*Examination of Chloroform for Phosgene.* Zeits. anal. Chem. 1894, 33, 4-8.

See under XXIII., page 1099.

*Solutions of Sweet Carbanides in Oils, Fats, Waxes, Resins, and Process of Making the Same.* A. Sommer. Cambridge, U.S.A. Eng. Pat. 15,468, August 14, 1894.

See under XII., page 1071.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*The Photographic Properties of Vanadium Salts.* A. and L. Lumière. Monit. Scient. 42, June 1894, 427.

From the position of vanadium in the series of metals, and its general properties, the authors concluded that its salts should be acted on by light. This they have verified as a fact, and give the results they have obtained, though both the cost of vanadium compounds and the feebleness of their photographic properties are likely to prevent their practical employment.

On reducing a solution of vanadic acid in sulphuric acid by zinc, the red colour first formed becomes blue, green, and lastly violet. This violet hypovanadious sulphate is an energetic developer, working even in a strongly acid solution.

Vanadic salts, obtained usually in unstable solution, by acting on vanadic anhydride with acids, are sensitive to light. The chloride, phosphate, and potassium vanadic tartrate have given the best results. The chloride is obtained by acting in the cold on vanadic anhydride with strong hydrochloric acid, and the tendency to dissociation is lessened by adding alcohol. With this a sensitive film can be prepared, giving after short exposure a faint picture which can be fixed and intensified by aromatic amines, as the authors have shown in the case of manganic salts. The phosphate, made by the action of phosphoric acid on vanadic anhydride at 150° C., is more stable than the chloride, but less sensitive to light. The potassium tartrate, easily made by shaking vanadic anhydride in solution of cream of tartar, gives films which are very sensitive and yet can be kept some weeks without change. The authors have tried to increase the differences between the vanadic salts and their reduction products, and thus add to the intensity of the pictures, but hitherto without success.—J. T. D.

### PATENTS.

*A New or Improved Process for Separating Photographic Gelatine Films from Celluloid Supports or Bases.* A. A. Barratt, Esher, and A. J. E. Hill, Surbiton. Eng. Pat. 19,810, October 20, 1893.

This process consists in soaking the celluloid negative in a solution of one part of amyl acetate or acetone in six parts of water, which penetrates the gelatin film and attacks the celluloid support, thereby loosening the film, which may then be stripped or floated off on to a glass plate and dried. If "enlargement" of the film be desired, an acid, preferably citric, tartaric, or acetic, may be mixed with the above solution, or, the stripped film may be treated with the acid in a second operation, suitable chemicals being employed to preserve the density of the image.—J. C. C.

*Improvements in or relating to Photography in Colours.* J. Joly, Dublin. Eng. Pat. 14,161, July 23, 1894.

The object of this invention is to obtain, by means of a single exposure, a representation of an object in its natural colours, or in colours appearing to be so to the eye.

A film sensitized for the red rays is exposed beneath a parti-coloured screen bearing lines ruled in transparent pigments: these lines are of three tints: Chrysoidine orange for the red-selecting line, Ethyl green and Chrysoidine orange for the green-selecting line, and Soluble blue for the blue-violet selecting line, being mentioned as suitable colours. The lines are about 200 or more to the inch, and the tints are in regular order. From this negative after development a positive transparency is printed by contact, and on this, if viewed through a screen bearing lines ruled of the same dimensions, but of the three primary colours, red, green, and blue-violet, the image is seen in its natural colours. Many modifications of this process are described, such as the various methods of ruling the lines, or having a pattern in place of lines on the screen, also modifications in the pigments used, the method of ruling, &c.—J. C. C.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*Smokeless Powder.* Annual Report of the Chief of Ordnance for 1893. Washington, U.S.A. 24—23; 245—265; 276—278; 303—306; 395—405.

During the year ending June 30, 1893, the following smokeless powders for small arms have been experimented with:—Peyton, Leonard, Du Pont, Axtell, Alters (all of American manufacture), Wetteren (Belgian, made in 1891), Troisdorf (German), B. N. F. (French), and Rifleite, S. R. and S. V. (English).

The best results were obtained with the Peyton and Leonard powders, but the number of experiments has not, so far, been sufficiently great to decide which of the two is the better one. It would appear, however, that the Peyton powder gives somewhat lower pressures for the same velocities, and is less affected by heat and moisture, whilst the Leonard powder with very reasonable pressures gives much greater uniformity of velocity.

The following tables give the results obtained in a 0.30-cal. rifle under ordinary conditions, and after the powders had been exposed to heat and moisture. The exposure to moisture was effected on a screen over water, in a closed receptacle, for 24 hours. The exposure to heat was for 24 hours at a temperature of 120° F.

The S.R. powder was tested in a 0.45-cal. rifle and in a 0.45-cal. carbine: in the latter it gave satisfactory results, but in the former it was inferior to ordinary black powder. S.V., a revolver powder, gave good results. The results from the Axtell and the Alters powders were quite unsatisfactory.

The recoil when using smokeless powders was found to be less, as was anticipated, than with black powder. This must not be attributed to the more progressive action of the former, but to the lighter charge required to give the same velocity.

The smokeless cannon powders tested were the French B.N. powder, the German smokeless, U.S. Navy smokeless, Cordite, Troisdorf, and Leonard. Details of the tests are given, but as they were not all conducted in the same size and type of gun, a direct comparison is not possible.

The Leonard powder is stated to have given remarkably good results. In a 5" gun in which the length of travel of the shot was 34.3 calibres and its weight 63 lb., a 21-lb. charge of Leonard powders gave a muzzle velocity of 2,874 ft.-sec., with a chamber pressure of 46,000 lb. per sq. in., which is claimed to be the best result yet obtained. An account of the general principles on which smokeless powders are manufactured is given. The measurements of velocity were made with a Boullengé chronograph, and of pressures with Rodman cutter gauges in large guns, and by crusher gauges in a special "pressure-rifle," which is illustrated, for small arm powders. The laboratory for the chemical examination of powders is described and illustrated.

## TESTS OF SMOKELESS POWDERS IN THE 0.30 CAL. RIFLE.

Powder.	Karl.	Weight.	Bullet Weight.	Rounds Fired.	Velocity.		Pressure per Square Inch.
					Mean at 33 Feet from Muzzle.	Mean Variation.	
Leonard, N.		Grains. 32	Grains. 220	No. 4	Ft. Secs. 1,991	22.0	Lb. 31,000
"		33	"	5	2,057	10.2	32,000
Peyton		37	"	6	2,013	18.5	31,500
Leonard, J.		31	"	6	2,019	15.8	33,500
Troisdorf		55	"	7	1,953	12.6	31,000
"		36	"	10	2,017	16.0	30,000
Wetteren		37	"	9	1,962	19.6	38,500
B.N.F.		34	"	12	1,931	10.5	47,000
Du Pont		35	"	5	1,964	9.0	54,000
Peyton		36	230	12	1,949	16.0	34,000
Wetteren		37	"	9	1,967	10.7	42,000
Rifleite		39	"	9	1,969	21.6	45,500

## EFFECT OF EXPOSURE TO HEAT AND MOISTURE.

Powder.	Exposed to Moisture.			Exposed to Heat.		
	Increase in Weight.	Loss in Velocity.	Pressure per Square Inch Reduced.	Loss in Weight.	Gain in Velocity.	Pressure per Square Inch Increased.
	Per Cent.	Ft.-Secs.	Lbs. Normal	Per Cent.	Ft.-Secs.	Lb.
Peyton	0.238	48		0.61	2	3,500
Leonard, J.	2.97	100	5,000	0.246	Normal	5,000
" N.	3.18	100	5,000	1.690	50	6,000
Troisdorf	1.64	110	6,500	1.290	50	10,000
Wetteren	2.01	230	"	1.590	61	"

The method of analysis devised by Captain Hoss of the Austrian army has been used, but it is said to be unsatisfactory.

It is stated that, notwithstanding the fact that the presence of nitro-glycerol is at present looked upon as a drawback, nothing against its use has as yet definitely shown itself. The following analyses, by Professor Monroe, are given amongst others.

## "Rifleite":—

Nitro-cotton	22.18
Gun-cotton	74.16
Phenyl amidazo-benzene	2.52
Graphite	Trace
Volatile	0.84
	100.00

## S.R. Powder:—

Nitro-cotton	28.18
Gun-cotton	46.97
Aurin (alkaline)	1.06
Barium nitrate	19.97
Potassium nitrate	2.35
Volatile	1.15
	99.98

## German Smokeless Powder:—

Nitro-cotton	48.83
Gun-cotton	7.45
Nitro-glycerol	43.15
Graphite	Trace
Volatile	0.53
	99.96

—R. B. P.

The Agglomeration of Explosive Substances. P. Vieille.  
Comptes rend. 118, 1894, 912–915.

THE dry agglomeration of black powders in a state of fine sub-division under increasing pressures in steel moulds, shows that there are four periods corresponding to different degrees of compression. *First*, under feeble pressure, the various grains still remain practically surrounded with interstitial spaces, which ensure the rapid ignition of the component particles; so that the rapidity of combustion is independent of the thickness of the mass. *Secondly*, the individual grains are partly united into groups, under higher pressures, but these groups, instead of the grains themselves, are separated by minute spaces, and a species of large grain cellular structure is obtained; and although the rapidity of combustion decreases with increasing density, it is still independent of the thickness of the agglomerate. *Thirdly*, under still higher pressures, the cavities are more nearly obliterated, and the groups of granules more nearly approximate to the size of the moulded grain; the rapidity of combustion still varies with density, but the influence of thickness is more and more clearly seen. *Fourthly*, under the highest compression, the time of combustion of the compact material is practically proportionate to thickness, and almost independent of density. The rapidity of combustion was found by firing the powders in closed vessels, in such a manner that the gas pressure finally increased to an extent equal to that obtaining in a gun at the time of discharge (see this Journal, 1894, 659).

In the following table are given the mean rapidities of combustion of different powders fired under pressures increasing from 100 to 2,500 kilos. per sq. cm. All samples, excepting the colloidal powders, were primarily powdered, sifted through silk gauze, and then agglomerated undre

pressure sufficient to insure combustion by parallel surfaces (fourth period of compression). The charges were made up

of plates geometrically similar, but of different thicknesses:—

#### AGGLOMERATION OF EXPLOSIVES.

Type.	Explosive. Class.				Density of Compressed Substance.	Rapidity of Combustion in C.m. per Second.
		KNO <sub>3</sub>	S.	C.		
Black or brown powders.	Mining powder .....	62	20	18	> 1.900	5.09
	French brown powder .....	78	3	19	"	1.45
	German brown powder (R.W.P.) .....	78	3	19	"	5.70
	French black powder ( <sup>26</sup> / <sub>31</sub> ) .....	75	10	15	"	8.50
	French sporting powder .....	78	10	12	"	9.48
	Sporting powder, very finely triturated ....	78	10	12	"	13.74
Colloidal powders.	German pure cotton powder .....				circa 1.600	5.80
	English ecobite (nitro-glycerin base) .....				" "	20.40
	French B.N., commercial, with nitrates .....				" "	7.58
	Nobel's ballistites .....				" "	20-40*
Various .....	Nitro-mannite .....				1.765	23.3
	Hexanitratèd diphenylamine .....				1.620	6.19
	Pieric acid .....				1.708	5.32

\* Varying with the sample employed.

Hence it is seen that in regard to rapidity of combustion, black or brown powders are similar to the colloidal cotton powders, whether pure or nitrated, but are greatly inferior to the nitro-glycerin colloids. The fact that to obtain equal ballistic results colloidal powders are substituted for service black powders of much larger grain, shows that these latter do not behave like the compact bodies of the fourth class above alluded to, but is another evidence of the disaggregation described in the previous paper (*loc. cit.*). It is also clear that among black powders of the same type the rapidity of combustion varies with the degree of trituration of the ingredients, and with the nature of the charcoal. The latter circumstance agrees with the slower action of the service powder containing lightly carbonised wood instead of charcoal.—W. G. M.

#### PATENTS.

*Improvements in Explosives.* K. C. Edmunds, Calcutta, India. Eng. Pat. 23,416, December 5, 1893.

THE inventor claims the use of mixtures of potassium nitrate, pieric acid, sulphur, Prussian blue, sawdust, or nitrated wood pulp, potassium chlorate and chloride, and silica in various proportions.—W. M.

*Improvements in Matches and Striking Surfaces for Use in connection therewith.* W. Herz, Vienna, Austria. Eng. Pat. 14,979, July 21, 1894.

THIS is an invention for "matches free from poison, and without heads, manufactured by treating splints of wood having rounded ends with a solution of phosphate of ammonia and alum, subsequently drying them, and then dipping their two ends in an ignition mass, of a gelatinous consistency, composed of a solution of chlorate of sodium, sulphate of alumina, sulphate of potassium, hyposulphite of soda, and saltpetre, to which is added tragacanth, gum arabic, and dextrin, again drying them, and then coating them with a solution of india-rubber, benzine, camphor, and

paraffin, and once more drying them," and "a friction surface for igniting matches forming the subject of the first claim, such friction surface consisting of a mixture of amorphous phosphorus with water, fish glue (syndeticon), sulphide of antimony, chalk, and manganese, which are well triturated together, and then painted upon the box, substantially as described."—W. M.

*Explosive Compounds.* G. J. Buechert, San Francisco, U.S.A. Eng. Pat. 15,887, August 21, 1894.

THIS invention relates to a compound consisting of ammonium chloride or sulphate, sodium nitrate, wood pulp, and nitro-glycerin. To prevent the ammonium salt from being in contact with the sodium nitrate and being transformed into ammonium nitrate, its particles are coated with some protective substance, such as aluminium oleate.

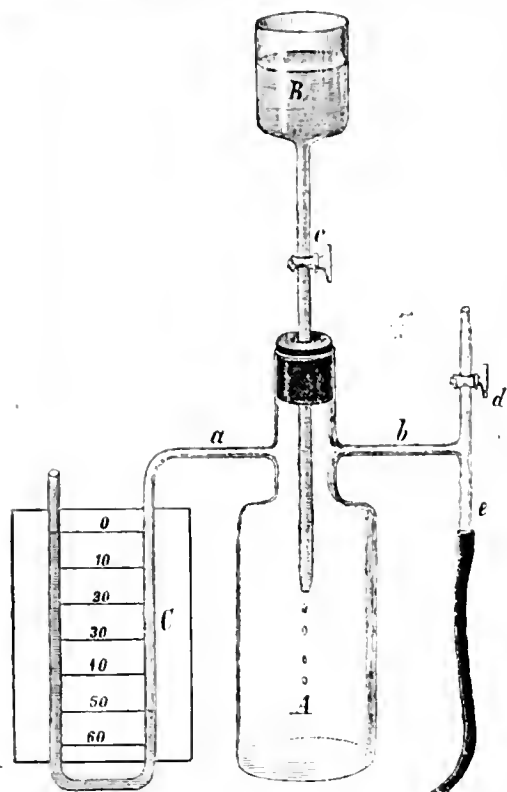
—W. M.

### XXIII.—ANALYTICAL CHEMISTRY.

#### APPARATUS, ETC.

*Apparatus for Obtaining a Constant 50-mm. Water Pressure during the Estimation of the Freezing Point of Oils.* F. Ganter. Zeits. anal. Chem. **33**, 1894, 343-344.

THE method of using this apparatus is apparent from the figure: the tube containing the oil is attached to the apparatus by the india-rubber tube *c*. It is advisable to surround the flask with felt to prevent the influence of draughts, &c. By means of this apparatus any required pressure may be readily maintained.



—L. T. T.

## PATENTS.

*An Apparatus for Notifying the presence in the Air, of Gases such as Fire-Damp.* E. Hardy, Dreux, France. Eng. Pat. 15,329, August 11, 1893.

THE principle of this invention is based upon the fact that the musical sound emitted by an organ-pipe, varies as the density of the vibrating air or gas contained therein. Two similar organ-pipes are employed, which are in perfect unison when sounded with the same gas or air, and arranged so that one is always blown with pure air, while the other receives the mixture of air and gases to be tested. According to the extent the air is mixed with other gas, so the density varies, and the sound emitted by the pipe supplied by the mixture differs proportionally from that fed with pure air. When the two pipes are sounded together, one with air and the other with the mixed gases, a greater or less number of musical beats are produced in a given time, which may be graphically indicated by the ordinary acoustical methods. The construction of the apparatus and method of working are such as to adapt themselves to the utilisation of the above principle for the purpose above set forth. The apparatus described may be used for testing the gases of flues, as well as those of mines.—S. P. E.

*Improvements in Apparatus specially suitable for Use in Estimating the Amount of Fat in Milk and other Substances.* R. W. Woosnam and the Dairy Supply Co., Lim., London. Eng. Pat. 23,039, November 30, 1893.

THE apparatus is designed to be used by unskilled persons. It consists of a two-necked bottle, containing sulphuric acid or hydrochloric acid dissolved in amyl alcohol, to which is attached a small burette and an india-rubber ball. By pressing the latter a definite volume of liquid is filled into the burette by an automatic siphon arrangement. This

volume of acid is then added to the milk to be tested, which is contained in a small bottle, into the neck of which a divided glass tube can be fitted by means of a ground joint. In the shoulder of this bottle a small perforation is made. The bottle being charged, is placed in a metal vessel containing hot water and caused to rotate in a centrifugal machine, the hot water enters by the above mentioned small perforation, and the specifically lighter fat collects in the divided tube.—T. E.

## INORGANIC CHEMISTRY.—QUALITATIVE.

*Detection of Hydrochloric Acid.* A. Villiers and M. Fayolle. Comptes rend. 118, 1152—1154.

THE method proposed by the authors is based on the difference of the action of halogens on an acid solution of aniline. Iodine does not produce an apparent reaction, or at all events gives a colourless soluble product; bromine forms white, insoluble substitution derivatives, and chlorine gives coloured oxidation compounds—black and insoluble if the quantity is large; but if it is small, reddish-violet in the hot, and blue, becoming red after some time, in the cold. The solution must be acid, but should not contain a large quantity of mineral acid or the reaction will fail to take place. The following solution is recommended as one which can be kept for an indefinite period in yellow glass vessels without becoming coloured:—Saturated aqueous solution of aniline (100 cc.), glacial acetic acid (100 cc.).

The liquid to be tested is brought to a volume of 10 cc. a mixture (5 cc.) of equal volumes of sulphuric acid and water, together with a saturated solution of permanganate of potassium, added. This mixture is heated, and the evolved gas is led into 3—5 cc. of the above-mentioned reagent contained in a tube kept cool by water. In the absence of bromine and iodine there is obtained with 0.0001 grm. of hydrochloric acid a bluish coloration, which slowly changes at a low temperature, rapidly at a high temperature into a rose coloration. When the amount of hydrochloric acid is augmented, there is obtained a very intense coloration or a black precipitate. When hydrobromic and hydriodic acids are present and the proportions above indicated are used, the iodine is completely oxidised and does not pass over; bromine is retained by the reagent as a precipitate; a certain quantity, however, enters into combination with the chlorine, the brominechloride considerably reducing the delicacy of the reaction. The results are shown in the following table:—

HI only .....	No reaction.
HBr .....	White precipitate colourless liquid.
0.05HI + 0.05HBr + 0.05HCl	{ Very intense blue liquid, the { rich violet; black precipitate.
" " + 0.01HCl	{ Liquid and precipitate blue, { then intense rose.
" " + 0.003HCl	Bluish tint then clear rose.
" " + 0.001HCl	{ Appreciably tint—rose after { some time.

—A. R. L.

*Detection of Hydrochloric Acid.* A. Villiers and M. Fayolle. Comptes rend. 118, 1204—1206.

THIS paper is supplementary to the former (last abstract). To obviate the vitiating effect of bromine when this halogen is present, the acids are precipitated with silver nitrate, and the washed precipitate is digested for some hours with a solution containing ammonia diluted with 10 parts of water. This dilution of ammonia does not dissolve silver iodide, only dissolves silver bromide sparingly, but dissolves the chloride readily. The ammonia is expelled from the filtrate by boiling and an excess of a solution of hydrogen sulphide added; the filtrate is then boiled and concentrated to 10 cc., the remainder of the operation being conducted as already described.

Simple cyanides when present are decomposed by dilute sulphuric acid, the hydrogen cyanide being expelled by evaporation, but ferrocyanides and the like are distilled with

dilute sulphuric acid until fumes of sulphuric anhydride appear. Water is then added to the residue and the distillation repeated; the distillates are then united and the hydrogen cyanide expelled from them by evaporation.

When the haloid acids are not precipitated with silver nitrate as in the method described in the former paper (last abstract), ammonium salts, when present, should be eliminated, because of their action on chlorine.—A. R. L.

*Testing for Hydrobromic Acid.* A. Villiers and M. Fayelle. *Comptes rend.* 1894, **118**, 1265—1268.

The authors describe the uncertainty attending the method of testing for small quantities of bromine in the presence of much iodine, which depends upon the successive absorption by, and coloration of, carbon bisulphide in contact with the halogen compounds, when they are attacked slowly by dilute chlorine water. They then give details of a process which is applicable to the indication either of large volumes or mere traces of bromine mixed with any proportion of iodine. A solution of the haloid salts, free from nitric acid, is mixed with a sufficient excess of ferric chloride free from chlorine (it should not impart colour to carbon bisulphide in presence of alkaline bromides), the proportion being about 5 cc. of semi-normal ferric solution to 0.1 grm. of iodine. The mixture is then evaporated to dryness on a water-bath. Thus the iodine is completely liberated and sublimed; the bromides on the other hand are absolutely unattacked. A few drops of water are added to the dry residue, iron is precipitated by an alkali and filtered off, an excess of hydrochloric acid is added to the filtrate, with some carbon bisulphide, and chlorine water is slowly introduced. The coloration or non-coloration of the bisulphide is a certain indication as to the presence of bromine. For general analysis, the haloids are precipitated as silver compounds; hydrocyanic acid is separated, as described in a previous paper, dealing with chlorine determination (see previous abstract); the precipitate is treated with hydrogen sulphide; excess of the gas is boiled off; and the liquid is ready for treatment as above described. The process may be used for the detection of free bromine in presence of iodine, by previously converting the halogens into hydrogen acids by means of aqueous hydrogen sulphide.—W. G. M.

*The Detection of Traces of Chlorine.* A. Villiers and M. Fayelle. *Comptes rend.* **118**, 1894, 1413—1414.

In a previous communication (see p. 1091, "Detection of Hydrochloric Acid") the authors have described a method for the detection of chlorine in presence of bromine and iodine, based on the fact that chlorine (liberated by means of potassium permanganate and sulphuric acid) when passed into an acid solution of aniline, converts the latter into coloured oxidation products. It appears, however, that the sensitiveness of the reaction varies greatly with different samples of aniline. Pure aniline yields with traces of chlorine only a comparatively slight brownish tint; whilst with certain commercial anilines a fine blue coloration changing gradually to reddish-violet is obtained; and so intense in character that less than 0.1 mgrm. of chlorine produces a distinct effect. This high sensitiveness has been traced to the presence in commercial aniline of certain impurities, especially *o*-toluidine. It is, therefore, recommended to make up the test solution as follows:—

	Cc.
Saturated aqueous solution of aniline (colourless) . . . .	100
Saturated aqueous solution of <i>o</i> -toluidine . . . . .	20
Glacial acetic acid . . . . .	30

—H. T. P.

*A Delicate Reaction for Copper Compounds.* P. Sabatier. *Bull. Soc. Chim.* 1894, **11**—12, 683—685.

The author has shown that cupric bromide, according to conditions of hydration, or of combination with hydrobromic acid, may give a blue, green, brown, or purple colour in solutions. The present paper deals with the formation of the purple salt, the hydrobromide, in its applications as a

sensitive means of detecting the presence of copper. A drop of the solution to be tested for copper is added to about a cubic centimetre of colourless concentrated hydrobromic acid; if the solution contains 1 atom of copper in 4 litres of fluid, a brilliant purple colour develops; a drop of a solution 100 times more dilute gives a lilac colour, and the reaction is still appreciable with 1 atom of copper in 2,000 litres solution, equivalent to 1 part copper in 30,000 of water. The amount of copper thus revealed in the drop of liquid tested, did not exceed 0.015 mgrm. If free bromine be present in the test solution it must be expelled by boiling.

Concentrated hydrobromic acid may be replaced by a saturated solution of orthophosphoric acid containing crystals of potassium bromide. A drop of the cupric solution is added, and on cooling, after heating to near 100° C., the coloration appears. This process answers with insoluble copper salts, as the sulphide, phosphate, and silicate. If the quantity of copper be very small, a little bromine is added, and the excess expelled by ebullition. The author states that the reaction is more delicate than with other quick methods, citing especially the reactions with potassium ferrocyanide and hydrogen sulphide as inferior in delicacy. Small proportions of gold, nickel, or cobalt in the solution to be tested for copper do not interfere. The reverse is the case if much iron be present, in which case the solution is electrolysed, so that any copper may be deposited on a piece of platinum foil, which is then moistened with a drop of nitric acid, then with sulphuric acid, and, after drying, is treated with hydrobromic acid. The author discusses the bibliography of the subject, and points out that, although previous observers had noticed the same or a similar reaction, they had failed to interpret it correctly, or to indicate the requisite conditions for its success.—E. S.

## INORGANIC CHEMISTRY.— QUANTITATIVE.

*Reliability of Magnesium Pyrophosphate for Estimating Phosphoric Acid.* H. Neubauer. *Zeits. anal. Chem.* **33**, 1894, 362, 370—from *Inaug. Dissert.* Rostock, 1893.

Phosphoric acid determinations made according to the usual methods of Abesser, Jani, and Marcker, or of Peitsch, Rohn, and Wagner, almost invariably give too low results. The author has made a long and careful investigation of the whole subject, and finds that the low result is caused by the volatilisation of phosphoric acid during ignition of the magnesium phosphate. His investigations have led to the following conclusions:—

1. If the ammonium magnesium phosphate is formed in a neutral or ammoniacal solution containing no excess of magnesium salt, the precipitate contains less than its normal amount of magnesium. On ignition some metaphosphate is formed, which, on continued heating in the blowpipe flame, is converted into pyrophosphate with volatilisation and loss of some phosphoric anhydride.

2. If the precipitate is formed in the presence of an excess of magnesium salt, and during its formation an excess of ammonia is never present, the normal precipitate  $MgNH_4PO_4$  is alone formed. This on ignition yields pure magnesium pyrophosphate, and no loss of phosphoric acid occurs.

3. If the precipitate is formed in presence of an excess of magnesium, and an excess of ammonia is also present, the precipitate always contains some trimagnesium phosphate.

In the case of 1, the analytical result would be too low; of 2, correct; and of 3, too high. It is, however, impossible in practice to maintain the conditions of 2. The best results are obtained by a slight modification of the Wagner method and the use of corrections which have been carefully determined by the author.

The precipitation with molybdate and washing of the yellow precipitate is carried out as usual, but care must be taken to prevent the precipitation (by heating or too long standing) of free molybdic acid. The washed precipitate is dissolved in 100 cc. of cold 2½ per cent. ammonia and

magnesia mixture added drop by drop with stirring until about as many cc. of the mixture have been added as there are centigrams. of  $P_2O_5$  present. The addition of 10 cc. of the magnesia mixture should take not less than a minute. The precipitate is once well stirred up and then allowed to stand for three hours. In igniting the precipitate, the heat must be increased slowly, and not allowed to exceed a dull red till the last trace of carbon of the filter paper has

disappeared. The precipitate is then heated in the blow pipe till half an hour's heating causes no further loss of weight.

The corrections are given in the accompanying table, in which the abscissæ represent the weight, in grms., of pyrophosphate found, the ordinates the weight, in mgrms., of pyrophosphate (equivalent to the loss of phosphoric anhydride) to be added.

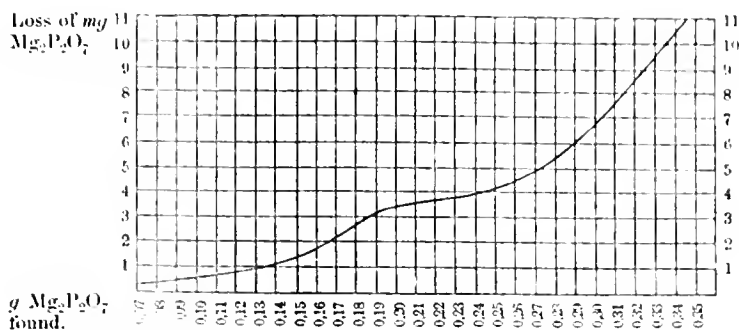


TABLE OF CORRECTIONS FOR PHOSPHORIC ACID DETERMINATIONS.

It is never advisable to attempt to estimate phosphoric acid by direct precipitation by magnesia, as the intervention of the molybdenum precipitate allows the proper conditions to be more certainly maintained.—L. T. T.

**Standard Solutions.** J. L. Buchanan. Communicated to the Glasgow City Analysts' Society. Abstracts. No. 4, 1893-4, 14.

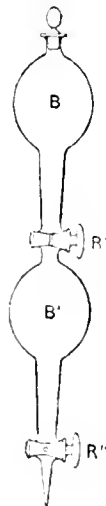
As, generally, the whole series of normal solutions used in acidimetry and alkalimetry depends upon tests made against pure sodium carbonate, the author endeavoured to prepare a normal acid solution directly, which would not require standardising, by using potassium tetroxalate which has been recommended for the purpose. The salt is very readily prepared in a pure state, and does not lose its water of crystallisation in the desiccator; but owing to its limited solubility, a stronger solution than N/5 cannot be prepared. Phenolphthalein is the only indicator available for use with it, so that altogether its usefulness is very circumscribed. Potassium bitartrate, recommended by Borotraeger, has all the disadvantages of the tetroxalate and is still less soluble. Therefore the author concludes that no substance yet proposed is a satisfactory substitute for sulphuric acid.

Sodium carbonate, purchased as pure, cannot be relied upon, and is troublesome to purify. The author recommends the use of borax instead. Pure borax is easily prepared by recrystallising the commercial salt and drying the crystals in the air until constant in weight. It is quite stable in the air, but loses water in the desiccator. The air-dried salt contains 47.12 per cent. of water (10 mols.). Simple ignition over the Bunsen flame, which expels all this water, serves to determine whether the salt is of normal composition. In standardising an acid, a suitable quantity of the salt should be weighed out, dissolved in water, tinted with methyl orange, and the standard acid added until the first tinge of red appears. Owing to the high molecular weight of the salt, it is obvious that an error in weighing the borax affects the result far less than the same error in weighing sodium carbonate.—L. A.

**The Estimation of Iodine.** A. Villiers and M. Fayolle. Comptes rend. 118, 1894, 1332-1335.

THE estimation of iodine in presence of chlorine and bromine is a somewhat difficult operation, and the various methods adopted for the purpose are all more or less faulty. The author has devised a volumetric process, which he claims to be very accurate and of which the following are

the details:—The solution to be examined, which must be free from nitric acid, is placed in a stoppered separator, carbon bisulphide added, and then 5 cc. of a N 10 solution of ferric chloride for every decigram of iodine supposed



to be present. The mixture is then gently warmed (the heat of the hand is sufficient), thoroughly shaken, and the carbon bisulphide drawn off and replaced by a fresh portion, the extraction process being repeated four times in all. The united carbon bisulphide extract is subsequently shaken in another separator with water to remove traces of ferric chloride, finally transferred to a stoppered bottle, and the dissolved iodine titrated in the usual way with a standard solution of sodium thiosulphate. A double bulbed separator, as shown in the accompanying drawing, may conveniently be employed. The extraction with carbon bisulphide is effected in the upper bulb, and the subsequent washing in the lower one. In analysis, where the three halogens are precipitated in combination with silver, the hydroacids are best regenerated by means of sulphuretted hydrogen, the excess of the latter being expelled by boiling the (dilute) solution.—H. T. P.



*The Analytical Separation of Chlorine and Bromine.*  
M. R. Engel. Comptes rend. 1894. 118, 1263—1265.

Ammonium persulphate may be made to separate the whole of the bromine from sufficiently dilute solutions of mixed bromides and chlorides without influencing the chlorine. From 1 to 2 grms. of the mixture of the alkaline haloid salts are dissolved in 150 to 200 cc. of water; from 3 to 5 grms. of the ammonium persulphate are then added, the liquid is heated to 70—80° C., and the liberated bromine is driven over by a current of air into a solution of sulphurous acid, the bromine being afterwards determined gravimetrically as silver bromide or volumetrically by titration with silver nitrate, with a potassium chromate indicator. The operation is complete in an hour, and the separation is theoretically perfect. The sulphurous acid is to be preferred to potassium iodide for the reception of the bromine, as the gases are apt to contain traces of ozone or of persulphuric acid sufficient to liberate 0.006 to 0.007 grms. of iodine in the hour. In a blank experiment with commercial sodium chloride, using silver nitrate instead of sulphurous acid, a just perceptible cloudiness of the nitrate is observed after a quarter of an hour, which may be due to hydrochloric acid or to a trace of bromine, or to a mixture of the two (not yet ascertained), but the error thus introduced cannot exceed one-tenth of a milligramme of salt in any experiment. Ammonium persulphate may be used to separate iodine from iodides in presence of chlorides and bromides, by operating in the cold with the addition of sodium acetate; the iodine is then separated by carbon bisulphide and titrated with sodium thiosulphate. At higher temperatures, some iodic acid is formed which invalidates the result.—W. G. M.

*The Estimation of Silica.* A. Cameron. Communicated to the Glasgow City Analysts' Society. Abstracts, No. 4, 1893-94, 22.

THE author, by experiments made with silica purchased as pure, but found to contain 98.5 per cent. of  $\text{SiO}_2$ , has confirmed the conclusions of previous investigators that repeated evaporations to dryness with hydrochloric acid are necessary in order to render the whole of the silica insoluble after fusion with alkaline carbonates. His experiments showed that these repeated evaporations lead to no useful result unless the silica rendered insoluble by the first evaporation be first removed by filtration; on evaporation of the filtrate more silica will be obtained, and for accurate work it is necessary to continue evaporating the successive filtrates until no appreciable quantity of insoluble matter is left on dissolving. The author found it preferable to evaporate the filtrate from the main silica with hydrochloric acid, rather than use sulphuric acid, as the results were quite as accurate, and, when iron and aluminium are present, sulphuric acid is apt to cause the formation of difficultly soluble salts. Fusion with potassium bisulphate gives too low results. (See also this Journal, 1892, 215—217.)—L. A.

*The Estimation for Industrial Purposes of Boric Acid in Boronatrocalcites.* G. A. Le Roy. Monit. Scient. 42, July 1894, 520—522.

THE author avails himself of the difference between Poirrier's Orange 3 (ammonium dimethylamidoazobenzene-sulphonate) and Poirrier's Orange 2 (sodium  $\beta$ -naphthol-azobenzene-sulphonate) as indicators; the latter is affected by boric acid, the former not. The method of procedure is as follows:—From 2.5 to 5 grms. of the powdered sample is treated, in a flask furnished with a reflux condenser, with a few cc. of sulphuric acid diluted with an equal volume of water, and a little hydrochloric acid. On cooling, the whole is thrown on a filter, the residue (silica and calcium sulphate) washed with acidulated water, the filtrate and washings neutralised with soda and boiled, to precipitate ferric oxide, alumina, &c., the filtrate and washings from this precipitate acidified with hydrochloric acid, boiled to expel carbon dioxide, and made up to a known volume. 20 cc. are placed in a beaker on white paper, beside 20 cc.

of water in a similar beaker. A 10 per cent. solution of Orange 3 is added in equal quantities to both, till the water is tinted orange-yellow. Normal soda solution is added to the acid liquor, till the cherry-red tint changes to that of the water. This gives the amount of hydrochloric acid. A second 20 cc. treated similarly, but using Orange 2, gives the total hydrochloric and boric acids, the boric acid being of course obtained by difference. The change of tint in the second case is not so sharp as in the first, but can be observed well enough when working in a good light and with the water-comparison vessel. The soda solution must be quite free from carbonate, silicate, or aluminate, and cannot be substituted by potash or ammonia.—J. T. D.

*The Volatility of Alkaline Salts in Connection with the Determination of Alkalis in Fire-clays, Manures, &c., &c.* A. Cameron. A paper read before the Glasgow City Analysts' Society. Abstracts, No. 4, 1893-94, 7.

0.5 GRM. of pure sodium sulphate heated in a platinum capsule lost nothing at the highest temperature attainable with the Bunsen flame, nothing in the muffle, but 0.4 per cent. every 10 minutes in the blow-pipe flame. The same weight of pure potassium sulphate lost nothing at a dull red heat, 0.4 per cent. in one hour at the highest Bunsen flame temperature, nothing during 10 minutes in the muffle, but 1.4 per cent. every 10 minutes over the blow pipe. A mixture of 0.2 grm. of each of the pure salts lost 0.25 per cent. in one hour at a bright Bunsen flame temperature, and 1.25 per cent. every 10 minutes in the blow-pipe flame, but after 40 minutes the loss ceased. This result was confirmed by a fresh experiment.

These results, obtained with the pure salts, led the author to experiment further with mixtures, such as are obtained in the estimation of alkaline salts in manures, &c. The influence of ammonium sulphate was determined by heating a mixture of 0.2 grm. each of pure sodium and potassium sulphates and 1 grm. of ammonium sulphate at a dull red heat till the weight was constant, which took one hour. The loss of alkaline sulphates amounted to 2.2 per cent. Considering the prolonged heating here employed, and the small amounts of alkaline salts usually found, this result is regarded as showing that ammonium sulphate does not injuriously affect the accuracy of the results. The influence of organic matter was ascertained by further experiments in which the ammonium sulphate was replaced by 1 grm. of gelatin, when the loss observed was 0.75 per cent., and by 1 grm. of sugar, when the losses in two experiments were 1.5 and 1.75 per cent. respectively.

The process in which the alkaline chlorides are evaporated with oxalic acid, ignited, and weighed as carbonates, is almost as accurate as the sulphating method. 0.2 grm. of each of the pure chlorides, evaporated and ignited with 1 to 2 grms. of oxalic acid, showed a loss, in three experiments, of 0.5, 1.25, and 1.75 per cent. during 10 minutes' ignition.

The method of estimating alkalis in fireclays by evaporating the solution of the chlorides and igniting without sulphating, as recommended in Thorpe's Dictionary, is said to be quite unreliable. 0.2 grm. each of pure sodium and potassium chlorides heated to dull redness lost 6.37 per cent. every 10 minutes during one hour's heating.

The three methods were tried with a sample of artificial manure, the experiments being made in duplicate. There were obtained—

	Na <sub>2</sub> O per Cent.	
	A.	B.
By the sulphating method .....	3.86	3.86
By the carbonating method .....	3.31	3.31
By the chloride method .....	3.16	3.18

Continued heating caused a steady loss in the chloride method. The author does not account for the discrepancy

between the other two methods, but recommends the first, since with it, precautions as to heating need not be taken, and none of the usually occurring substances affect the accuracy of the results.—L. A.

*The Influence of Various Substances on the Estimation of Potash by Tatlock's Process.* H. Dods. A Paper read before the Glasgow City Analysts' Society. Abstracts, No. 4, 1893-94, 12.

SOME years ago (1877) a committee appointed by the British Association recommended Tatlock's process as the best all-round method for estimating potash. (See Chem. News, 1877, 36.) Its characteristic feature consists in washing the chloroplatinate precipitate first with platinic chloride solution (containing 7 grms. Pt per 100 cc.), then with alcohol of about 85 per cent. strength, drying at 100° C. and weighing. For the following experiments pure salts were prepared, except in the case of calcium sulphate, the commercial quality of which was used.

**Potassium Chloride.**—A standard solution of the pure salt was made, which, by the process, gave a yield equal to 100.5 per cent. of KCl, a result which was repeatedly confirmed.

**Potassium and Sodium Chlorides.**—In presence of 5 per cent. of NaCl the result was 99.85 per cent. of the KCl taken, while with 50 per cent. of NaCl the result was only 99.40 per cent. These results confirm those of the B. A. Committee, who suggested that the  $K_2PtCl_6$  precipitate is soluble in a solution of sodium chloroplatinate.

**Potassium Sulphate.**—The results obtained were very low, an average of only 99.13 per cent. being obtained. The author doubts the purity of the salt used.

**Potassium Bichromate.**—The results of two experiments were 99.16 per cent., which the author regards as satisfactory for this class of salt.

**Potash Alum.**—A mixture of 0.183 grm.  $K_2SO_4$  and 0.73 grm.  $Al_2(SO_4)_3$  gave 99.81 per cent. of the "calculated percentage of potash," but great care is necessary to wash out all the aluminium sulphate with the platinic chloride solution, before washing with alcohol. (See Chem. News, 1877, 36, 47—48; also see whole Report.)

**Calcium Phosphate.**—It is frequently requisite to estimate potash in presence of calcium phosphate; as, for instance, in the analysis of vinegar. A mixture of 50 per cent. of pure precipitated and ignited  $Ca_3P_2O_8$  and 50 per cent. of KCl, was dissolved in excess of hydrochloric acid and evaporated with platinic chloride. The results of two experiments were exactly 99.98 per cent. of the KCl taken.

**Calcium Sulphate.**—The author's experiments gave 121.8 and 130.5 per cent. of the  $K_2SO_4$  taken, showing that the  $CaSO_4$  had not been washed out, although in one experiment, additional hydrochloric acid was used for the purpose. The author is making further experiments.—L. A.

*Estimation of Nitrates and Nitrites in Waters.* J. Fraser. Communicated to the Glasgow City Analysts' Society. Abstracts, No. 4, 1893-94, 17.

In searching for a rapid and accurate method, the author tried Harrow's process (this Journal, 1891, 727), but in numerous experiments such results were obtained as finding nitrates and nitrites when none were present, besides variations in duplicate tests, showing the process to be unreliable. Horsley's process, in which pyrogallol, sulphuric acid, and common salt are used, which has been recommended as extremely delicate, was also found to indicate nitrates and nitrites strongly when none were present. In using the nitrometer, it has been suspected that oxides of nitrogen may be lost during evaporation of the water owing to the reduction of nitrates by organic matter. The author has proved that this does occur, by evaporating a solution of potassium nitrate, yielding 5.5 cc. of NO when treated directly in the nitrometer, with 500 cc. of a water containing 3 grains per gallon of oxidisable organic matter, and on treating the concentrated solution in the nitrometer only 3.1 cc. of gas were obtained. When 1 cc. of normal caustic

soda was first added to the water before evaporation, 4.2 cc. of gas were obtained. An attempt to oxidise the organic matter before evaporation with neutral potassium permanganate was not entirely successful, but acid permanganate completely solved the difficulty. The author recommends the following procedure:—To 250 cc. or 500 cc. of the water add 10 cc. of normal sulphuric acid, and then enough permanganate to render the liquid pink. Heat for about 1 hour, adding more permanganate if required. Now add rather more than 10 cc. of normal caustic soda, and evaporate to a small volume. Filter off the precipitated manganese oxide and transfer the concentrated filtrate to the nitrometer. Three tests by this method with known quantities of potassium nitrate gave exactly correct results in each case.—L. A.

*Analysis of Pyrites with Sodium Peroxide as Oxidising Agent.* C. Glaser. Chem. Zeit. 18, 1894, 1448.

THE author does not find it so easy as Hoeftel anticipated it would be, to substitute with success the sodium peroxide process proposed by Hoeftel, for the older fusion method with carbonate and nitrate, and so to avoid the repeated evaporations with hydrochloric acid necessary for the expulsion of nitric acid.

A pyrites containing 41.12 per cent. of sulphur, by Hoeftel's method only indicated 38.37 per cent. On acidifying the filtrate from the lixiviated fused mass, it was found that some sulphur had separated, and an odour was developed which indicated the presence of decomposition products of hyposulphurous acid.

In subsequent experiments more peroxide was consequently taken, and at last the proportion was adopted of 5 grms. of  $Na_2O_2$  with 4 grms. of  $Na_2CO_3$  to each amount of 0.5 grm. of pyrites weighed out. In order to anticipate any danger of subsequent losses, a few cc. of brominated hydrochloric acid were added, before the more complete acidification with hydrochloric acid. The mixture was then boiled until nearly all the bromine was expelled. Precipitation with barium chloride followed. The result indicated 41.13 per cent. To avoid the destruction of the nickel crucible it is recommended that a flame as small as possible be employed, so that the mixture in the crucible merely softens and cakes together. The heat is then raised for from 15 to 20 minutes, by the use of a strong flame. Towards the close of the process complete liquid fusion must be brought about, by which an oscillatory motion of the liquid contents of the crucible is induced. If this climax be not attained, it will be subsequently found that the brown metallic oxides become black on lixiviation through re-formation of more or less iron sulphide, and on testing the residue on the filter after a separate treatment with brominated hydrochloric acid, it is found that a considerable quantity of barium sulphate is formed. When the thorough liquid fusion indicated, has been brought about however, the filtrate will be found colourless, and without the addition of brominated hydrochloric acid, there appeared merely a slight separation of sulphur, the insoluble residue proving to be quite free therefrom. The following directions must therefore be kept well in mind:—1. Gentle heating at the beginning of the fusion. 2. Towards the conclusion, heating strongly till complete liquid fusion with swinging motion is attained. 3. Oxidation by means of brominated hydrochloric acid. By observing these precautions a determination may be made in from 2 to 2½ hours.

Sodium peroxide is conveniently employed for determining the sulphur in burnt ores. One gramme of the ore is taken, 0.5 grm.  $Na_2O_2$  and 2 grms. of  $Na_2CO_3$ , and the mixture is heated in a nickel crucible for 15 minutes over a moderate flame.

The melt is easily lixiviated. Nevertheless, it is advisable before acidifying with hydrochloric acid, to add a few drops of brominated hydrochloric acid. This is followed by precipitation with barium chloride. Watson's method, strongly recommended by Lunge, is stated not to be applicable for American burnt pyrites, though yielding good results with burnt Spanish ores. This apparent enigma is probably solved in the fact of the strongly siliceous character of American pyrites.—W. S.

*Quantitative Estimation of the Impurities most commonly found in Commercially Pure or Rolled Nickel.* T. Fleitmann. *Zeits. anal. Chem.* **33**, 1894, 335—338.

THE most commonly occurring impurities are iron, copper, cobalt, zinc, and manganese. These may be easily and quickly estimated as follows:—About 5 grms. of the nickel are dissolved in nitric acid, and the excess of the latter destroyed by repeated evaporation with hydrochloric acid. Any carbon or silica is of course left on solution of the residue in water, and may be estimated if desired. By the slow and careful addition of dilute sodium carbonate and subsequent boiling, the iron (generally not more than 1 per cent.) may be precipitated without the admixture of weighable quantities of copper or nickel. The separation is assisted by the addition of a drop of acetic acid before boiling. The iron precipitate is dissolved in hydrochloric acid, precipitated by ammonia, and weighed as oxide. If any copper has been precipitated with the iron it is left in the ammoniacal filtrate, and may be precipitated in the ordinary way and added to the subsequent copper precipitate. To the filtrate from the iron a drop of hydrochloric acid is added and then a solution of sulphuretted hydrogen drop by drop as long as copper sulphide is precipitated. Care must be taken to avoid any excess of sulphuretted hydrogen, or some of the zinc or nickel may be precipitated. After filtering off the copper sulphide, sulphuretted hydrogen is passed into the cold filtrate to precipitate the zinc. The zinc precipitate is dissolved in hydrochloric acid, precipitated as carbonate, and weighed as oxide. The filtrate from the zinc is boiled to expel the sulphuretted hydrogen, neutralised, heated to 60–80° C., and a slightly alkaline solution of sodium hypochlorite slowly added. The manganese is first precipitated as brown oxide, the cobalt next as brownish-black peroxide, and the nickel last as deep black peroxide. As soon as the precipitation of nickel begins—which is also noticeable by a marked evolution of oxygen—no more hypochlorite is added, and the mixture is boiled and filtered. In this way the quantity of nickel precipitated can easily be restricted to about that of the cobalt present, and the further separation of the manganese, cobalt, and nickel may readily be effected in the usual way.

Any traces of arsenic, antimony, and tin are precipitated with the iron, traces of lead with the copper.—L. T. T.

*Note on the Estimation of Iron.* C. R. Mitchell. Communicated to the Glasgow City Analysts' Society. Abstracts, No. 4, 1893–94, 11.

THE author has examined the modification of the bichromate process, in which the ferric chloride solution is

reduced by the addition of stannous chloride in slight excess, the overplus of stannous salt being destroyed by adding excess of mercuric chloride, and the liquid then titrated as usual. He finds that the mercurous and stannic chlorides formed by the action of the mercuric chloride on the excess of stannous salt do not affect the bichromate at all, and that the results are equally correct, no matter what excess of mercuric chloride is used or at what temperature—cold, warm, or hot—the titration is performed.

—L. A.

*The Various Methods of Estimating Carbon in Iron.* Göttig. *Verhandl. Ver. zur Beförd. des Gewerbel.* October 1893.

THE author has investigated, from the technical point of view, the various methods used, proposing especially the inquiries, whether oxidation in the dry way without previous separation of the carbon gives the whole of the carbon, whether wet methods in which the carbon is not first separated are exact, and whether, when the iron has been treated with a copper salt, the metallic copper must be removed before oxidising the carbon. He gives a résumé of usual methods, grouping them into those where the carbon is not separated from the iron before oxidation, and those in which it is: each group being sub-divided into dry and wet methods. The dry methods of the first group are all combustions of the finely-divided metal with copper oxide, lead chromate, potassium chromate, potassium chlorate, or varying mixtures of these, with or without a current of oxygen; the wet methods consist in oxidation with chromic acid and sulphuric acid of various strengths (sometimes with a preliminary treatment by a copper salt in the same vessel), the carbon dioxide evolved being either weighed in potash bulbs or measured over water. The dry methods of the second group agree in converting the iron into volatile chloride by chlorine, hydrochloric acid gas, or mercuric chloride, the carbon remaining being then burnt as in the first group; in the wet methods the iron is dissolved by the action of a copper salt, iodine or bromine, moist silver chloride, or hydrochloric acid aided by a voltaic current, and the carbonaceous residue oxidised by wet or dry methods, with or without previous removal of the copper or silver. The author has tested these methods on graphitic and non-graphitic irons and steels, containing varying amounts of carbon, manganese, and silicon. His results, for graphitic irons, are given in three tables, which are again summarised in a condensed table given below:—

TABLE I.

Method.	Nature of Metal.	Total Carbon found.	Total Carbon, Actual.
Direct combustion in the dry way (ultimate analysis) .....	Grey pig. ....	2·946	3·395
Direct oxidation in wet way (Gmelin, Jüptner, Rürup, Wiborgh).....	" .....	3·326	3·395
	" .....	4·069	4·0735
Combustion in oxygen of the carbonaceous residue separated in dry way (Wöhler, Deville) .....	" .....	3·153	3·395
	" .....	3·464	4·0735
Oxidation in wet way of residue separated by copper salt, without previous separation of copper (Ulrichsen, Clausen),	" .....	3·361	3·395
As above, but copper salt previously removed (Elliot).....	" .....	3·333	3·395
Oxidation of residue got by dissolving metal in HCl with a voltaic current passing (Weyl, Binks).	" .....	3·36	3·395

The author's conclusions are:—The combustion methods give discordant results; the end of the operation is difficult to recognise, and the grains of metal are apt to become coated with oxide, which protects them from further action: this is avoidable only if extreme care be taken to regulate the heat and prevent the fusion of the oxide; the operation,

too, takes a long time. As to the wet methods of group I., those of Gmelin and Jüptner (oxidation by chromic and sulphuric acids) give low figures, unless the sulphuric acid be strong, the amount of acids plentiful, and the attack continued for a sufficient time; Rürup's method (the same oxidiser, with previous separation of carbon by a copper

salt, in the vessel) gives good results, and there is no danger of production of sulphur dioxide if the sulphuric acid be of density not higher than 1.1; Wiborgh's method (similar to Rurup's, but the carbon dioxide is measured over water instead of being weighed) does not give regular results, no doubt owing to the absorption of carbon dioxide by the water. The dry methods of group II. are inexact. Of the wet methods, that of Ullgren, modified by Claassen

(separation by copper sulphate, and oxidation of washed residue by chromic and sulphuric acids) gives good results; most of the others can be made to furnish accurate results, but demand in that case extreme care, a long time, or both.

For non-graphitic irons, the author gives four tables of results, which are again summarised in the following table:—

TABLE II.

Method.	Nature of Metal.	Carbon Found.	Carbon, Actual.
Ultimate elementary analysis .....	White pig .....	3.324	4.301
	Cast steel .....	0.988	1.075
Direct oxidation in the wet way (Gmelin, Japtner, Rurup, Wiborgh) .....	White pig .....	4.342	4.301
	" .....	4.296	..
Combustion in oxygen of the residue separated in the dry way (Wobler) ...	Bessemer steel ..	0.4715	0.474
Ultimate analysis of residue got by a copper salt, without previous separation of the copper (Langley, Parry) ..	White pig .....	4.249	4.301
As above, with previous removal of the copper (Berzelius, Sprenger, Pearse and Creath) ..	" .....	4.279	..
Oxidation in the wet way of residue got by copper salt, without previous separation of copper (Ullgren, Claassen) ..	" .....	4.308	..
As above, but with previous removal of copper (Elliot) ..	" .....	4.268	..
Solution in iodine or brominated hydrochloric acid, and combustion of carbonaceous residue (Ergert, Brandt) ..	" .....	4.304	..
	Bessemer steel ..	0.478	0.474
Solution with mercuric chloride: residue weighed before and after combustion (Boussingault) ..	White pig .....	4.355	4.301
	Thomas steel ..	3.980	4.105
	Forged iron .....	0.284	0.290
	Bessemer steel ..	0.1465	0.174
Oxidation of residue got by electrolytic decomposition (Weyl) ..	Fine steel .....	1.0865	1.098
	Cast steel .....	1.059	1.075
	White pig .....	1.275	4.301

TABLE III.

Method.	Nature of Metal.	Graphite found.	Graphite, Actual.	Remarks.
Dissolved in HCl at a gentle heat; residue washed with water, potash, alcohol, and ether; oxidised by $\text{CrO}_3$ and $\text{H}_2\text{SO}_4$ ..	Grey pig ....	3.224	3.104	Attacked for six hours.
	" .....	3.193	3.104	" " five "
	" .....	2.890	2.855	" " three "
Dissolved in boiling HCl; residue washed with warm water; oxidised by $\text{CrO}_3$ and $\text{H}_2\text{SO}_4$ ..	Grey pig ....	3.306	3.104	Attacked for one hour.
	" .....	3.030	3.104	" " two hours.
	" .....	3.135	3.104	" " three "
Dissolved in $\text{HNO}_3$ ; residue washed with potash (no alcohol or ether); oxidised by $\text{CrO}_3$ and $\text{H}_2\text{SO}_4$ ....	Grey pig ....	3.140	3.104	Attacked for five hours.
	" .....	2.944	3.104	" " one hour.
Dissolved by prolonged boiling in $\text{HNO}_3$ ; residue washed with warm water; oxidised by $\text{CrO}_3$ and $\text{H}_2\text{SO}_4$ ..	Grey pig ....	3.169	3.104	Attacked for one hour.
	" .....	3.080	3.104	" " two hours.
	" .....	2.821	2.855	" " one hour.
Dissolved in boiling dilute $\text{H}_2\text{SO}_4$ ; residue washed with boiling water; oxidised by $\text{CrO}_3$ and $\text{H}_2\text{SO}_4$ ..	Grey pig ....	3.558	3.104	Attacked for two hours.

In the case of non-graphitic irons, the author's chief conclusions are:—The combustion methods give results too low; but when the oxidising material is infusible and porous, higher figures are got than when it is a fusible body. The same remarks as made above in the case of graphitic irons apply here, too, to the wet methods of the first group, though they are here more easily carried out, and need less time; the author recommends Wiborgh's method where approximate results are sufficient, on the

ground of its ease and rapidity. The dry methods of group II. are not recommended, save for rough work; and where many samples have to be treated by them successively, large tubes must be employed to avoid choking by condensed chlorides. As to the wet methods, that of Ullgren-Claassen gives good results, though an almost imperceptible quantity of hydrocarbon gas nearly always comes away during the solution of the iron; no evolution of sulphur dioxide takes place if there is enough chromic acid present;

the absorption of carbon dioxide by sulphuric acid is quite negligible. Eggertz' iodine method gives good results in all cases; 24 hours should be given for the solution of the iodine. Weyl's electrolytic method also gives good results, provided the current is not too strong, and provided the metal is homogeneous, so that any fragment can be used as a fair sample for analysis.

In an appendix to the paper, the author deals with the determination of the graphite. His work is summarised in Table I. I. :—

He concludes that: the washing with water, potash, alcohol, and ether is shortened if the boiling with acid is sufficiently prolonged; nitric acid gives slightly lower results than hydrochloric, no doubt because it oxidises a small portion of the graphite; yet nitric acid is preferable, because, in a much shorter time than hydrochloric acid, it yields a residue free from non-graphitic carbon.—J. T. D.

*The Determination of Lead in Small Quantities.* J. A. Williamson. A paper read before the Glasgow City Analysts' Society, Abstracts, No. 4, 1893-94, 16.

As phosphoric acid is largely used instead of tartaric and citric acids in manufacturing mineral waters, it is of importance to note that in the ordinary colorimetric test free phosphoric acid seriously prevents the formation of lead sulphide, but if the free acid be neutralised by ammonia the results are accurate. The neutralisation is best effected by adding to a measured volume of the liquid some methyl orange and a dilute solution of ammonia to neutrality. The same volume of the ammonia solution is then added to another equal volume of the liquid without methyl orange, and the quantity thus neutralised is used for the test. Excess of ammonia does not interfere with the lead reaction, but if lime be present in the liquid it causes a cloudiness.

Iron has no effect on the modified test. Copper must be estimated separately and allowed for, unless excess of ammonia can be used, when the interference of copper can be prevented by adding a little potassium cyanide and tartaric acid. Hydrochloric and nitric acids injuriously affect the estimation unless previously neutralised. Free acetic acid does not affect the result.—L. A.

## ORGANIC CHEMISTRY.—QUALITATIVE.

*The Distinction between Coal Tar and Blast-Furnace Pitches.* D. G. Buchanan. A paper read before the Glasgow City Analysts' Society, Abstracts, No. 4, 1893-94, 21.

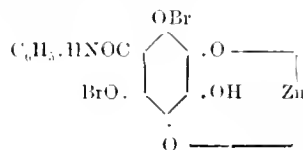
Pitch is graded commercially as "hard," "medium soft," and "soft." A common test is to place a small piece in the mouth, and after a minute or so to try to force the teeth into it; if it breaks and crumbles it is considered to be hard; if the teeth mark it with difficulty it is medium soft; if one can chew it, it is soft. The author has examined all these varieties.

The chief difference between the coal-tar and blast-furnace pitches lay in the amount of ash, which in the coal-tar pitches never reached 0.1 per cent., whilst in the blast-furnace pitches it varied from 6.8 to 11.1 per cent. As it does not seem practicable to remove the dust before collecting the tar from the blast-furnace gases, a sample containing less than 1 per cent. of ash may at once be put down as a coal-tar pitch. In cases where the amount of ash renders the denomination of a sample doubtful, an analysis of the ash should be made to see if it corresponds with that from blast-furnace pitch. Coke was estimated in the different kinds of pitch. Hard pitch yielded about 38 per cent.; medium soft, 30 per cent.; soft, 26 per cent. The yields from some samples of coal-tar and blast-furnace pitches were almost identical. The melting point or softening point was determined by warming a piece of the sample and placing it on the end of a thermometer, the latter being then fixed in a test-tube and the tube heated in a beaker of water. The pitch glazes when it begins to soften, and shortly after begins to fall—this is taken as the melting point. Hard pitch melted at about 80° C., medium soft at

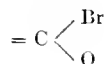
about 55° C., and soft at about 50° C. Blast-furnace pitch has a somewhat higher melting point than coal pitch. The solubility in ether and gasoline (petroleum spirit) gave no means of distinction. Ether dissolved about 77 per cent., and gasoline about 44 per cent. of soft pitch from both sources. The colour of the ether solution is rather darker in the case of the blast-furnace pitches, and this is suggested as the basis of a confirmatory test.—L. A.

*Some Blue Lakes derived from Dibromogallanilide and some Blue Reactions of the Polyphenols.* P. Cazeneuve. Comptes rend., 118, 1046—1048.

On carefully adding ammonia, potash, or soda to a solution of dibromogallanilide an unstable blue precipitate is obtained, easily changed by excess of air or of the base, becoming green and then yellow. Lime or baryta water give a white precipitate, which rapidly changes on shaking with air to a beautiful blue, which is stable below the surface of the water. The addition of a weak acid, even a current of CO<sub>2</sub>, is sufficient to decompose the lake and liberate the free acid which is of a reddish colour, and thus in its turn is destroyed by an excess of an organic acid. These calcium and barium lakes are so unstable in air that they cannot be filtered off, but it has been possible to prepare and analyse the corresponding zinc lake, the constitution of which has an important bearing on the blue products derived from gallic acid, dibromogallie acid, methyl dibromogallate, gallanilide, pyrogallol, purpurogallin, and pyrogallolquinone. The zinc lake is prepared by adding an excess of an aqueous solution of zinc acetate to a dilute aqueous alcoholic solution of dibromogallanilide in the proportion of about 2 molecules of the former to one of the latter. The precipitate, at first white, rapidly turns blue in the air, and can be filtered off and dried in a vacuum over sulphuric acid. It has the formula—



or else that of a compound containing the two groupings—



connecting two molecules by oxygen as in the case of purpurogallin and pyrogallolquinone. The spontaneous decomposition of these lakes by oxygen, which no doubt removes the hydrogen of the phenolic group and causes further condensation, is always accompanied by the production of a bromide. In this case zinc bromide is formed. The final yellow product is in all probability a quinone or polyquinone. It is noteworthy that all the polyphenols derived from pyrogallol give blue coloured bodies. Gallic acid, dibromogallie acid and its ethers, and gallanilide give blue colouring matters with baryta or lime water, which are stable under water, that from dibromogallie acid being the least stable. Purpurogallin and pyrogallolquinone give unstable blue products with ammonia.—T. A. L.

*A Reaction of the Aldehydes. Differentiation of Aldoses and Cetroses.* A. Villiers and M. Fayolle. Bull. Soc. Chim. 1894, 11—12, 691—693.

The authors show that the recoloration of a solution of magenta decolorised by sulphurous acid is not affected by both aldehydes and acetones, but by aldehydes only. Ordinary specimens of acetones produce the coloration owing only to the presence of impurities. Urine containing acetone gives the reaction because aldehydes are almost always formed simultaneously with acetone. The authors obtain the reagent in a highly sensitive state by avoiding

the use of excess of sulphurous acid, a day being then needed to complete the gradual and exact decoloration of the magenta solution, which must be preserved in closed tubes, as a red colour appears on prolonged exposure to air.

Ordinary sugar, maltose and lactose, gives no coloration with the reagent, until after several days, when the colour develops, as under the influence of water, aldehyde-glucoses are formed, such glucoses having the property, like aldehydes, of producing an intense coloration. Glucose, invert sugar, galactose, and reduced dextrins colour the reagent, whereas pure levulose and sorbine are without action. The presence of acids, interferes with the reaction of aldehyde-sugars, so that neutral solutions must be used, and the reagent should be prepared in the manner described above.—E. S.

*Examination of Chloroform for Phosgene.* Zeits. anal. Chem. 1894, **33**, 488.

THE pharmacopœias direct for this purpose that the sample shall be shaken with water and the aqueous liquid tested for hydrochloric acid, which is one of the decomposition products of phosgene with water.

According to Scholvién (Ber. pharm. Ges., **3** 213) this method does not always yield trustworthy results. He adopts a direct test for the detection of phosgene. To carry this out a drop of amidophenetol is dissolved in benzene (the benzene must be quite dry and free from carbon bisulphide) and the suspected chloroform added to this solution. If phosgene is present, there is produced an immediate turbidity, and later crystals of diparaphenetol carbamide separate out; these are insoluble in chloroform. Instead of amidophenetol, aniline may be used; in that case diphenylcarbamide being formed.

—A. R. L.

*A New Reaction of Colchicine.* E. Barillot. Bull. Soc. Chim. **11**—**12**, 1894, 514—516.

THIS reaction, which affords a very reliable test for the detection of colchicine, morphine, and codeine, is obtained by mixing the alkaloid in the state of free base very intimately with 0.25 gm. of oxalic acid and 1 cc. of sulphuric monohydrate. This mixture is sealed up in a small glass tube, which is kept at 120° C. for one hour. Subsequently the tube is opened, the colour of the mixture observed, and excess of alcoholic soda added. By operating in this way the following results are obtained with the above-named alkaloids:—

Alkaloids.	Coloration in the Cold.	Coloration after Heating.	Observations.
Colchicine.	Golden yellow.	Dark red-dish-brown, not modified by addition of water.	On treating the aqueous solution with an alkali and reacidifying a yellow precipitate is obtained, which is soluble in chloroform, and, on addition of nitric acid of sp. gr. 1.4, is precipitated from this solution in the form of a powder at first yellow and then peach-coloured.
Morphine.	Light blue	Reddish-brown.	On addition of a large volume of water the colour sometimes changes into blue. Treating this solution with caustic potash and alcohol, then acidifying it, and shaking it with chloroform, the latter assumes a blue colour. Ether takes a purplish-red colour. On evaporating these two solvents morphine-blue remains behind.
Codeine...	Bright blue.	Greenish-blue.	Like morphine.

None of the ptomaines shows any similar reactions. With 1 mgrm. of colchicine the above reaction can be repeated ten times.—C. O. W.

*The Detection of Abrastol in Wines.* L. Briand. Comptes rend. 1894, **118**, 925—926.

TO 50 cc. of the wine add 1 cc. of pure sulphuric acid, shake well, introduce 25 grms. of lead peroxide, agitate for five minutes and filter. Shake 40 cc. of the filtrate with 1 cc. of chloroform for the space of 1 minute. In the presence of abrastol the chloroform becomes yellow in colour (sensitive to 0.01 gm. per litre), whilst with natural wines it remains colourless. On evaporating the yellow chloroform solution, a yellow crystalline residue is left, which gives a fine green colour when treated with a few drops of sulphuric acid, the latter reaction being obtainable from wines containing 0.02 gm. of abrastol per litre. (This Journal, 1894, 548.)—W. G. M.

*Testing Milks with Rennet.* R. Lozé and E. Hil-sont. Comptes rend. **118**, 1069—1071.

THE authors in their investigations on the coagulation of milk by rennet have observed that the operation takes more or less time according as circumstances, and the properties of the curd chance to vary. They have devised a practical method for analysing milk based on their results. This consists in adding a certain quantity of rennet to 100 cc. of milk at a temperature of 35° C. and noting the time required to coagulate it. Commercial rennet is usually employed of such a strength that 1 litre would coagulate 10,000 litres of pure fresh milk at 35° C. in 10 minutes. The rennet solution employed by the authors is ten times this strength, so that the addition of 1 cc. of their rennet solution to 100 cc. of normal milk causes coagulation in four minutes. The time is taken by a seconds counter and the authors have investigated the effect of various influences, such as temperature, or the presence of carbonic acid, fatty matters, solid bodies (sawdust, starch) and water, which accelerate or retard the coagulation and as a result of their experiments they arrive at the following conclusions. A good milk is coagulated by the rennet solution referred to in 3½ to 4 minutes and the curd is clear, white and homogeneous. If, however, it does not possess these properties but is cloudy and separates in clots and requires a much longer or shorter time than 4 minutes, the milk is doubtful and ought to be further examined. The addition of water or of an alkaline salt considerably retards curdling, but it must be remembered that boiling produces the same effect. Milks which rapidly coagulate either contain foreign substances or have begun to decompose and a milk which coagulates in less than two minutes should be unhesitatingly rejected either as a fool, or for technical purposes.—T. A. L.

*A Natural Unsaturated Ketone in Lemon-Grass Oil.* P. Barbier and L. Bouveault. Comptes rend. **118**, 1893.

See under XX., page 1086.

ORGANIC CHEMISTRY.—QUANTITATIVE.

*Volumetric Estimation of the Three Isomeric Cresols and also of Xylenol.* F. Keppler. Archiv. f. Hygiene. Zeits. anal. Chem. **33**, 473.

THE method employed by the author is a modification of Koppeschaar's method of estimating phenol.

Into a flask, furnished with a well-fitting stopper and of such a capacity that the volume of liquid to be mentioned fills it to about two-thirds, is introduced a mixture of 50 cc. of potassium bromide solution (5.94 grms. per litre) and 50 cc. of potassium bromate solution (1.667 grms. per litre); to this mixture 5 cc. of concentrated sulphuric acid are added. A measured volume of a solution of cresol in caustic soda lye is then added, the quantity of the latter being so adjusted that a small amount of bromine remains in the free state. The mixture is now well shaken for about 10—15 minutes, when the precipitate subsides. The liquid is then filtered through glass wool, and an

aliquot portion of the filtrate treated with potassium iodide, the free iodine being titrated with  $\frac{N}{10}$  or  $\frac{N}{100}$  thio-sulphate solution and starch in the well-known way. A calculation then gives the excess of bromine; this is deducted from the amount of bromine set free from the potassium bromide and potassium bichromate (0.24 gram. Br.), and the difference gives the amount of bromine which has entered into reaction with the cresol; the percentage of the latter may then be arrived at by the equation:— $C_6H_3(CH_3)OH + 6Br \rightarrow C_6HBr_5(CH_3)OH + 3HBr$  from which it is found that 1 gram. of bromine corresponds to 0.225 gram. cresol. These directions must be obeyed in order to obtain good results; if the liquid containing the bromo-cresol be treated with potassium iodide direct without filtering, inaccurate values are obtained on titrating the free iodine.

The estimation of xylenol is carried out in the same manner, 1 gram. of bromine corresponds with 0.2542 gram. of xylenol.—A. R. L.

#### *Analysis of Sealing Wax.* C. Mangold. Zeits. anal. Chem. 1894, 33, 477–478.

The substance (5 grms.) is boiled with alcohol (150 cc.) and the solution, after cooling, diluted with alcohol to 250 cc. After settling, 50 cc. of the clear liquid are withdrawn and the iodine number of the resinous constituents contained therein determined by Hübl's method. The portion insoluble in alcohol is filtered off, washed, dried at 110 C., and weighed. It consists of inorganic matter and resin, the latter being mostly a mixture of shellac and colophony (rosin). The iodine number may be used to calculate the relative amounts of these two constituents. The percentage of the constituents is calculated from these data by the formula—

$$i = \frac{k \cdot i_1}{100} + \frac{S \cdot i_2}{100} \text{ and } S = 100 - k,$$

whence—

$$k = 100 \cdot \frac{i - i_2}{i_1 - i_2},$$

in which  $i$  is the found iodine value,  $i_1$  the iodine number of the colophony, and  $i_2$  that of the shellac;  $k$  is the percentage of colophony and  $S$  that of the shellac.

It may be remarked that a difficulty arises when the shellac has become, as it sometimes does, partially or wholly insoluble in alcohol.—A. R. L.

#### *Studies in Oil Analysis.* H. Ballantyne. A Paper read before the Glasgow City Analysts' Society. Abstracts. No. 4, 1893–94, 6.

SIMULTANEOUS exposure to sunlight and air lowers the iodine absorption of vegetable oils, and increases the temperature reaction and specific gravity (this Journal, 1891, 32). Determination of the specific temperature reaction is preferable to the simple Mauméné test (this Journal, 1891, 233). The author finds that the Hübl reagent loses iodine far less rapidly than has been stated by Fabron: thus Fabron observed a loss of 17 to 23 per cent. of active iodine in 8 days, whereas the author observed a loss of only 5 to 8 per cent. in 38 days. Raising the temperature to 25 C. made no appreciable difference. Contrary to statements made by some, the use of starch solution in determining the end reaction was found to have no influence upon the results, whether the solution used were unfiltered or filtered, the quantity large or small, or the solution added at the beginning or near the end of the titration. Experiments also showed that the age of the iodine solution has no effect upon the results provided sufficient excess of iodine be used. The titration of a blank quantity, containing the same volume of Hübl reagent as that added to the oil, does not obviate all error due to the loss of active iodine on standing, because the blank quantity contains more free iodine, and consequently loses more on standing, than the test-quantity; hence the results are too low, the error increasing with the time of

standing. Dieterich (this Journal, 1893, 381) has shown how to partly compensate for this, but the author is endeavouring to find a more satisfactory method.—L. A.

#### *Valuation of Wool-fat.* E. v. Cochenhausen. Dingl. Polyt. Jour. 292, 1891, 51–96; 112–116.

On the basis of the well-known quantitative methods practised in fat analysis, and further with due regard to the peculiar properties of wool fat and its fatty acids, already demonstrated by Lewkowitsh (this Journal, 1892, 135), the author gives the following outline of the operations adopted by him in the valuation of wool fats:—

*Foreign substances*, insoluble in ether, are determined in the usual way.

*Water* is best found by difference, since dehydration of the free fatty acids partially takes place on drying.

*The acid value*, i.e., the number of mgrms. of KOH required to saturate the free fatty acids of 1 gram. of wool fat, is determined in the usual manner.

*The free fatty acids* are separated as described (this Journal, 1892, 136), with the modification that the neutralised wool fat is shaken with 30 per cent. alcohol, and the soap solution boiled down to dryness, dissolved in 50 per cent. alcohol (Spitz and Hönig, this Journal, 1891, 1039), and exhausted with petroleum ether. But also in this process insoluble soaps of higher fatty acids separate between the two layers as flocculent matter and must be filtered off (this Journal, 1892, 136). The molecular weight of the free fatty acids is then ascertained in the usual manner.

*Saponification Value of Wool Fat.*—From experiments published by Herbig (see page 1068), the conclusion is drawn that wool fat contains certain ethers that are easily saponified by alcoholic potash (see also Eng. Pat. 14,114, this Journal, 1892, 928), and that working under certain conditions constant numbers for these easily saponifiable ethers are obtained. The remainder of the ethers can, as is well known, only be saponified by alcoholic potash under pressure (this Journal, 1892, 137). The author, neglecting the proportion of the latter compounds, adopts as saponification value the number obtained by boiling the wool fat with half-normal alcoholic potash for one hour in a flask connected with an inverted condenser.

*The volatile fatty acids* are determined by saponifying the wool fat with half-normal alcoholic potash under the conditions just stated, and subjecting the mass, termed “decomposed wool fat”—(analogous to the term “decomposed wax” in the Hübl-Benedikt-Mangold process for the analysis of bees' wax, although the “decomposed wool fat” still contains unsaponified ethers)—to Reichert's distillation process. There is, however, this modification introduced, that the “decomposed wool fat” is distilled until 1,000 cc. have passed over. The values thus obtained are still considered too low, and a correction is made subsequently by the author.

The residue left in the distilling flask is weighed and returned as the sum of the *total non-volatile fatty acids*, of the *alcohols*, and of the *unsaponified ethers*.

Next the acid value of the “decomposed wool fat” is determined, and now the total non-volatile fatty acids may be isolated as described above for the free fatty acids. Finally the molecular weight of the total non-volatile fatty acids is determined.

From the data thus obtained the composition of the sample may be calculated. The wool fat examined by the author yielded the following results:—

	Per Cent.
Free fatty acids (hydrated) molec. weight, 292 .....	25.77
Combined fatty volatile .....	2.20
acids..... non-volatile molec. weight, 348 ...	39.74
Free and combined alcohols, plus unsaponified substance .....	33.486
Water and foreign substances .....	1.20
	102.396

The author acknowledges himself that this process is too tedious for commercial analysis; it is, however, recommended in case of dispute, or for arbitration purposes.—J. L.



*Estimation of Saccharose.* Zeits. anal. Chem. 1894, 33, 474—475.

A. BORNTRAGER (Zeits. angew. Chem. 1893, 600) has given instructions for the preparation of a solution of invert sugar for the control of Fehling's solution (see this Journal, 1893, 1063). To standardise Fehling's solution 10 cc. of the latter are diluted with 40 cc. of water, boiled, and the invert-sugar solution run in. If the liquid be boiled for two minutes, 10 cc. of Fehling's solution require exactly 10.3 cc. of 0.5 per cent. invert sugar solution.

Samelson (Zeits. angew. Chem. 1891, 267) recommends estimating the exact titre in copper of Fehling's solution, as 50 cc. of this solution will only correspond with 0.2375 gm. of sugar when it contains 0.4420 gm. of copper. Employing Fehling's solution standardised in this way, the author contends that volumetric determinations of cupric-reducing power are as accurate as gravimetric determinations.

J. Jais (Zeits. gesamt. Brauw. 1893, 349) has carried out experiments on the estimation of saccharose in mixtures of maltose, isomaltose, and dextrin, using a method similar to that of Meissl. Meissl dissolves a quantity of sugar corresponding to 10 grams. of invert sugar in 700 cc. of water, inverts with 100 cc. of  $\frac{N}{5}$  HCl for half an hour on the water-bath, and, after neutralising, makes up to 1,000 cc. He thus employs for the inversion 1.4 per cent. solutions and for the reduction 1 per cent. solutions of invert sugar. The author worked with solutions of at most 1 per cent. concentration. The duration of boiling before inversion was 4 minutes, and after inversion 2 minutes; 50 cc. of Fehling's solution diluted with 25 cc. of water were employed for every 25 cc. of invert-sugar solution. The author's results may be summarised as follows:—

1. The reducing power of mixtures of maltose, isomaltose, dextrin, and saccharose is equal to the sum of the reducing powers of the single constituents.

2. Solutions of maltose, isomaltose, and dextrin, separately or in mixtures, give no increase in reducing power when inverted by Meissl's method. Hence these sugars are unaltered under the circumstances.

3. When saccharose solution is added to the above solutions and the mixture inverted by Meissl's method, the increase in the cupric-reducing power is in accordance with the saccharose added. From this increase, moreover, the saccharose may be calculated by Wein's table (No. IV.).

4. Added quantities of saccharose may be estimated by Meissl's inversion process in unhopped worts as also in beer worts, together with the quantity of saccharose already present in them.

5. Neutralisation is not necessary for the determination of the reducing power after inversion by Meissl's method.

6. Inversion by Meissl's method in malt extract solutions more concentrated than 1 per cent. (up to 8 and 9 per cent.) with the corresponding quantity of  $\frac{N}{5}$  HCl, gave on 100 cc. of wort the same results as 1 per cent. solutions.—A. R. L.

*The Estimation of Sugar: the Sugar Content of Tanning Materials, Tanning Extracts, Tanning Liquors, and Weighted Leather.* Von Schroeder, A. Bartel, and W. Schmitz-Dumont. Dingl. Polyt. J. 1894, 293, 229—237, 252—260, 281—287, 297—304.

Of late, increased attention has been called to the function of the non-tannins as a source of organic acids in liquors used for tanning (this Journal, 1894, 650 and 964), and it has been shown that it is the sugar, or at all events the compounds capable of reducing Fehling's solution, which, by fermentation, gives rise to these acids. The weighting of heavy leather with sugar is no novelty. The object of the authors of this paper is to ascertain the normal content of Fehling-reducing compounds in leather.

Comparative experiments were made on the use of lead acetate and magnesia as precipitants for non-sugars in extracts of various tanning materials, it having been stated by Kohnstein (Gerber, 1886, 253) that the latter precipitant is the better. In each case the liquor which had been

treated with lead acetate showed a higher sugar-content by the use of Fehling's solution, than that which had been treated with magnesia.

For most technical purposes it is held that the excess of lead present in a solution which has been treated with lead acetate, does not interfere with the subsequent estimation of sugar, unless the excess be large. Experiments made to ascertain whether this is true for tanning extracts gave no very definite conclusion, but it seemed on the whole that the results were better when the excess of lead was precipitated, and the best precipitant for the purpose proved to be sodium or potassium sulphate. It was further proved that the addition of lead acetate and the subsequent removal of the lead as sulphate, either in the presence or absence of tannin, had no appreciable effect upon the percentage of sugar found; thus, when working with a solution of glucose of predetermined sugar-content, there was found as a mean of several experiments 100.5 per cent. of the sugar present.

As a further objection to the magnesia method it is urged that when this precipitant is used the cuprous oxide invariably contains magnesia, a fact which entails the dissolution of the oxide in hydrochloric acid and the precipitation of the copper as sulphide before it can be weighed.

A series of experiments was next undertaken to prove whether the sugar in a sample of leather can be completely extracted from the powdered and fat-exhausted sample by water, and whether hide powder, when employed in analysis to remove tannin, absorbs any sugar from a saccharine solution. The results show that hide fibre is not, in this sense, capable of absorbing sugar.

It is next shown that for the estimation of sugar in tanning materials a longer heating with the Fehling's solution than that recommended by Allihn is necessary, on account of the presence of unknown and slowly reducing sugars, 30 minutes having been found to give the best results. This alteration of method renders Allihn's table useless, so that another has been constructed showing the mgrms. of glucose corresponding with mgrms. of copper weighed, when the Fehling's solution is heated with the sugar solution for 30 minutes. The mean experimental values for the construction of the table are given below; the rest of the table is filled in by interpolation from these values.

		Mgrms.			
Anhydrous glucose,	248.8	223.5	198.3	173.6	148.7
Copper, weighed ...	469.1	431.3	388.5	345.5	299.5
<hr/>					
Anhydrous glucose,	123.9	99.4	57.2	24.5	9.0
Copper, weighed ...	252.4	205.2	120.0	56.2	22.0

The following procedure is adopted when it is desired to determine the sugar in a solid tanning material. The weighed quantity of the ground material (see below) is extracted with 1 litre of water at 100° C. in the apparatus described by Kech (this Journal, 1888, 528), the process occupying two hours. 600 cc. of the filtered extract are concentrated to 200 cc., and the tannins, &c., are precipitated from this solution by the addition of 20 cc. of lead acetate solution (see below); after 15 minutes, during which time the flask has been frequently shaken, the liquid is filtered through a dry filter, and to 100 cc. of the filtrate, 10 cc. of sodium sulphate solution (equivalent to the lead acetate solution) are added; the lead sulphate is removed by filtration through a dried filter and 25 cc. of the filtrate are taken for the estimation of the sugar. There are mixed in a beaker, 30 cc. of copper sulphate solution, 30 cc. of alkaline Rochelle salt solution (both prepared according to Allihn's directions), and 80 cc. of water. The whole is heated to boiling and the 25 cc. of extract are added; the beaker is then placed in a boiling water bath for 30 minutes. The cuprous oxide is filtered, dried, heated, and reduced in the usual manner. Should the tan-stuff be poor in sugar, or, in case the method is applied for the analysis of a leather, this be not weighted with sugar, a larger volume



than 25 cc. may be taken for the estimation, but a correspondingly smaller volume of water must be added to the Fehling's solution, in order that the total volume from which the cuprous oxide is to be precipitated may not exceed 145 cc.

The quantity of material extracted must vary with its nature, as follows:—

	Grms.
Oak-wood, chestnut-wood, and spent bark .....	30
Oak-bark, willow-bark, pine-bark, fir-bark, larch-bark, &c. ....	20
Quebracho-wood, sumach, rove, cayota-bark, garonille, canniers, .....	10
Myrabolans, valonia, knoppern .....	7-10
Mimosa-bark .....	5-10
Divi-divi, algarobilla and trillo .....	5

In the examination of extracts, 8-20 grms. of the sample are dissolved in 250 cc. of water, and 200 cc. of this solution, without filtration, are treated with lead acetate, &c., exactly as described above, the sugar determination being made with 25 cc. or more of the final filtrate under the conditions already given. Tanning liquors must generally be concentrated before the precipitation with lead acetate.

In estimating the sugar in a leather it is usual to use the 20 grms. of the finely-divided sample which have already served for the estimation of moisture, and have been extracted with carbon bisulphide for the estimation of the fat. This portion is extracted in a Koeb's apparatus at the ordinary temperature in the manner described above. The litre of extract is concentrated to 500 cc., and 200 cc. of this solution serve for the precipitation with lead acetate, &c. Some 40 cc. of the final filtrate are used for the sugar determination.

The lead acetate solution for this work is prepared by mixing 300 grms. of lead acetate with 100 grms. of litharge and 50 cc. of water, heating on the water-bath, with replacement of evaporated water, until the mixture is white, digesting with a litre of cold water, and filtering.

A large number of figures showing the percentage of tannin and of substances capable of directly reducing Fehling's solution, summarised under the name sugar, in commercial tanning materials and extracts, is quoted. The values in each case are calculated for the mean water-content of the material (see page 1072). In this place it is only possible to give a tabular summary of these numbers.

	Mean		"Sugar"-Content.			Proportions of Acid-yielding Substances to 100 Tannin.
	Tannin-Content.	Mean.	Minimum.	Maximum.		
	Per Cent.	Per Cent.	Per Cent.	Per Cent.		
Pine-bark extract .....	25.00	7.84	4.58	9.44		31.4
Pine-bark .....	11.63	3.53	2.65	4.47		30.4
Oak-bark (young) .....	10.10	2.65	1.75	3.46		26.2
Divi-divi .....	41.50	8.39	7.98	8.83		20.2
Algarobilla .....	43.00	8.23	6.24	10.49		19.1
Myrabolans .....	30.09	5.35	3.15	7.05		17.8
Sumach (Italian) .....	28.00	4.53	..	..		16.2
Oak-bark extract (Slavonic) .....	28.00	3.07	2.47	3.92		11.0
Chestnut-wood extract (normal liquid) ....	30.00	2.87	2.61	3.53		9.6
Valonia .....	28.80	2.60	1.21	3.57		9.3
Cayota-bark .....	22.00	1.65	..	..		7.5
Trillo from Valonia .....	43.50	2.41	..	..		5.5
Garonille .....	25.00	1.00	0.67	1.51		4.0
Rove .....	29.00	1.13	..	..		3.9
Quebracho extract (solid) .....	70.00	2.41	1.04	3.80		3.4
Mimosa-bark .....	32.00	0.91	0.33	1.57		2.8
Knoppern .....	30.00	0.65	0.54	0.71		2.2
Quebracho-wood .....	22.00	0.25	0.10	0.65		1.1

The materials mentioned in the body of the paper, but not included in the above list, apparently because of their slight importance in the German market, are as follows:—

	Tannin.	"Sugar."	"Sugar": 100 Tannin.
	Per Cent.	Per Cent.	
Willow-bark .....	10.00	2.16	21.60
Hemlock-bark* .....	12.32	6.71	5.76
Canazze .....	30.00	6.24†	20.80
Gambier‡ .....	47.18	1.85	3.92
Catechu§ .....	39.89	0.50	1.25

\* Water, 11.50 per cent.

† Water, 17.19 per cent.

‡ Sugar-content varies greatly.

§ Water, 20.00 per cent.

An inspection of the table will show that those materials which contain a high ratio of sugar to tannin are those which have always been used for the tanning of such leather as requires an acid liquor for the production of full weight. It must not be forgotten, however, that although there is direct evidence that the Fehling-reducing compounds give rise to acid, there is not yet evidence that they are the sole cause of acidity; starch under certain conditions can give rise to acids. Furthermore, the influence of the temperature of extraction and of the presence of the tan-stuff itself in the layers, on the production of acid, and the influence of the nature of the acid in producing the plumping effect, have yet to be elucidated.

Several useful remarks as to the application of extracts in Germany are to be found in the original paper.

A number of figures is quoted to show that in the process of tanning the sugar decreases and the acid increases in the liquors as the hides pass from pit to pit.

The following analyses of extracts are of interest:—

	Water.	Tannin.	Organic Matter, not Tannin.	Extract Ash.	Insoluble Matter.	Sugar.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Chestnut wood extract .....	69.73	26.19	19.13	0.27	2.98	2.61
" " (probably adulterated) .....	49.17	25.64	21.47	0.29	3.43	11.70
Hemlock extract .....	50.60	31.90	..	..	..	4.12
Oakwood extract .....	59.60	26.52	11.72	1.52	0.64	2.36
" " from young wood .....	65.12	11.64	16.12	3.01	0.81	1.87
Quebracho wood extract, solid .....	16.72	73.39	6.67	1.17	1.45	1.04
" " syrupy .....	46.06	47.90	5.96	0.70	2.28	0.90
" " adulterated with molasses* .....	15.11	51.33	28.92	2.91	1.73	..

\* Contains sugars capable of directly reducing Fehling 1.76 per cent.; sugar capable of inversion, calculated as cane sugar, 13.86 per cent.

To an acid liquor from a tan yard there was added some glucose; the liquor was examined from time to time during 10 days, when it was found that the total acidity (calculated as acetic acid) had decreased from 0.453 grm. to 0.436 grm. per 100 cc., whilst the sugar had decreased from 0.415 to 0.053 grm. per 100 cc.; at the same time the non-tannins in the liquor had decreased from 1.101 to 1.095, the tannins and ash remaining constant. From this it would seem that the sugar in a liquor is capable of direct conversion into gaseous or very volatile products without passing through the stage of an acid. The authors have never found more than 0.5 per cent. of total acid in a tanning liquor.

The authors conclude with a table showing the minimum, maximum, and mean sugar content of 14 different classes of air-dried leather.† The sugar-content in any particular class of leather does not seem to be characteristic; it will suffice, therefore, to say that the mean content as shown by 171 analyses is 0.22 per cent., and that the maximum in any individual is 1.40 per cent., the minimum being a trace. In weighted leather from 1.50 to 16 per cent. of sugar has been found.—A. G. B.

#### *The Quantitative Separation of Amorphous Nitrogenous Organic Compounds contained in Beer Wort. II. Schjerning. Zeits. anal. Chem. 33, 1894, 263—299.*

THOUGH the results of various workers have been somewhat divergent, there is little doubt that beer worts contain true albumens, hemialbuminoses or propetones, and peptones, but no satisfactory method for the separation of these groups has yet been found. In this paper, the author describes a process, which, though not yet fully worked out, seems to him an advance on those yet used. As precipitants he employs caustic soda, barium hydrate, stannous chloride, normal lead acetate, ferric acetate, uranium acetate, and phosphomolybdic acid in the order named. Each precipitant precipitates (with but slight exceptions which are noted) the whole of the nitrogenous compounds of those preceding it, and in addition a special class which the author denominates its own "absolute precipitate." The precipitates are collected on a filter which has been washed with hydrofluoric acid, and the nitrogen estimated by Kjeldahl's method. If Schleicher and Schüll's 11 c.m. hydrofluoric washed filters are used, the correction necessary for the nitrogen contained in the filter is 0.2 cc. of decinormal acid. It is necessary to keep strictly to certain conditions as to quantity of reagent used, temperature of liquid, time allowed for precipitation, and liquid used for washing the precipitate, &c., and these have been ascertained by the author by careful experiments.

**Caustic Soda Precipitation.**—2—4 cc. of a 10 per cent. solution of caustic soda is added to 100 cc. of wort and the

whole allowed to stand for 20—24 hours. The precipitate, which is crystalline, is then collected on a filter, and washed with cold water. The nitrogen in the precipitate is estimated by Kjeldahl's method, and a correction of 0.27 cc. of decinormal acid made for every 100 cc. of liquid + wash-water.

**Barium Hydrate Precipitation.**—25—50 cc. of wort are treated with one-third to one-half that quantity of 5 per cent. solution of barium hydrate and left to stand for 20 hours out of contact with the air. The precipitate is washed with 1.2 per cent. barium hydrate and a correction of 0.05 cc. decinormal acid for every 25 cc. filtrate + washing solution made to the nitrogen determination.

**Stannous Chloride Precipitation.**—50 cc. of the neutral or slightly acid (with HCl) wort are treated with 10 cc. of stannous chloride solution, the mixture allowed to stand for at least 6 hours at the ordinary temperature and the precipitate washed with cold water. No correction is needed. The stannous chloride should be made in the following way:—50 grms. of granulated tin are dissolved in concentrated hydrochloric acid and the excess of acid evaporated till the residue weighs 120—130 grms. This residue is diluted with water to 1 litre and should give a perfectly clear solution, which should be kept over tin.

**Lead Acetate Precipitation.**—25—30 grms. of neutral or very slightly acid wort are mixed with one-fifth to one-third of the bulk of a 10 per cent. normal lead acetate solution, the mixture heated to boiling, allowed to cool again, and filtered. The precipitate is washed with cold water. The correction to be added is 0.15 cc. decinormal acid for each 100 cc. of filtrate and wash water.

**Ferric Acetate Precipitation.**—20—25 cc. of slightly acid (with acetic acid) wort are mixed with one and a half to twice the volume of ferric acetate, and the mixture heated to boiling, and if necessary a drop or two of sodium acetate added. The precipitate is at once collected and washed with cold water. No correction is needed. The ferric acetate solution is prepared by dissolving 25 grms. of pure ferric acetate in 1 litre of water and adding 25 cc. of 45 per cent. acetic acid.

**Uranium Acetate Precipitation.**—25 cc. of neutral or very slightly acid wort are mixed with 20 cc. of a 10 per cent. solution of uranium acetate, the mixture heated to boiling, and then allowed to stand for 20 hours at the ordinary temperature. The precipitate is washed with a 1 to 2 per cent. solution of uranium acetate, and a correction made of 0.1 cc. of decinormal acid for each 100 cc. of filtrate and wash water.

**Phospho-molybdic Acid Precipitation.**—25 cc. of wort are mixed with 5 cc. of 10 per cent. sulphuric acid and 25 cc. of 10 per cent. phospho-molybdic acid. The mixture is left at the ordinary temperature for 20 hours, the precipitate washed with 1 per cent. sulphuric acid, and a correction

† In bark-tanned leather the results are calculated on the basis of 18 per cent. of water in the air-dried leather.

made of 0.5 cc. deci-normal acid for each 100 cc. of filtrate and wash-liquid.

*Character of the Precipitates.*—The caustic soda precipitate consists of a part of the ammonia present in the wort, combined with magnesium, lime, and phosphoric acid also present. It generally also contains some calcium or magnesium saccharate. About one-quarter of the ammonia of the wort is so precipitated. The examination of the other precipitates has not yet been definitely ascertained, but some information thereon has been obtained. The baryta precipitate contains the same ammonia precipitate as the soda, and in addition a precipitate (the absolute baryta precipitate), containing probably the nuclein-like, acid decomposition products (denuclein) from diastase. The tin precipitate does not contain the soda precipitate, and is probably identical with the absolute baryta precipitate. The lead precipitate does not contain the soda precipitate, but consists of the barium precipitate and a precipitate containing probably mucodin or an acid albumin. The ferric acetate precipitate does not contain the soda precipitate, but consists of the lead precipitate plus its own special precipitate containing probably propeptones. The uranium precipitate contains the soda and iron precipitates plus the true peptones and the phospho-molybdic acid precipitate, the whole of the ammonia, the iron precipitate, and the true peptones.—L. T. T.

*Centrifugal Methods of Milk Analysis.* A. Scott. A paper read before the Glasgow City Analysts' Society. Abstracts No. 4, 1893-94, 9.

THE author's experience with the Leffmann-Beam process is very satisfactory; out of 38 tests, the difference between duplicate tests varied from 0.0 to 0.15 per cent. Examples are given to show that the results obtained by this process are the same as by the Adams process.

With the Lister-Babcock process, out of 130 tests, the difference between duplicate tests was never greater than 0.1 per cent., and the deviation from results yielded by the Adams process was never greater than 0.12 per cent. The machine used was of the new low form, and Embrey's directions were followed. The author finds the correction, + 0.3 per cent., a practically constant figure, though, with milks containing only 1 to 2 per cent. of fat, + 0.35 seems in many cases a better correction. The author believes the necessity for the correction is due both to the method of graduating the bottles and to some of the fat remaining in the acid liquid; thus more fat was obtained both by longer and by more rapid whirling. That the graduation has something to do with the necessity for the correction is shown by the fact that a separated milk, which, by the Adams process, gave 0.24 per cent. of fat, gave 0.2 per cent. in six tests by the Lister-Babcock method, no correction being applied. All bottles should therefore be standardized with a sample of milk which has been tested by Adams' process.

The author regards the two centrifugal processes as equally accurate; but he prefers the Lister-Babcock machine because it is the better design, free from danger, cheaper, and does not involve the use of fusel oil.—L. A.

*Soxhlet's Areometric Method of Determining the Fat in Milk.* H. Timpe. Chem. Zeit. 1894, 18, 392-394.

THE author investigates the causes of discrepancy between the results obtained by Soxhlet's areometric method for the determination of the fat in milk, and those given by the Adams' gravimetric process. He points out that the amount of ether forming the upper layer after the usual process of shaking out the fat direct from the milk, depends both on the quantity of ether used and on the quantity of water in the aqueous layer below the ether, the solvent action of the water and the ether being mutual. Thus, adopting the usually accepted figures for the solubility of ether in water and of water in ether, viz., 1 part in 12 and 1 in 35, respectively (at 17.5° C.) the amount of aqueous ether left

as a separate layer and containing the fat of the milk in solution, is expressed by the equation—

$$X = \frac{132W}{419} - 36W \quad \text{I.}$$

where X is the quantity of aqueous ether, A that of dry ether and W that of water free from ether. It is obvious that a variation will be produced when a milk containing more or less water than the average is used, and the total quantity of aqueous liquid will thus be affected and the amount of ether which it takes into solution, correspondingly influenced. This variation becomes appreciable when rich milks (containing more than 5 per cent. of fat) are treated by Soxhlet's method, as in their case it is usual to dilute the sample with a known amount of water before analysis. The diminution of the volume of aqueous ether which is thus brought about, causes a rise in the specific gravity of the ethereal solution of fat and a plus error in the final result. This error may amount to as much as 0.1 per cent. It is evident, therefore, that allowance must be made for the actual percentage of water in any given sample of milk, when precise results are required. This can be done by means of one of the formulæ connecting the total solids with the fat and specific gravity of the milk, e.g. Fleischmann's. From this it follows that W in the equation given above is a function of the specific gravity *s* of the milk and of the specific gravity *S* of the ethereal solution of fat. As always the same quantity of ether is used, *X* is constant and the resulting amount of aqueous ether *N* is simply a function of *s* and *S*.

Assuming therefore that no alteration of volume takes place on dissolving fat in ether, the percentage of fat *Y* can be calculated from the quantity of ether, and the specific gravity of the ethereal solution of fat, the specific gravity of the fat itself being taken as constant; the following equation represents these relations—

$$Y = \frac{X(S - 0.7211)0.93}{0.7211(0.93 - S) \frac{rs}{100}} \quad \text{II.}$$

*X* being the quantity of ether calculated according to equation I, *S* the specific gravity of the ethereal solution of fat, *r* the volume of milk taken, *s* its specific gravity, and the constants 0.7211 and 0.93 the specific gravities of the aqueous ether and of the fat at 17.5° C. respectively. Seeing that *X* is a function of *S* and *s*, this equation can be written—

$$Y = \frac{f(Ss)(S - 0.7211)0.93}{0.7211(0.93 - S) \frac{rs}{100}} \quad \text{III.}$$

Whence it appears that the percentage of fat in the milk is only dependent on the specific gravity of the milk and on that of the ethereal solution of fat. It is therefore practicable to construct a table by means of which the percentage of fat can be arrived at with great exactitude when these two factors are known. The author gives a voluminous table showing that alterations of the quantities of milk, ether, and water used, have but a small effect on the percentage of fat found. The results are, however, uniformly lower than those given by the unmodified Soxhlet method, the difference increasing with the content of fat.

—B. B.

*Estimation of the Total Alkaloids in Cinchona Bark.* C. C. Keller. Apoth. Zeit. 8, 542; Zeits. anal. Chem. 1894, 33, 490-491.

THE author has modified Haubensack's method (this Journal, 1892, 779) as follows:—The dry powder (12 grms.) is shaken in a medicine glass with ether (120 grms.) and 10 per cent. ammonia (10 grms.) added thereto. The shaking is then repeated and continued for half an hour; water is then added (10 grms. for *Cinchona Succirubra*, and 15 grms. for *Cinchona Calisaya*), and the mixture shaken very vigorously for a minute. Of the clear ethereal solution, 100 grms. are poured off, and very vigorously shaken with 3 cc. of dilute sulphuric acid and 37 cc. of

water. The greater portion of the ether is then decanted, and the residual liquid introduced into a separating funnel, the acid solution of the alkaloids is run off and the flask and separating funnel washed with 10 cc. of water. The aqueous solution, after being freed from ether, is shaken with a mixture of chloroform (30 grms.), ether (10 grms.), and ammonia (5 grms.). The extract is run off and the extraction repeated with chloroform (15 grms.) and ether (5 grms.). The combined extracts are then filtered through a filter moistened with chloroform to effect their partial drying, into a tared flask from which the solvent is then distilled. From *Calisaya* bark the alkaloids usually remain in the crystalline condition; from *Succirubra*, however, in the form of a varnish, which persistently occludes chloroform. In this case the residue is treated with 3–5 cc. of absolute alcohol, which is boiled off on the water bath. The alkaloids then remain in the crystalline condition and may be easily dried.—A. R. L.

*Estimation of the Alkaloids in Nux Vomica Seeds.* C. C. Keller. Apoth. Zeit. 8, 542; Zeits. anal. Chem. 1894, 33, 491–493.

15 grms. of the well-dried and finely-powdered substance are freed from fat as follows.—The material (15 grms.) is introduced into a tube 12 c.m. long and 25 mm. wide, which at its lower end is drawn out to 7 mm. wide and 5–6 c.m. long, the point terminating obliquely: within the constricted portion of this tube is placed a loosely-fitting wad which must be free from fat. The sample is then washed with ether until the ethereal washings no longer give a residue on evaporation. By careful manipulation about 100 cc. of ether are requisite. The ethereal solution contains, however, some alkaloid which is recovered by treatment with 5 cc. of N hydrochloric acid and shaking repeatedly with 10 cc. portions of water until the aqueous extract amounts to 25 cc. The fat-free powder is then introduced into a flask, mixed with a sufficient quantity of ether, so that the whole weighs 100 grms., together with 50 grms. of chloroform and 10 grms. of 10 per cent. ammonia, the whole being shaken for half an hour. The acid solution of the alkaloids (mentioned above) is now added, and after well shaking and allowing to subside, 100 grms. of the ether-chloroform solution are filtered into a tared Erlenmeyer's flask, from which the solvent is distilled, leaving the alkaloids as a varnish. The residue is treated two or three times with absolute alcohol, which is evaporated off on the water bath: the alkaloids are thus obtained in the crystalline condition, and after drying at 95°–100° C. may be weighed. The purity of the bases may be established by titration with alkali and iodoquin. It is absolutely important not to employ more chloroform than above prescribed: if more chloroform be taken the alkaloids will be contaminated by the glucoside Loganiin, whilst if too little chloroform be used a loss is to be feared, for inasmuch as the alkaloids are sparingly soluble in pure ether, they separate out.

*Strychnos nux vomica* bark is extracted in the finely-pulverised condition first with a mixture of chloroform and ether and subsequently with pure ether: the remainder of the process is conducted as above described.

The author treats "*Extractum strychni spirituosum siccum*." Pharm. Helv. III. Ed., thus:—1.5 grms. of the dry finely-ground extract are introduced into a flask, shaken with 10 grms. of water until the whole is moistened, and 30 grms. of chloroform and 60 grms. of ether are added. After shaking for some time 5 grms. of 10 per cent. ammonia are added, and, after shaking, the mixture is allowed to separate. A known weight (60 grms.) of the chloroform-ether extract is then filtered, and the remainder of the operation conducted as already described. An extract prepared by the author was found to contain 21.2 per cent. of alkaloids.

*Quantitative Separation of Strychnine and Brucine.*—0.2–0.4 gm. of the dry mixture of alkaloids is dissolved in 10 cc. of 10 per cent. sulphuric acid by heating on the water-bath. After complete cooling, 1 cc. of nitric acid of sp. gr. 1.41–1.42 is added and the mixture shaken. The strychnine sulphate which usually separates from the

solution is redissolved on addition of the nitric acid; simultaneously, however, the red brucine nitric acid coloration is developed. After allowing the mixture to remain for 1–1½ hours whereby the brucine is completely destroyed, 40 grms. of chloroform, an equal quantity of ether, and 10 cc. of 10 per cent. ammonia are added, and the whole shaken. Forty grms. of the extract are filtered into a tared flask, from which the mixture of chloroform and ether is distilled. The residual strychnine is dried at 95°–100° C. and weighed. The author found as a mean value 47.16 per cent. of strychnine in *strychnos nux vomica* seeds.

—A. R. L.

*New Method for the Estimation of Salicylic Acid and Salicylates.* L. Barthe. Bull. Soc. Chim. 11—12, 1891, 516–522.

This new method is based upon the observation that salicylic acid can be accurately estimated by titration with caustic soda, using phenolphthalein as indicator, and further that salicylates are completely decomposed by hydrochloric acid, a slight excess of which is without effect upon the salicylic acid at the temperature of the water-bath. In carrying out the new method the salicylate is decomposed with hydrochloric acid in slight excess, the whole then being evaporated to dryness on the water bath at a temperature not exceeding from 50 to 60° C. The residue is dissolved in boiling water and the free salicylic acid titrated with caustic soda in the presence of phenolphthalein. This titration being finished, some nitric acid is next added and the hydrochloric acid present estimated by titration with silver nitrate, yellow potassium chromate serving as indicator. The amount of hydrochloric acid thus found is equivalent to the amount of base present in the salicylate. This method answers well for the analysis of the salicylates of the alkalis and alkaline earths. In the salicylates of manganese and bismuth the metal is best estimated by ignition, in those of zinc and lead by the general methods. *Mercuric salicylate* is analysed by first dissolving it in a moderate excess of caustic soda, acidifying with hydrochloric acid, and evaporating to dry mass at a temperature not exceeding 45° C. The dry mass is then dissolved in a definite volume of one fifth normal potassium iodide, and the amount of salicylic acid present estimated by titration with standard soda. The quantity of mercury contained in the solution is then determined by titration according to Personne's method, the rose colour of the phenolphthalein having been removed by the least possible quantity of hydrochloric acid. For the analysis of the salicylates of the alkaloids this new method is not generally applicable, but on the other hand it may be mentioned that it answers very well for the analysis of the alkaline benzoates.

—C. O. W.

*Investigation of Certain Commercial Forms of Artificial Musk.* Bericht von Schimmel and Co., October 1893. Zeits. anal. Chem. 1894, 33, 487–488.

The properties of Baur's artificial musk have been described (this Journal, 1891, 1024).

The sample examined, melted at 108° C., and dissolved somewhat readily in hot water, but only very sparingly in petroleum spirit. It was extracted about 20 times with boiling petroleum naphtha, the residue being dried and crystallised 6–8 times from boiling water. It proved to be acetanilide.

To estimate the amount of adulteration in this sample, a determination of nitrogen existing as nitro-groups (NO<sub>2</sub>) was made by Spindler's modification (Annalen, 224, 288) of Liempicht's process (Ber. 11, 35). This gave 3.25 per cent. of NO<sub>2</sub>, corresponding with 6.67 per cent. of trinitrobutyltoluene.

Acetanilide is used as a vehicle in the cases of other odorous products made by the same firm which manufactured this particular sample of artificial musk.—A. R. L.

*The Examination of Wool-fat.* W. Herbig. Dingl. Polyt. J. **292**, 1894, 12 and 66.  
See under XII., page 1068.

### PATENT.

*An Improved Process for the Extraction and Estimation of Eucalyptol.* L. R. Scammell, Adelaide. Eng. Pat. 14,178. July 23, 1894.

A SOLUTION of phosphoric acid, preferably of sp. gr. 1.785, is added to the oil containing eucalyptol. A crystalline precipitate of eucalyptol phosphate is thus produced, which is collected and decomposed by hot water, the quantity of liquid eucalyptol thus separated is easily measured.—T. E.

### ANALYTICAL AND SCIENTIFIC NOTES.

*Remarks on the Heating and Spontaneous Combustion of Hay.* M. Berthelot. Bull. Soc. Chim. 1894, **11**—12, 810—812.

THE spontaneous combustion of heaped up and badly dried hay has been often attributed to a peculiar kind of fermentation; but the author refers to his work, on this subject, of 1855, showing that all fermentation is based upon an exothermic chemical reaction, the ferment itself acting only to start the reaction, the combustion which may follow being due to an oxidation process. Herbs, heaped up as soon as cut, rot from excess of moisture, without much rise of temperature, and become unfit for use as cattle-food; but if they are spread out, they die, and then lose rapidly the water which they obstinately retain while living. At the same time they undergo oxidation and set free carbonic acid, such being the case with good hay, when stacked. But if the hay be heaped up before being sufficiently dry, it is subject to alterations of another kind, with considerable evolution of heat, due to fermentation. Ferments, however, cease to act at 40° C.; yet the temperature may continue to rise, even to 100° or more, with absorption of oxygen from the air, as the author has proved by direct experiment. The oxidation which goes on, increases with the rising temperature, and is of a purely chemical character, empyreumatic products being generated, giving the taste and odour recognisable as emanating from "heated" hay. At a temperature far below a red heat, combustion may ensue. Such spontaneous combustion has occurred in hay contained in a stove maintained at 140°. Charcoal prepared for making powder has been observed to give off carbonic acid at 100°; and some specimens, after cooling in a close vessel, will inflame on contact with air. All such phenomena belong to the same order. The conclusion is, that the rise of temperature leading to the spontaneous combustion of hay, results from purely chemical reactions, in connection with products modified by previous fermentations.—E. S.

*The Crystallisation of Cellulose and the Chemical Composition of the Vegetable Cellular Membrane.* E. Gilson. Bull. Soc. Chim. **11**—12, 1894, 590—591.

THIS work, originally a microscopic investigation of vegetable morphology, has led to an interesting chemical research. In treating microscopic sections by Schweitzer's reagent, and then by ammonia, the author has obtained crystals presenting all the characteristics of cellulose. He has prepared cellulose in quantity by Schulze's method, employing the stalks of young cabbages, and has proved its identity with the crystals before prepared, since when treated first with the copper solution and then with ammonia it was completely converted into a crystalline mass; further, on treating a great number of sections with Schweitzer's solution and ammonia to cause the cellulose to crystallise, and then submitting them to Schulze's treatment for the preparation of pure cellulose, nothing was left but crystals of cellulose. The cellulose thus obtained is the

only hydrate which in the membrane is coloured by iodised chloride of zinc, and which by hydrolysis gives exclusively dextrose.

In certain cases the author found, besides cellulose, another carbohydrate, which he names "paramannane," and which was prepared as follows:—Coffee, after treatment with ether, dilute potash, and dilute sulphuric acid, was dissolved in Schweitzer's solution. Through this solution, containing an excess of ammonia, a current of carbon dioxide was passed, when the cellulose was precipitated, whilst the paramannane remained in solution. This last solution was rapidly evaporated and washed with very dilute acid. Paramannane thus prepared is a white powder resembling sperm, insoluble in water and alkalis, soluble in concentrated sulphuric acid, but attacked with difficulty by dilute sulphuric acid. Hydrolysed it yields manna. Microscopically it is seen in the form of sphere-like crystals.

The researches of the author shew that cellulose is always found in the interior of cells and that it is not removed completely by Schweitzer's reagent. This explains why Frémy admits a variety of cellulose (paracellulose) which only dissolves after first acting on it with dilute acids.

—A. W.

*A Novel Application of Bichromated Gelatin.* Izarn. Comptes rend. **118**, 1894, 1314—1315.

THE author recommends coating silvered surfaces in general, and the mirrors of astronomical telescopes in particular, with an extremely thin film of bichromated gelatin in order to protect them from atmospheric tarnish. Such films are stated to be very adhesive, durable, and transparent; and it was found by experiment that surfaces thus protected remained perfectly bright, even after prolonged contact with sulphuretted hydrogen. The process has been applied to the mirror of a telescope at the Toulouse observatory with very satisfactory results, the sharpness of definition, &c. of the instrument being in no way deteriorated.—H. T. P.

*Cobalt Nitrate as an Antidote for Cyanide Poisoning.* Eng. and Mining J. **58**, 1894, 418.

COBALT nitrate is found by Dr. Johann Antal, a chemist, of Hungary, to be an antidote to prussic acid and cyanide poisoning. First he tried the cobalt on animals, and then, presumably at different times, on 40 living persons who had been accidentally poisoned by prussic acid, and in all cases the results are reported to have been satisfactory.

### New Books.

THE RISE AND DEVELOPMENT OF ORGANIC CHEMISTRY. By CARL SCHORLEMMER, LL.D., F.R.S., late Professor of Organic Chemistry in the Owens College, Manchester. Revised Edition. Edited by ARTHUR SMITHELLS, B.Sc., Professor of Chemistry in the Yorkshire College, Leeds. London and New York: Macmillan and Co. 1894. Price 5s.

SVO VOLUME, bound in cloth, and containing, as frontispiece, an excellent photo-engraving of Schorlemmer, with his autograph beneath. The work commences with a biographical sketch of Schorlemmer by Professor SmitHELLS, a bibliographic list of his investigations, preceded by a brief Preface, a Table of Contents, and the text of the work, covering 268 pages. The work is greatly enhanced in value by the addition of an alphabetical index

of names of investigators and authors, and also of subject-matter, a means of guidance lacking in the original. In the Table of Contents, the sub-division of the text, or analysis of the matter, is made clear. Chapter I. Chemical Knowledge of the Ancients, sketched down to the times of Lavoisier, Berzelius, and Gmelin. II. Vital Force. First Artificial Production of an Organic Compound. Theory of Substitution, &c. III. Dumas's Theory of Types, &c. IV. Introduction of Radicals, Types, and the Beginning of the Theory of Valency. V. Compound Radicals, inorganic as well as organic. Definition of Organic Chemistry, &c. VI. Organic Analysis. Calculation of Formulae. VII. Constitution of Organic Compounds. Isomerism. VIII. Isomeric Paraffins. Alcohols. Unsaturated Compounds. IX. Aromatic Compounds, Kekulé's and Ladenburg's Views. Geometric Isomerism, Kolbe's Views. X. Synthesis of Organic Compounds. XI. Synthesis of Aromatic Compounds. XII. Synthesis of Indigo, and Problems still to be solved.

**CHEMIKER-KALENDAR, 1895.** Ein Hilfsbuch für Chemiker, Physiker, Mineralogen, Industrielle, Pharmazeuten, Hüttenmänner. Von Dr. RUDOLF BIEDERMANN, Sechszehnter Jahrgang. Mit einer Beilage. Berlin: Verlag von Julius Springer, 1895. London: H. Grevel and Co., 33, King Street, Covent Garden. Price 4s. 6d. Post free.

THIS well-known pocket-book and vade mecum for chemists and analysts, has now appeared for 1895. It is strongly bound in cloth, and contains an almanack for 1895 inside the cover, and a table of frequently used atomic weights and their multiples. Among the additions and improvements in this edition for 1895, may be mentioned Lunge's completed tables of reductions of gas volumes. Knietzsch gives the results of his work on liquid chlorine. Brendel contributes a valuable table on the Frigorific Mixtures investigated by him, whilst Stohmann adds many new results of his in Thermo-chemistry. In matters relating to Solubility, Heats of Formation, Refraction of Light, Diffusion, &c., partly the original work and partly the Physico-chemical Tables of Landolt and Börnstein have been laid under contribution. Among the "Chronological Investigations," there will be found important additions to and extensions of the Tables of Colouring Matters, as well as new data bearing reference to the fats and tanning materials.

A Chronological Table of the principal events in the History of the Physical Sciences is given, and also a Notebook Calendar with blank pages for the different days of the year, in which observations, data, facts, &c., may be entered up and recorded. Then follows tabulated matter of Specific Gravities, of Gases and Liquids, Salts and Alcohols, Tables of Solubilities, Properties of Chemical Substances, Analysis, Volumetric Analysis, Spectrum Analysis, Gas Analysis, Physiological Chemical Analysis, and Technological Investigations relating to:—I. Water. II. Assaying. III. Fuels. IV. Sulphuric Acid. V. Alkali. VI. Bleaching Powder. VII. Nitre and Explosives. VIII. Textile Fibres, Paper. IX. Photography. X. Starch and Sugar. XI. Colours, &c. XII. Fats and Oils. XIII. Tanning Materials. XIV. Ceramics. XV. Glass. XVI. Mortars and Cements, Mineralogy.

#### BEILAGE ZUM CHEMIKER-KALENDAR. 1895.

THIS forms a supplement of useful tables of a more thorough-going technological character:—I. Leading Foreign Money Currencies. II. Series of Mathematical Tables. III. Weights and Measures. IV. Tables of Heat and Cold Measurements. V. Expansion and Contraction. VI. Tension of Vapours. VII. Barometry. VIII. Sound. IX. Light. X. Electricity. XI. Magnetism. XII. Diffusion and Capillarity. XIII. Elasticity, &c. XIV. Specific Gravity.

**A TREATISE ON CHEMISTRY.** By SIR H. E. ROSCOE, F.R.S., and C. SCHÖDDEMMER, F.R.S. Vol. I. THE NON-METALLIC ELEMENTS. New Edition, completely revised by Sir H. E. Roscoe, assisted by Dr. H. G. Colman and A. Harden. With 374 illustrations and a Portrait of Dalton, engraved by C. H. Jeens. London and New York: Macmillan and Co. 1894. Price 21s.

THIS is a new, completely revised and reprinted edition of Volume I. of this work of Roscoe and Schödemmer's. Compared with Volume I. of the previous edition, it has grown considerably in bulk. It contains besides the Prefaces (the latter one for this, the third edition), also a list of contractions of the names of journals referred to in the text, the table of contents, and 875 pages of subject-matter. The book ends with a complete alphabetical index of subject-matter. The text is profusely illustrated with admirable engravings, and the technical bearings of the science have received careful attention.

Turning to the divisions of the work, the opening chapter forms an HISTORICAL INTRODUCTION. It is followed on page 41 by another division of the work devoted to GENERAL PRINCIPLES OF THE SCIENCE, and dealing with *Properties of Matter, Elementary of Compound Bodies, Properties of Gases, Boiling Points of Liquids, Laws of Chemical Combination, Experimental Methods for the Determination of Molecular Weights*. On page 126 commences the description of the NON-METALLIC ELEMENTS, among which—following the Compounds of Carbon with Hydrogen, the Halogens, Oxygen, Sulphur, and Nitrogen—are descriptions, theoretical and technical, of Coal-Gas, Wood-Gas, Oil-Gas, Water-Gas, with a chapter on the Nature of Flame, including a description of the experiments of Smithells. A tolerably full account of the Elements of the Science of Crystallography and of its applications fills pages 827 to 873.

**EINFÜHRUNG IN DAS STUDIUM DER QUALITATIVEN CHEMISCHEN ANALYSE.** VON CARL FRIEDHEIM, PH.D., Privatdozent an der Universität Berlin. Ste gänzlich umgearbeitete Auflage von C. F. Rammelsberg's Leitfaden der qualitativen chemischen Analyse. BERLIN: Carl Habel, Verlag-buchhandlung, Wilhelm-Strasse 33, S.W. (48). LONDON: H. Grevel and Co., 33, King Street, Covent Garden. 1894. Price 7s. 6d.

THIS work on Qualitative Analysis is dedicated by the author to Professor Clemens Winkler. It takes the form of an 8vo volume in paper cover, containing preface, table of contents, 335 pages of text, and a full alphabetical index of subject matter. The book really consists of a completely renovated edition of Rammelsberg's "Leitfaden der qualitativen chemischen Analyse." The subject-matter is sub-divided as follows:—I. GENERAL RULES IN CARRYING OUT QUALITATIVE ANALYSIS. II. SYSTEMATIC DEVELOPMENT. III. REACTIONS.

**A.—METALS.** (1.) Group of the Alkali Metals. (2.) Group of the Alkaline Earth Metals. (3.) Metals proper. (3a.) Metals not precipitated by sulphuretted hydrogen from acid solution, but precipitated by ammonium sulphide. (3b.) i. Metals precipitated by sulphuretted hydrogen from acid solution, the sulphides being insoluble in ammonium sulphide. ii. Metals precipitated by sulphuretted hydrogen from acid solution, the sulphides being soluble in ammonium sulphide.

**B.—METALLOIDS.** 1. Acids precipitated by barium chloride from neutral solution. 2. Acids precipitated by silver nitrate from acid solution. 3. Acids precipitated neither by barium chloride nor silver nitrate.

**IV. Employment of foregoing methods.** 1. Analysis of simple substances. 2. Analysis of complex mixtures. 3. Analysis of Alloys. 4. Analysis of Metallic Sulphides and Arsenides. 5. Analysis of Silicates.

**APPENDIX I.** Reactions of Rare Metals. **APPENDIX II.** List of necessary Reagents, and the usual impurities of injurious character to be found in them.

**DIE MASCHINELLEN HILFSMITTEL DER CHEMISCHEN TECHNIK.** Bearbeitet von A. PARNICKE, Civil-Ingenieur, vormals Ober-Ingenieur der Chemischen Fabrik Griesheim. Mit 337 Abbildungen. Frankfurt a. M.: Verlag von H. Bechhold. London: H. Grevel and Co., 33, King Street, Covent Garden. 1894. Price 10s.

This is a text-book for students of chemical technology desirous of acquiring a sufficient book-knowledge of descriptive chemical engineering. It contains a description of the construction and modes of employment of the large scale apparatus used in the chemical factory. The work consists of a large 8vo volume containing Preface, Table of Contents, and Subject-matter, filling 314 pages. An alphabetical index of subject-matter ends the book. The text is illustrated by 337 excellent engravings of all manner of plant and apparatus used in chemical factories.

The subjects treated of in the work, described and illustrated, are as follows:—

General. Packing and laying lines of pipes. Packing material for economising heat. Packing of Stuffing-boxes. Lubrication of Apparatus and Machinery. Taps and Cocks. Valves, &c. I. Sources of Power. II. Transmission of Power. III. Apparatus for Transport or Transmittal of Materials and Products. IV. Pulverising and Disintegrating Apparatus. V. Mixing Machines, including Towers and Columns for Absorbing, Denitrating, &c. VI. Melting, Dissolving, and Lixiviating Apparatus. VII. Concentrating Apparatus. VIII. Arrangements and Plant for Mechanical Separation, Extraction, and Fractionation. IX. Drying Plant. X. Apparatus for estimating Weight, Temperature, Pressure, Power of Draught, and Tensile Strength. XI. Legal Enactments.

**DIE FABRIKATION VON SCHWEFELSAURE THONERDE.** Von Dr. KONRAD W. JURISCH. Dozent an der Königl. Tech. Hochschule zu Berlin. Berlin: Fischer's technologischer Verlag. Fischer und Heilmann, Köthener Strasse 44. London: H. Grevel and Co., 33, King Street, Covent Garden. 1894. Price 5s.

LARGE 8vo volume, with a full description of the modes of manufacture of sulphate of alumina. The work contains a brief Preface, Table of Contents, and subject-matter covering 113 pages. The work is subdivided as follows: Natural Occurrence of Sulphate of Alumina. Historical Development and Patent Statistics. Manufacture from Cryolite. Manufacture from Clays. Manufacture from Bauxite. Bauxite. Manner of Working. Description of Manufacture. Calcining, Melting, Lixiviating, Filtering the Liquors. Carbonating. Filtering off the Alumina. Solution. The Product. Analysis of Sulphate of Alumina. Properties. Pure Aluminium Sulphate. Basic Aluminium Sulphate. Commercial Sulphate of Alumina. Examination of Sulphates of Alumina. Employment of the Salt. Economy. Statistics.

At present the import has not only completely ceased, but it would not even be astonishing to find that Austrian cement is imported into England and France.

There are many cement factories at the present time in Austria, established mainly on account of the high price of the English article. These factories produce cement of different qualities, but, quality for quality, the Austrian article is stated to be equal to the English cement.

At the present time the price of Austrian cement at Trieste is very nearly the same as that of English cement in England; there is therefore no inducement to import the latter, because of the import duties and freight.

It appears that all or nearly all the Austrian cement factories have adopted the principle of delivering their speciality at the same price in any place in the Empire, themselves paying the difference in the carriage rates. With this system, which is remunerative owing to the very low rates of the railway tariff, and being given also the increased cost of maritime freight and the securing of a return freight by English vessels, it would be sufficient for a demand to arise in England for Austrian cement to be exported with profit to that country.

On the other hand, it is asserted that the abolition of the free port of Trieste has been the last blow to the importation of foreign cement through that port.—*Board of Trade Journal*.

#### ALGERIAN PHOSPHATE DEPOSITS.

Under date of August 11, 1894, our consul at Algiers, Africa, Mr. Charles T. Grellet, writes to the Department of State as follows:—

Ever since the year 1886, the French explorers of Tunisia have mentioned the existence, in the western part of that country, of numerous and important phosphate lands. Some two years ago similar discoveries were made in the "Department of Constantine" (in the "Djebel Dir") (Eastern Algeria), "Djebel Kouik" (Djebel—native for mountain), and at Kissa in the region of Tebessa, 250 km. south of Bone. The Tebessa fields are situated in the north-west of that city, extending over an area of several hundred miles (square), and are said to be almost inexhaustible. The fertiliser, mostly composed of bones and teeth of sharks, is found in a semi-pulverulent state, in strata from 5 metres to 3 metres (10 ft.) thick, intercalated in white marl and siliceous limestone; its grade averages from 60 per cent. to 70 per cent, and at places reaches as high as 85 per cent. of tri-calcic phosphate of lime. Owing to the character of the deposit, the mining of the phosphate is exceedingly easy and cheap; the freight by railroad from the mine to Bone is from 2.31 dols. to 2.50 dols. per ton, and it is calculated that, including all expenses, the cost to the miner free on board vessel at the lading port does not exceed 4.63 dols. per 2,240 lb. The cost of transportation from Bone to the principal countries of Europe, and more particularly Great Britain, France, and the Baltic and Mediterranean ports, is at present per ton as follows:—

	Dols.
To England .....	1.94
To France (northern ports) .....	2.19
To Baltic ports .....	2.67
To France (Mediterranean ports) .....	1.46
To Italy .....	1.66
To Austria .....	1.46

The approximate cost of the phosphate delivered f.o.c. at the port of destination would thus be:—

	Dols.
To England .....	6.57
To France (northern) .....	6.82
To Baltic ports .....	7.30
To France (Mediterranean ports) .....	6.09
To Italy .....	5.72
To Austria .....	6.09

The phosphate is as a rule sold on the basis of the unit of tri-calcic phosphate of lime per 1,000 kilos., delivered

## Trade Report.

### GENERAL TRADE NOTES.

#### PORTLAND CEMENT INDUSTRY IN AUSTRIA-HUNGARY.

An article recently published in the Brussels *Bulletin du Musée Commercial*, deals with the subject of the development of the Portland cement industry in Austria-Hungary. The opinion is expressed that at an early period it will compete with the Russian and French factories.

A few years ago the import of Portland cement through the port of Trieste was considerable; the larger part of the cement imported came from Newcastle, but Dunkirk also sent a small quantity.



f.o.b. at the ports of arrival, and the prices obtained at present in Great Britain and Italy average according to grade of mineral from 14 to 14½ cents per unit.

The phosphate lands of Tebessa, as they stand at present, are under the control of three firms, two of which are English. A railroad 12 km. long has been built to connect at Morsott with the Bone Guelma Railroad from Tebessa to Soukarras. The Djebel Dir mine is 15 km. north-east of Tebessa and 1,500 metres above sea level. It can turn out from 200 to 300 tons daily.

The beds of Djebel and Kouik are in the hands of the Constantine Phosphate Company, with head office at Leith, Scotland. This layer is situated some 25 km. north-west of Tebessa, 1,000 metres above the sea level. The quantity of phosphate existing there has been estimated at 40,000,000 tons; the works are not in full operation, and the connection of the mine with the main line at Tebessa will require the construction of a railroad of about 20 miles in length. The third field, that of Kissa, is in the direct vicinity of Djebel Dir, and is the property of Colonel Corp. But little has been done up to the present date; the work is, nevertheless, progressing steadily.

The general output of these mines was about 5,000 tons in 1893; it will reach at the least from 30,000 to 35,000 tons during the present year, the firm of Crookstone Brothers being the largest contributors.

Other fields have lately been discovered in the same district, and numerous claims been filed, with the view of obtaining from the French Government the concession of these lands. Beds of phosphate have also been found in other parts of the colony, in the Department of Oran, in the region of the Rio Salado, close to the boundary of Morocco, and at Inkermann, 254 km. east of Oran, on the railroad from that place to Algiers. Here the deposits are of a different character, and are generally found in a solid rock. The mineral is also of a lower grade. The output has amounted to very little if anything. In fact, the Tebessa mines are at present the only ones worth mentioning.

As to general conclusions with regard to the industry in Algeria and the effects it will have upon American phosphates, it may be fairly said that the discovery of phosphates in Algeria in almost incredible abundance and of the highest value is indeed a notable event in the history of the colony, and it cannot be doubted but that in the very near future the North African phosphates will prove serious competitors against our American fertilisers in the European markets.—*Engineering and Mining Journal*.

#### SECURING THE FOREIGN MARKETS.

In his general commercial report to the British Foreign Office, Sir C. Oppenheimer, Consul-General at Frankfurt-am-Main, makes some remarks which are intended especially for English manufacturers. We give the following extracts from these remarks:—

"It might be of interest for English trade to study in many directions the business tactics of German merchants and industrials, and to adopt from it what is good and what has proved successful. It would promote English export if its representatives accustomed themselves to facilitate for customers the purchase of goods, as the Germans do; by shipping or consigning them to their warehouses, freight and duty free; by having the goods packed and made up according to the local taste of the customers; by making their offers in the coinage of the country for which the goods are destined; by sending round travellers to customers who not only know the language but are also acquainted with the customs of the country, and, finally, wherever a special traveller would be too dear, by several non-competing firms clubbing together to send a joint representative at their common expense. All these points mentioned here may be regarded as the articles of faith of German exporters, and every Englishman knows how strange in most instances they still appear to the English merchant.

"One can give an idea in a few words of the difference between the old English and the new German tactics in the

export trade. As long as England possessed an unlimited supremacy in the world's markets as exporter of industrial products, the customers were, of course, obliged to come to her, accommodate themselves as best they could to English customs, English taste, English usages, and the English industrial export trade thoroughly anglicized the world; nowadays, when we have to take into account an over-production in many branches of industry, increased offers in the export, and the competition of foreign states (to use a well-known simile) 'the prophet must go to the mountains,' the exporter must visit the customers and accommodate himself to their wants and habits as much as possible. It will be the task of the English trade to acquire these new tactics in the export trade if it does not wish to be left behind by Germany."

The German manufacturers are taking English trade by systematic efforts, as the Consul's statements show. They are reaching out in many directions, not blindly or in a haphazard way, but carefully and systematically. They take pains to make themselves known to their customers in the first place, then to study their wants, find out the kind of goods they want and their methods of doing business; then they seek to conform to the requirements of the special market, believing that in this way they can not only secure new trade, but can also hold the markets when their goods are once introduced. How well they are succeeding and what inroads they are making into British trade the complaints of English manufacturers and their representatives, the trade papers, show.—*Ibid*.

#### GERMAN IMPORTS AND EXPORTS OF DYES FOR THE FIRST SIX MONTHS OF 1894.

*Färber Zeit*, 1894, 5, 368

##### Summary.

		Imports.		Exports.	
		1893.	1894.	1893.	1894.
Indigo.....	100 kilos, net.	7,756	11,541	3,285	3,369
" carmine.....	"	277	130	824	227
Logwood.....	"	244,001	220,500	46,082	35,672
Fustic.....	"	25,254	30,975	5,200	7,275
Brazil wood.....	"	12,388	17,561	1,700	5,537
Dyewood extracts.....	"	27,041	21,588	8,016	6,370
Madder.....	"	976	877	703	830
Cochineal.....	"	309	208	192	144
Safflower.....	"	9	6	1	9
Quercitron.....	"	5,208	5,590	382	679
Catechu.....	"	24,407	28,598	8,230	6,280
Archil, endbear, and lignins.....	"	2,610	637	634	1,761
Alizarin.....	"	77	127	37,536	33,457
Aniline and other coal tar colours.....	"	3,985	3,188	57,512	57,530
Lakes.....	"	35	35	2,630	2,258
Cinnabar.....	"	91	44	1,267	1,502
Minium.....	"	1,909	1,608	28,923	24,305
Prussian blue.....	"	1,679	539	3,060	3,197
Ultramarine.....	"	205	229	20,879	19,665
Printers' black.....	"	141	107	4,394	4,933
Lampblack and "Russ- butter".....	"	1,498	1,492	4,813	5,713
Natural colours.....	"	48,319	32,717	52,102	52,551
Painters' and wash colours.....	"	350	385	7,864	7,049



GERMAN IMPORTS AND EXPORTS OF DYES AND COLOURS  
FOR THE FIRST SIX MONTHS OF 1894.

SOURCES OF SUPPLY AND FOREIGN CONSUMPTION.

Imports.	
Article.	Whence Imported.
Indigo..... 100 kilos. net.	Great Britain (3,245), British East Indies (5,780), Holland (737), Central America (916).
Logwood..... "	Holland (5,304), British West Indies (60,397), Hayti (24,552), Mexico (103,645), U.S.A. (17,351).
Fustic..... "	Columbia (7,111), Mexico (13,760), Venezuela (961).
Brazil wood..... "	West Africa (5,421), British East Indies (1,265), Mexico (438).
Dye-wood extracts.. "	Belgium (1,618), France (8,596), Great Britain (1,489), U.S.A. (7,894).
Madder..... "	Holland (792).
Quercitron..... "	U.S.A. (5,225).
Catechu..... "	Great Britain (4,092), British East Indies (23,693).
Aniline dyes..... "	Belgium (213), France (555), Great Britain (152), Holland (150), Swit- zerland (1,975).
Minium..... "	Great Britain (1,558).
Lampblack..... "	U.S.A. (932).
Natural colours.... "	France (14,278), Great Britain (8,203).
Painters' colours... "	France (69), Great Britain (95).

Exports.	
Article.	Where Exported.
Indigo..... 100 kilos. net.	Austria-Hungary (1,132), Hamburg (662), Holland (338).
Logwood..... "	Denmark (11,850), Austria-Hungary (2,018), Russia (13,000), Switzer- land (2,849).
Fustic..... "	Russia (4,188).
Brazil wood..... "	Russia (3,525).
Dye-wood extracts.. "	Great Britain (572), Austria-Hun- gary (2,762), Switzerland (519), China (556).
Madder..... "	Austria-Hungary (437).
Catechu..... "	Austria-Hungary (1,746), Russia (1,375).
Archil..... "	France (729), Austria-Hungary (290).
Alizarin..... "	Belgium (519), France (1,161), Great Britain (11,757), Italy (763), Hol- land (963), Austria-Hungary (1,414), Russia (1,477), Switzerland (734), Spain (1,041), Turkey (929), British East Indies (6,771), U.S.A. (5,169).
Anilin and other coal-tar dyes.. "	Belgium (1,476), Denmark (284), France (2,046), Great Britain (11,674), Italy (2,950), Holland (954), Norway (297), Austria-Hungary (5,239), Portugal (249), Roumania (367), Russia (1,850), Sweden (1,244), Switzerland (2,030), Spain (1,359), Turkey (167), British East Indies (3,663), China (5,791), Japan (1,161), U.S.A. (9,135).
Lakes..... "	Great Britain (844).
Gumabrar..... "	Great Britain (691), Russia (287).
Minium..... "	Belgium (2,016), France (1,850), Great Britain (4,346), Holland (3,723), British East Indies (1,057), U.S.A. (3,072).
Prussian blue..... "	Belgium (453), Great Britain (815), British East Indies (339), U.S.A. (166).
Ultramarine..... "	Belgium (1,883), France (884), Great Britain (7,505), Holland (1,390), Spain (678), U.S.A. (1,227).
Painters' black..... "	Austria-Hungary (1,152).
Lampblack..... "	France (1,997).
Natural colours.... "	Belgium (4,206), Great Britain (3,491), Holland (1,759), Austria-Hungary (14,215), Russia (8,961).
Painters' colours... "	Hamburg (713), Belgium (703), Great Britain (4,086), Austria-Hungary (553), Russia (489), U.S.A. (226).

*Ibid.*—A. R. L.

THE GERMAN CHEMICAL TRADE SINCE 1885.

M. Diezmann. *Chem. Ind.* 1894, 388—401 (conclusion).

The variations of the import values are chiefly due to "chemical products not specified," and when excluding these the total import values vary between 22—24·25 million m. One-third of this amount falls on *sulphate of ammonia*, the imports of which, notwithstanding the increased home-production, remain fairly constant at 31,000—35,000 tons, an exception being made in 1893 with 42,600 tons. The chief importing countries are England (two-thirds of the total), Austria, France, &c. The exports reached a maximum of 957 tons in 1891. The average values varied between 22—25 m. per 100 kilos. As regards *iodine*, it is kept separate from potassium iodide and other iodine preparations only since 1888. From this date onward the imports of the latter amounted to 1—6 tons annually; hence the imports of iodine may be assumed as having increased from 70 tons to 157 tons between 1885 and 1889. In 1890 they amounted to 138 tons; 1891, 156 tons; 1892, 169 tons; 1893, 324 tons. The exports of iodine preparations, including iodine, rose from 34 to 74 tons in 1889, and amounted since then to 100—110 tons yearly. They are taken up chiefly by Russia, Austria, England, France, and Japan. The average value of the imported iodine was taken at 2,200 m. per 100 kilos. in 1889, increasing to 2,650 m., and that of the exported iodine preparations also to 2,200 m., and since then to 2,270 m.

*Phosphorus*, not being produced in Germany, the whole of the exports of this duty-free article is in reality transit, and the home consumption is determined by deducting exports from imports. The consumption is thus shown to be—

—	1885.	1886.	1887.	1888.	1889.	1890.	1891.	1892.	1893.
Tons	92	90	208	98	163	142	138	166	176

In the year 1887 the unusual amount of 104 tons is supposed to have arrived from Holland. This may be due to an error of classification. The seeming increase of consumption since 1889 may be due to the manufacture of phosphor bronze. The chief importing country is England, and small quantities arrive from France and Switzerland. The exports are chiefly to Austria and Japan. The average values receded from 500 m. per 100 kilos. in 1885 to 320 m. in 1892, and increased to 420 m. in 1893. The figures referring to the traffic in *borax* and *boric acid* are not easily understood, perhaps owing to the circumstance that raw material and final product are not separated. *Chlorate of potash* and *chlorate of soda* not in capsules, notwithstanding the increase of production in England and in Switzerland, belong to the goods which experienced a strong increase of exports—from 94 tons in 1885 to 682 tons in 1892, and to 850 tons in 1893. The increase of consumption falls on Belgium, the States (with strong variations), England, Austria, and Sweden. The imports from England, France, Austria, and, since 1890, from Switzerland, which, between 1885 and 1889, varied between 730 and 920 tons, decreased in the years 1890—1892 to 550 tons annually, and reached in 1893 again 764 tons. The import values fell chiefly in consequence of increased English competition and of the electrolytic production in Switzerland, from 125 m. per 100 kilos. in 1885, without interruption, to 90 m. in 1890, increasing in 1891 to 95 m., 1895 to 125 m., and 1893 to 135 m. For exports, the values are estimated 10 m. higher, with the exception of 1893 (5 m. higher).

The trade in *chromate of potash* was subject to great variations even before 1885. The imports, chiefly from England, which amounted in 1884 to 3,470 tons, fell in 1885 suddenly to 864 tons, rising up to 1,759 tons, decreasing in 1892 to 796 tons, and to 734 tons in 1893. The exports moved since 1885 between 107 and 276 tons. They are directed chiefly to Austria, Russia, &c. On the other hand, the exports of *chromate of soda* show an uninterrupted increase since 1888, especially for France, amounting to 345 tons in 1889, 686 tons in 1892, and 708 tons in 1893;

whilst the imports show a complete falling off (from about 800 tons round 1889 to 138 tons in 1893). Chromate of soda seems more and more to take the place of the potash salt. Prices were greatly influenced by the arrangements of the German and English producers, varying since 1885, for imports as well as for exports, between 61 and 85 marks per 100 kilos, for chromate of potash, and 50—68 marks for chromate of soda.

The imports of *lime salts*, such as the artificial carbonate and sulphate of acetate, phenolate, citrate, &c., rose from 1,232 tons in 1885 to 7,422 tons in 1892 and 6,184 tons in 1893, chiefly owing to consignments of American origin. The average values decreased gradually from 40 marks per 100 kilos, in 1885 to 20 marks.

Among potash compounds *potassium chloride* is to be mentioned in the first instance. The variations in the consumption of this article, including the year 1893, are given below:—

	Tons.
United States .....	23,284 to 38,107
England .....	8,532 to 14,891
France .....	8,749 to 12,310
Belgium .....	7,131 to 7,903
All other countries .....	11,175 to 16,296

Since 1885 the value of potassium chloride is estimated at 14 m. per 100 kilos., with the exception of the years 1892 and 1893, at 14·25 m.

The chief consumers for potassium sulphate (including potassium-magnesium-sulphate) are the States, as by the following statement, embracing the years 1885 and 1893:—

	Tons.
United States .....	6,580 to 19,257
England .....	1,879 to 5,735
Sweden .....	2,047 to 6,933
France .....	945 to 2,226
All other countries .....	1,655 to 2,068

The great increase in the American exports is due to the exemption from duty by the McKinley tariff. The average value amounted in 1885 to 10 m. per 100 kilos.; 1886, 7 m.; 1887, 9 m.; 1888 to 1891, 8·5 m.; 1892, 13·25 m.; and 1893, 13 m.

The chief consumer of *sulphate* is England, taking since 1889 annually between 3,368 and 5,349 tons, out of a total of 8,100 to 10,100 tons. The values, both for imports and exports, vary since 1885 between 38 and 42 m. per 100 kilos.

A remarkable feature in the *potash* trade is the increase of the exports to the States (from 243 tons in 1889 to 2,154 tons in 1893), and the gradual ascension of Austria over France in the imports; both countries export chiefly molasses. The export values were estimated at 35 m. in 1885, 34 m. in 1886 per 100 kilos., increasing gradually to 41 m. The import values are less by 2 m. up to 1888, and by 4 m. since then.

*Prussiate of Potash* was in request in 1892, in which year the English demand rose to 444 tons, only to decrease to 96 tons in 1893, and the total exports to 219 tons. There was an unprecedented demand for *potassium cyanide*, chiefly used for extracting gold. The exports rose uninterruptedly from 17 tons in 1885 to 164 tons in 1892, and 720 tons (value 2,087,000 m.) in 1893.

The considerable progress made in the German soda industry is evinced chiefly by the decrease of imports and the increase of exports. Thus the imports of *soda ash* amounted at the beginning of the last decade to 8,000—9,000 tons annually; they are now reduced to a few hundred tons. But the exports rose from 2,000—3,000 tons to more than 30,000 tons. Chief consumers are Switzerland, Italy, Sweden and Norway, Belgium, Holland, and, since 1890, in increasing measure, the United States. The Austrian and Russian markets, however, are almost completely lost. The estimated values per 100 kilos. were: 11 m. in 1885, 8·5 m. in 1888, and 12·5 m. in 1893. Similarly the imports of *caustic soda*, amounting to 8,000 tons in 1880, 3,260 tons in 1885, receded gradually to 400 tons; whilst the exports, which consisted of 240 tons in 1880, 1,100 tons in 1885, and 1,800 tons in 1890, suddenly increased to 3,190 tons in 1891

and 5,820 tons in 1892, and falling back somewhat to 4,910 tons in 1893. The increased exports of the last few years were taken up chiefly by Belgium, Russia, and Spain, and the decreased consumption of the two last named countries caused the falling back noticed in 1893. The export value of caustic soda amounted to 25 m. per 100 kilos. in 1885, 22 m. in 1888, and 27 m. in the last three years.

The exports of *saltcake* amounted to 15,000 tons in 1885 and 37,100 tons in 1893, the chief consumers being Belgium and Austria. The average values per 100 kilos, were 5 m. in 1885, 3·5 m. in 1889, 4·5 m. in 1890, and 3·5 m. in 1893.

The imports of *ammonium carbonate*, including sal ammoniac and liquor ammoniac, are chiefly of English origin, and remain since 1889 at between 1,360 and 1,590 tons. The exports again rose, almost without exception, to 3,094 tons in 1893, a demand being created by the United States in 1892 and by Russia in 1893. The average values for imports remained between 60—75 m. per 100 kilos.; for exports they are fixed at 5—13 m. less.

There is a continual increase in the exports of *alum*, including sulphate, acetate, palmitate of alumina, &c., especially for England, the United States, Belgium, Switzerland, and Holland. Estimated value up to 1890: 15 m., then falling to 13 m., and reaching 10 m. per 100 kilos. in 1893.

The exports of *copper sulphate* vary as a rule from 2,100 to 2,300 tons, reaching 3,009 tons in 1889 and 4,323 tons in 1890, chiefly owing to its extensive use against the phylloxera; this demand fell off in 1891, and with it the exports to 1,368 tons. About 300 tons are sent for amalgamating purposes to Mexico and Chili. The imports from England and Austria amount, as a rule, to 400—600 tons. Prices varied from 35 marks per 100 kilos, in 1885, to 29 marks in 1887, 40 marks in 1888, 46 marks in 1889, 29 marks in 1892, and 31 marks in 1893.

*Sulphuric acid* is imported chiefly from Austria, Holland, and Belgium, in quantities varying from 8,708 tons in 1888, 6,712 tons in 1889, 8,801 tons in 1890, to 5,747 tons in 1893. The exports amount of late years to 18—20,000 tons, and are taken up in Holland, Belgium, France, Switzerland, and Argentina. The average values fluctuate between 6·5 and 8 marks per 100 kilos.

Considerable variations are noticed in the exports of *tartaric acid*, amounting to 1,056 tons in 1885, 1,598 tons in 1889, 1,021 tons in 1890, 777 tons in 1891, and about 1,160 tons in the next two years. The chief consumer is England, with 500—800 tons; next follow Russia and France. The average values receded from 330 marks per 100 kilos. in 1886 to 210 marks in 1893.

*Salicylic acid* is exported in steadily increasing quantities to the United States, England, and Japan (for conserving "saki"), but prices fell, owing to the expiration of the patents, from 1,300 marks per 100 kilos. in 1889 to 900 marks in 1893.

An upward movement is experienced in the exports of *tannic acid*, chiefly owing to the demand for England, Russia, Austria, and France. The values show a decrease from 300 marks per 100 kilos. to 240 marks.

The trade returns of the class here spoken of were greatly influenced by *manufactured products and preparations, not specified, of the chemical industry and pharmacy*. The imports rose almost without break from 1,371 tons in 1885 to 5,938 tons in 1892, falling back to 3,177 tons in 1893. The average values were 150 marks per 100 kilos. The exports show a continuous increase from 3,550 tons in 1885 to 10,525 tons in 1893, the average values being 250 marks per 100 kilos.

The values of the *essences* (scented waters and fats, and perfumes not named) vary considerably for each year, both for imports and exports. The chief importing countries are Austria and France, and lately China, whilst the exports are taken up in the United States, Austria, Russia, France, Spain, England, and Argentina.

*Mineral waters* are exported chiefly to Holland (for Java) and to Belgium. Average values: 28 marks per 100 kilos. for imports, 19 marks for exports.

## II. B.—ETHERS, ESSENCES, MEDICINES, AND PERFUMES.

	Imports.		Exports.	
	Tons.	Thousand Marks.	Tons.	Thousand Marks.
Essences.....	190 up to 252	3,684 up to 5,172	197 up to 365	2,968 up to 6,208
Mineral waters.....	4,813 „ 6,900	1,361 „ 1,582	25,331 „ 36,383	4,813 „ 4,909
Quinine.....	6 „ 41	1,287 „ 1,416	104 „ 226	5,680 „ 9,326
Other alkaloids.....	7 „ 33	1,900 „ 2,345	10 „ 66	1,078 „ 6,066
Sap, scented.....	75 „ 107	1,150 „ 1,215	1,168 „ 1,370	1,753 „ 2,055
Perfumes.....	187 „ 263	1,191 „ 1,608	1,077 „ 1,371	5,989 „ 6,674
Others.....	512 „ 770	1,216 „ 1,507	573 „ 814	863 „ 1,376
Total.....	6,954 „ 6,932	9,270 „ 12,345	28,778 „ 40,235	26,575 „ 33,168

The exports of *quinine* rose gradually from 104 tons to 226 tons in 1892, falling back suddenly to 135 tons in 1893, chiefly owing to decreased demand for the United States, England, and Holland. The values fell uninterruptedly from 9,000 marks per 100 kilos. to 2,800 marks in 1892 and 1894.

The export values of *other alkaloids* enjoyed a more rapid increase than that of any other chemical industry, notwithstanding a decrease in the average values from 11,000 marks per 100 kilos. to 9,000 marks in 1893. The imports and their average value suffer considerable variations in the individual years, due to variations in a cheap Russian alkaloid (900 marks per 100 kilos.) and to the high price of cocaine (55,000 marks per 100 kilos.).

The imports (chiefly from England and France) and exports of *scented and shaped soaps* show a gradual increase, the latter reaching a maximum of 1,612 tons in 1893. The average values per 100 kilos. are 200 marks for imports, 150 marks for exports.

Among *perfumes* the scented waters occupy the most important position, increasing in value from 3·8 million marks to 5·63 million marks in 1893; the imports are valued at 1,000 marks up to 1889 and 950 marks since then; the exports at 500 marks per 100 kilos.

In *other perfumes* the important position attained by German West Africa has to be noticed (72 tons out of 288 tons in 1893). The import values are 500 marks, the export values 400 marks per 100 kilos.

## II. C.—RESIN OILS, LACQUERS, AND GLUE.

	Imports.		Exports.	
	Tons.	Thousand Marks.	Tons.	Thousand Marks.
Turpentine oil.....	9,816 up to 15,823	4,908 up to 7,524	1,110 up to 1,658	540 up to 895
Lacquers and varnishes.....	573 „ 678	1,431 „ 1,695	559 „ 750	1,050 „ 1,462
Glue.....	1,681 „ 2,085	354 „ 1,299	3,060 „ 4,042	2,976 „ 3,840
Dextrine and.....	129 „ 265	49 „ 119	4,361 „ 9,453	1,211 „ 2,930
Gelatine.....	56 „ 114	112 „ 571	389 „ 502	1,134 „ 1,277
Other materials.....	367 „ 446	430 „ 792	1,275 „ 1,849	728 „ 1,116
Total.....	12,879 „ 19,135	8,654 „ 11,006	11,611 „ 16,904	8,512 „ 16,123

The imports of *turpentine oil* are on a steady increase, three-fifths of the total amount coming direct from the United States and a considerable amount from Russian Poland. The exports are directed chiefly to Austria and Switzerland. The import values amounted to 50 marks in 1885, 58 marks in 1888, and 437 marks in 1893. The export values between the years 1887 and 1892 were 7—12 marks below the former.

One half of the imports of *lacquers and varnishes* are derived from England; the remainder from Holland, the States, France, &c. Since 1891 the exports remain fairly stationary at 550—600 tons, divided among a number of countries. The imports are valued at between 220—250 marks per 100 kilos., the exports at 165—290 marks.

*Glue* lost the Austrian and Dutch markets, but found compensation in England, the States, and English North America; the prices, however, receded from 100 marks per

100 kilos. to 70 marks in 1893. The imports are valued at 25 marks less (1893: 15 marks), and reached a maximum in 1890, 1,734 tons in 1892, and 1,958 tons in 1893.

Owing to the McKinley tariff and the high price of potatoes the exports of *dextrine* and *gelatine*, which rose from 4,657 tons to 9,455 tons in 1890, rapidly fell back to 4,361 tons in 1892, but recovered in 1893 to 7,366 tons. The prices varied in accordance, rising from 24 and 32 marks per 100 kilos. (1885—90), to 35 and 37 marks (1891 and 1892), and receding in 1893 to 25 marks.

The exports of *gelatine* lost ground in Holland, Belgium, and Russia, but this was more than compensated by the increased demand for England and the States. There is a simultaneous decrease of imports to 60—75 tons. The average values receded from 310 marks to 220 marks for exports, and from 290 marks to 200 marks per 100 kilos. for imports.

## H. D.—EXPLOSIVES.—MATCHES.

	Imports.		Exports.	
	Tons.	Thousand Marks.	Tons.	Thousand Marks.
Gunpowder .....	284 up to 1,002	68 up to 1,875	2,772 up to 6,431	4,127 up to 10,267
Explosives.....	1 " 600	16 " 950	1,457 " 2,578	2,315 " 4,125
Artillery.....	2 " 1,593	61 " 3,184	582 " 2,374	1,959 " 5,461
Matches .....	167 " 475	109 " 285	1,532 " 2,592	767 " 1,385
Other materials.....	157 " 225	246 " 310	345 " 567	433 " 707
Total .....	655 " 2,831	722 " 4,723	7,689 " 11,944	12,844 " 18,750

When deducting imports due to exceptional circumstances, the total import value of this sub-division fluctuates between 604,000 and 1,062,000 marks.

The chief consumers of German *gunpowder* and *blasting powder* are to be found in the West of Africa. The following are the turning points of the export trade:—

	1885.	1887.	1890.	1892.	1893.
Africa.....Tons	1,089	649	2,050	1,559	1,611
England..... "	1,658	816	554	70	1,016
Others..... "	1,531	1,307	1,707	1,533	
Total..... "	4,278	2,772	4,311	3,162	2,627

Other countries concerned are chiefly China, Bulgaria, and Brazil. The Russian, Dutch, Australian, and Japanese demands have, more or less, ceased. The values receded from 240 marks to 140 marks per 100 kilos.

The exports of *explosives* reached a maximum in 1890; they amounted in 1893 to 1,723 tons. The Transvaal

market is now in French hands, the Cape Colony in English hands. Notwithstanding conventions and trusts, the average values show a decrease from 250 marks per 100 kilos, in 1885 to 160 marks in 1889, and remain now stationary.

The exports of *artillery* were greatly influenced by Government orders. Chinese orders brought them up to 923 tons in 1885; Turkish orders, since 1889, and Chinese in 1892, up to 2,374 tons; Brazilian, Argentine, and Spanish orders up to 3,683 tons. The values were: from 1885 to 1889, 330—340 marks per 100 kilos.; 1890, 255 marks; 1891, 245 marks; 1892, 230 marks; 1893, 275 marks.

The imports of *matches* from Sweden and Austria are on the decrease. The exports amount to one-fourth of the Scandinavian, and have been greatly hampered in the East by the Japanese competition, and in Austria by the high protective duties which came in force in 1888. New markets were opened up in Belgium, Holland, Central America, Hayti, and Brazil. The decrease was most marked in 1888 and 1889, with 1,712 and 1,532 tons respectively. The average values for exports are, per 100 kilos, 55 marks for 1885 and 1886, and since then 50 marks; for imports they are 10 marks higher.

## H. E.—PITCH, TAR, AND TAR DISTILLATES.

	Imports.		Exports.	
	Tons.	Thousand Marks.	Tons.	Thousand Marks.
Pitch .....	2,313 up to 18,113	324 up to 2,536	7,999 up to 14,980	1,280 up to 2,357
Tar.....	29,392 " 36,890	1,617 " 2,868	9,015 " 17,448	496 " 1,221
Light tar oils.....	5,683 " 7,618	2,536 " 6,501	224 " 916	94 " 779
Anthracene .....	4,252 " 6,699	2,224 " 5,359	0 " 25	0 " 12
Carbolic acid .....	698 " 3,530	798 " 5,351	662 " 3,860	777 " 3,560
Wood spirit.....	490 " 937	490 " 1,124	477 " 817	668 " 1,143
Aniline oil .....	276 " 470	413 " 836	1,680 " 4,660	2,548 " 8,366
Other materials.....	2,306 " 5,237	332 " 786	1,213 " 3,445	198 " 524
Total .....	47,893 " 74,434	9,356 " 21,364	25,321 " 6,584	7,240 " 15,600

The commerce in *pitch* is characterised by two phenomena. On the one hand the imports, amounting to not more than 3,500 tons up to 1887, show suddenly a considerable increase, chiefly from England and Holland, thus:—

	1888.	1889.	1890.	1891.	1892.	1893.
Total imports .. Tons	8,319	12,111	18,113	13,819	11,506	27,769
English and Dutch imports.	6,592	10,323	14,695	11,310	9,649	25,02

The increase is most likely due to the utilisation of *pitch* for briquette making. On the other hand, the exports to France show most erratic variations since 1880:—

	1880.	1883.	1885.	1892.	1893.
Exports to France.. Tons	4,437	12,236	1,411	8,323	3,385
Exports to other countries.	6,082	12,108	6,588	6,651	5,793
	10,519	24,434	7,999	14,974	9,178

The exports of *tar* moved from the years 1886-92 between 9,000 and 12,000 tons, increasing to 14,500 tons in 1893, chiefly on Russian orders. The imports rose in 1893 to 39,722 tons.

Pitch was valued since 1885 at 14 marks per 100 kilos, for imports and at 16 marks for exports; tar at 8 marks for imports and 7 marks for exports in 1885, 6·5 marks for both in 1886, 5·5-5·3 marks up to 1892, and as low as 4·5 marks in 1893.

The demand for *light coal-tar oils* of English origin is undoubtedly on the increase, notwithstanding the increased home production in the manufacture of coke. The average values for imports and exports increased gradually from 40 marks in 1885 to 85 marks in 1890-92, with a reduction in 1893 to 55 marks for imports and 45 marks for exports.

The imports of *anthracene*, chiefly from England, reached a maximum in 1891 and suffered a reduction to 5,523 tons in 1893. The average values per 100 kilos, increased gradually from 50 marks in 1886-87 to 80 marks in 1890-93.

*Carbolic acid* was subject to great variations owing to its use for the manufacture of picric acid, which in its turn is required for explosives, and furthermore owing to the prevalence of cholera in 1892. The imports, chiefly from England, fluctuated between 700-800 tons in 1885 and

1887, 1,192 tons in 1888, 2,432 tons in 1889, 1,543 tons in 1891, 3,530 tons in 1892, and 2,572 tons in 1893. The exports kept from the years 1885 to 1891 within 660 and 1,380 tons, increasing in 1892 to 3,860 tons, chiefly on Russian and Austrian account, and falling to 1,820 tons in 1893. The import values per 100 kilos, were 110 marks in 1885, 220 marks in 1889, 115 marks in 1891, 130 marks in 1892, and 75 marks in 1893. The export values were 160 marks in 1885, 180 marks in 1889, 80 marks in 1891, 100 marks in 1892, and 55 marks in 1893.

The imports of *wood spirit* rose from 772 tons in 1892 to 1,581 tons in 1893, chiefly owing to its use for denaturing alcohol. The chief importer is Austria. The average values for imports and exports amounted to 140 marks in 1885, 100 marks in 1886; in the subsequent years 100-130 marks for imports and 15-20 marks more for exports.

The exports of "*aniline oil*, aniline salts, and other not enumerated coal-tar substances," are continually on the increase: for 1893, however, a fall has to be recorded to 4,996 tons, chiefly due to Russia and the States. The average import and export values varied between 150 and 200 marks from 1885-1891, receding to 140 marks in 1892 and 120 marks in 1893.

## II. F.—WRITING AND DRAWING MATERIALS, DYESTUFFS.

	Imports.		Exports.	
	Tons.	Thousand Marks.	Tons.	Thousand Marks.
Indigo .....	1,266 up to 2,008	12,658 up to 25,502	49 up to 744	5,832 up to 8,374
Dye-wood extracts .....	4,327 .. 5,629	3,462 .. 4,563	1,157 .. 1,683	919 .. 1,498
Catechu .....	5,653 .. 7,350	2,827 .. 4,153	894 .. 2,207	465 .. 1,192
Aniline and other tar colours .....	442 .. 698	3,239 .. 3,890	4,046 .. 10,725	34,816 .. 52,553
Alizarine .....	13 .. 139	21 .. 308	4,284 .. 8,169	7,925 .. 12,906
Lead- and zinc-white .....	2,554 .. 4,262	949 .. 1,932	15,034 .. 24,307	5,574 .. 8,265
Minium .....	303 .. 449	82 .. 146	4,989 .. 6,697	1,847 .. 2,044
Ultramarine .....	43 .. 200	30 .. 48	4,353 .. 5,421	2,525 .. 3,524
Cinnabar .....	14 .. 24	56 .. 143	166 .. 266	663 .. 1,237
Berlin blue .....	26 .. 274	39 .. 298	413 .. 684	660 .. 1,110
Gold preparations .....	0·1 .. 0·6	46 .. 276	5·6 .. 7·5	2,576 .. 3,436
Paints, pigments .....	50 .. 71	53 .. 74	1,021 .. 1,822	1,225 .. 2,186
Lead- and coloured pencils .....	17 .. 115	50 .. 184	655 .. 978	2,292 .. 3,318
Dyestuffs not specified .....	415 .. 244	465 .. 2,170	2,692 .. 5,401	5,549 .. 10,802
Other materials .....	.. ..	.. ..	.. ..	.. ..
Total .....	27,654 .. 34,019	2,9177 .. 42,462	55,263 .. 72,827	81,732 .. 108,022

The home consumption of *indigo* must be characterised as most erratic; thus, when taking the difference between imports and exports, the home consumption for 1892 amounts to 1,124 tons, for 1893 to 681 tons. The direct imports from the East Indies amount to one-third of the total imports. The average values were 1,300 marks per 100 kilos, in 1885, falling uninterruptedly to 950 marks in 1890, and rising to 1,150 marks in 1893.

The chief consumer for *dye-wood extracts* is Austria, with 30 per cent., and in 1893 with 37 per cent. of the total exports. The values are pretty constant at 75 to 84 marks for imports and 80-89 marks per 100 kilos, for exports.

*Catechu* is obtained in increasing measure direct from the East Indies. The variations in the exports are due to the fluctuating Russian demand. The average values up to 1890 were between 49 and 57 marks per 100 kilos, falling to 40 marks in 1893.

*Coal-tar colours* constitute the leading article of export of the German chemical industry, alizarine and aniline dyes alone amounting to 20-26 per cent. of the total.

*Aniline and other coal-tar colours* (exclusive of alizarine and picric acid) show a most remarkable increase of exports, from 2,141 tons in 1880 to 11,560 tons in 1893. On the other hand, the average values decreased from 1,450 marks per 100 kilos, in 1880 to 460 marks in 1893. The chief consumers are the Continent, England, the United States, South and East Asia. The imports, chiefly from Switzerland, are on the increase since 1885.

*Alizarine* had its own vicissitudes, partly owing to the lapse of Gräbe and Liebermann's English patents. The exports to the various countries are shown in the table on next page.

The average values were 350 marks per 100 kilos, in 1880, 222 marks in 1885, falling after the dissolution of the German alizarine syndicate in 1886 to 175 marks, in 1892 to 155 marks, and in 1893 to 145 marks.

The great demand for *picric acid* in 1889 (for explosives) considerably increased the imports and the values; the latter amounted to 235 marks per 100 kilos, in 1885, 350 marks in 1889, and 170 marks in 1893.

*White lead* and *zinc white* are kept separate since 1888. The former is exported chiefly to England and lately to English North America, the total amounting to 11,700—13,700 tons; the imports, chiefly Dutch, fell from 1,100 tons to 700 tons (1893: 932 tons). The exports of zinc white are increasing from about 8,500 tons in 1889 to 12,300 tons in 1893, whilst the imports are stationary at 2,600—3,300 tons.

	1880.	1885.	1889.	1892.	1893.
England .....	3,716	1,910	3,164	2,224	2,727
United States.....	589	877	1,291	1,877	1,234
English East Indies ..	..	..	877	1,585	1,911
France.....	129	139	291	515	228
Other countries ....	1,454	1,328	2,170	1,976	1,966
Total ....	5,888	4,284	7,793	7,677	8,036

*Minium* was in request up to 1887, but since then the exports move between 5,100 and 6,000 tons.

The exports of *ultramarine* are decreasing, and amount to one-half to two-thirds of the total German production. The imports share a similar fate. The import values are estimated at 69 m. per 100 kilos. from 1887 to 1893, the export values to 56—58 m.

*Cinnabar* is consumed chiefly in England, Russia, and Austria. *Berlin blue* in England, Belgium, South-east Asia, and the States. The imports of Berlin blue are on the increase, chiefly on Dutch and Belgian account, but the stuff turns out to be of French origin. Remarkable is the simultaneous fall in the import values from 185 m. per 100 kilos. in 1891 to 80 m. in 1893, whilst the export values for the same period move between 185 and 220 m.

*Gold preparations* are valued at 46,000 m. per 100 kilos. England takes about one-half of the exports.

The exports of *paints and pigments* increased from 1,021 tons in 1888 to 1,354 tons in 1893, chiefly to England, the States, Russia, and South America. The average values are 120 m. per 100 kilos. for exports, 105 m. for imports.

The exports of *black and coloured lead pencils* increased to 1,090 tons in 1893; the destination of one-fourth of this quantity is England. The increase in the imports is due to a reduction in the duty on graphite in compressed tablets, according to the Austrian commercial treaty of 1891. The average import values amounted to 270—300 marks per 100 kilos. up to 1891, and 160 marks in 1892. The export values were 350 marks up to 1889, and receded lately to 300 marks.

In consequence of a change of classification the imports of "dyestuffs, tanning materials, and pigments not enumerated" record a considerable increase, with a corresponding decrease of the values from 90 marks to 80 marks. The exports reached a maximum in 1891 and receded to 4,523 tons in 1893, with an average value of 200 marks per 100 kilos.

On the whole the above is to Tentons a very satisfactory picture of the development of the German chemical industry. The exports of many important products indicate phenomenal progress, and in many instances foreign competition is successfully repelled at home. But in some cases new successes are achieved by foreign goods.—H. A.

#### PHOTOGRAPHIC EXHIBITION AT THE IMPERIAL INSTITUTE.

The Executive Council of the Imperial Institute announce that a special exhibition of photography in its applications to the arts, sciences, and industries will be held at the Imperial Institute during the summer season of 1895. An influential committee of advice has been formed, and sub-committees have been appointed in connection with the seven sections of the exhibition, viz.:—

1. The history of photography, including illustrations of early processes, the progressive development of processes,

the early processes of photomechanical work and modern photographic literature. 2. Artistic photography, with illustrations of the present condition of photographic art in the various colonies and in India. 3. Photography as an industry, demonstrating the apparatus used in photography and the special processes connected with the preparation of lenses, the production of brass fittings, cameras, &c., shown in actual operation; the preparation of dry plates, coating of sensitive media, printing processes also shown in actual operation; reproduction of pictures, and the production of portraits by daylight and artificial light. 4. Photography in its application to industries, such as reproductions having photography as their basis. 5. Applications of photography to the sciences, including orthochromatics, optics, stereoscopy, photomicrography, spectroscopy, metrology and magnetism, astronomy, automatic recording apparatus, &c. 6. Applications of photography to educational purposes. 7. Miscellaneous applications of photography.

Notification of the exhibition will shortly be transmitted to the principal firms engaged in the manufacture of apparatus connected with photography in the United Kingdom. The Governor-General of India, the Governor-General of the Dominion of Canada, and the Governors of the various colonies have, by a recent mail, been requested to invite Indian and Colonial manufacturers to exhibit.—*Chemist and Druggist*.

#### BUYING TURPENTINE BY WEIGHT.

In Canada a system is coming into use of buying turpentine by weight, owing to the losses suffered by retailers through the exudation of the liquid and other causes. It is found that a barrel, supposed to contain 50 gallons, never contains more than 49. The rule for ascertaining by weight the contents of a barrel is, after deducting the tare of barrel from the gross weight, to divide the product by  $8\frac{2}{3}$ , the number of pounds to the imperial gallon, the remainder representing the number of gallons contained therein. Thus—

450 lbs. gross.
75 lbs. tare.
—
$8\frac{2}{3}$ 350
—
41 $\frac{1}{3}$ gallons.

Manifestly the only proper way, the only just way, to buy turpentine is by weight.—*Ironmonger*.

#### INDIA AS A FIELD FOR INDUSTRIAL ENTERPRISE.

The numbers employed in the trades of India are as follows:—There are nearly 2,000,000 oil pressers—showing what an opening there is for a cheap and useful oil mill; on the other hand, of gas and candle makers there are only 1,233—while the use of kerosene oil is spreading enormously—another indication of material progress. There are also:—

	Nos.
Arms and ammunition, blasting powder, fireworks, swords, bows, and arrows.	42,365
Brass and copper .....	105,610
Brick and lime burners, besides those included among potters.	410,142
Earthenware and stoneware, potters, &c. ....	2,346,204
Glass and china .....	11,419
Gums, dyes, and drugs. ....	391,575
Iron and steel makers and sellers .....	1,572,911
Leather tanners, mostly villagers .....	3,285,397
Paper, exclusive of prison labour .....	78,153

AMMUNITION, &c.—The very roughest kind of gunpowder is manufactured all over the country and used for blasting and for fireworks. The necessary ingredients are found everywhere, and improved methods of manufacture should be introduced. That they may be taught the trade is proved by the success of the Government establishment for the manufacture of service powder and cordite.

**EARTHENWARE, &c.**—There is no reason in the world why the skilled potters of Hindustan should not be taught the higher branches of ceramic art. Even such common articles as gallipots are imported.

**LEATHER, &c.**—The Cawnpore factories, where leather goods of all kinds are manufactured, could hold their own anywhere; it seems incredible under these circumstances that any hides should be exported, or leather goods imported, not to mention the horns and bones, which should all be kept in the country for the country's good.

**MASONS, &c.**—Of these only 17,678 are returned as builders. Amongst the stonecutters are reckoned the carvers in steatite or soap stone, also called French chalk. There are large quantities of this stone to be found at the marble rocks near Jabulpore, at Mora in Jaipur, near Tehri, and Orchha in Bundelkand, and in the Kurnal district in Madras, where it is called *bulpum*. These places are easy of access, and the men who have worked in it for generations might be trusted to select the right kind of stone for gas-burners, fire-proof paint, lubricants, and marking-chalk for tailors. German steatite costs quite three times as much as the Indian stone would come to landed in England. Some 50 specimens were sent home some years ago, but nothing came of it.

**Music, &c.**—The musical instrument *par excellence* throughout India is the tun-tum or hand-drum. Square acres of parchment, made of goat or sheep skin, are used for the heads; this parchment should be prepared by machinery.

**PAPERMAKERS, &c.**—The number given is exclusive of prison labour, which turns out a rough article made by hand, much affected by the natives for writing on with their woden styles. The same kind of paper is also made elsewhere; but paper mills are being erected, as will be stated further on. The whole of the 290 tons of paper used in the last Indian census was made near Calcutta. In Gwalior there are also mills, which use the special quality of grass employed for tough paper. Poona and Lucknow have their paper mills too. There is also a project on foot for using bamboos as pulp in Burma. Tenders should be asked for in India by the British Government for blue books and other publications, especially as they claim that they go to the open market for the enormous quantity of paper required.

**TIN, &c.**—Kerosene oil tins are now converted into lamps, water cans, and plates for roofing. But besides the kerosene tins there is an enormous amount of tin which now finds its way into the scrap heap—the tin lining of cases, the empty provision tins, &c. If a process of removing the tin could be introduced, the material is all there ready to hand.

**TOOLS, &c.**—Sugarcane presses are a success; Swedish ploughs are obtaining a footing; but a fortune awaits the man who successfully introduces an oil press or a hand loom that can be manufactured in the country and be repaired by village artisans. Some very fair cutlery is turned out in some parts.

**FUEL.**—The fuel consumed on the railways was, in tons:—

Coal, English .....	178,656	Indian .....	919,943	Coke .....	1,957	Patent fuel.....	6,135	Wood .....	358,756
As compared with previous year...decrease	25,711	Increase .....	153,801	Decrease .....	1,541	Increase .....	1,996	Increase .....	55,916
Percentage .....	12.58	" .....	6.21	" .....	48.22	" .....	48.22	" .....	18.46

As compared with previous years the total consumption of coal increased 2.62 per cent. The consumption per 1,000 tons gross miles varies from 127.13 lbs. on the Bombay, Baroda, and Central India, to 286.82 lbs. on the South Indian; and the average cost of fuel per ton, from Rs. 1.86 on the East Indian, which uses Indian coal exclusively from its own collieries, to Rs. 4.89 on the North-Western, where the average lead for the coal varies from 140 miles for the very indifferent coal found on the line, to 255 miles for Bengal coal, and where large quantities of wood fuel are used, amounting to 30 per cent. of the whole. This wood has an average lead of only 50 miles, but the supply is gradually diminishing.

There was actually less coal got in India in 1893 than in 1892, as the following figures will show, the decrease being entirely due to restricted output in Bengal. The total output in 1880 was 1,019,793 tons.

coal resources of the country, and, if the colliery labour is interfered with, another obstacle will be put in the way of progress.

Most of the imported coal is English, but some has been sent from Australia and a small quantity from Japan. Briquettes are not manufactured or imported in any large quantity, but there is a good market for them, both for machinery and for domestic use.

With regard to petroleum, only the figures for 1892 are available, and are as under:—

—	Assam.	Baluchistan.	Burma.	Punjab.	Total.
	Gallons.	Gallons.	Gallons.	Gallons.	Gallons.
1891	23,000	138,000	5,793,000	2,000	6,136,000
1892	22,000	3,000	8,098,000	2,000	8,725,000

The Yenangyaung field in Upper Burma has been worked for eighteen years by the Burma Oil Company, which has a refinery at Rangoon, and a sufficiency of machinery and skilled workmen; the increase in the Burmese figures is almost entirely due to their operations. The Assam oil field is valuable, and when the railway reaches the tract an enormous increase in the yield may be expected. Locomotives should be so designed as to use either coal or crude oil, or a mixture of both. The slack coal in the Punjab and Baluchistan, which at present breaks the hearts of the engine drivers, should be made into briquettes. Meanwhile the imports of petroleum are also increasing enormously, and will continue to do so as tank steamers and tank waggons increase. Already the earth oil is displacing vegetable oil in even the remotest villages in the land; but a cheap safe lamp is wanted and wick manufactories should spring up everywhere.

—	Assam.	Baluchistan.	Bengal.	Burma.	Central India.
	Tons.	Tons.	Tons.	Tons.	Tons.
1892	164,050	13,284	1,920,050	3,670	88,623
1893	164,420	20,094	1,870,720	9,938	91,318

—	Central Provinces.	Madras.	Nizam's Territory.	Punjab.	Total.
	Tons.	Tons.	Tons.	Tons.	Tons.
1892	132,005	61	119,601	66,352	2,537,006
1893	135,115	502	157,421	77,294	2,529,855

Cost of transport, for want of rail or tramways, is the greatest impediment to the development of the enormous

## Imports of Petroleum.

From	1883-84.	1891-92.	1892-93.
	Gallons.	Gallons.	Gallons.
United States.....	12,714,697	27,291,000	26,265,000
Russia.....	..	28,334,000	38,087,000
Elsewhere.....	1,109,141	2,734,000	2,481,000
Total.....	13,883,838	58,069,000	67,096,000

Iron ore of surpassing richness is found in the Central Provinces in Madras and in other parts, but is not worked to any large extent, except at Barrakur, in Bengal, about 150 miles from Calcutta, near the East Indian Railway. Here are found coal, iron, and lime close to the works, and yet it has taken 20 years to make them a success. The company turned out last year 10,017 tons of pig iron and 3,970 of castings, and sold 12,917 tons of pig and 4,253 of castings; this being the first year in which a market has been found for all the output of the year. "The capital expenditure of the company up to September 1892, was Rs. 185,000. The total profit on the working of the year in India came to 10½ per cent. on the total capital, against 5 per cent. in the preceding year."

## Imports into United Kingdom from India in 1892.

Article.	Value.	Remarks.
	£	
Caoutchouc.....	108,701	Rising
Dyes.....	1,192,821	Risen
Hides, dry.....	392,327	Falling
Leather, undressed.....	2,151,943	Rising
Tin.....	2,081,831	"
Oil, cocoanut.....	191,380	Falling
Seeds, cotton, linseed.....	2,812,408	Fallen
Sugar, unrefined.....	355,472	"

## Exports from United Kingdom into India in 1892.

Article.	Value.	Remarks.
	£	
Beer and ale.....	243,743	Risen
Painter's colours.....	134,463	Falling
Paper, other than hangings.....	175,905	"
Spirits.....	179,487	Risen

Note.—"Risen" and "Fallen" indicate that the figures have changed from the year before: "Rising" and "Falling," that the change has been going on for some time.

The export trade to the United Kingdom does not keep pace with the increasing export trade as a whole; and the direct shipments to the continent of Europe, especially to Belgium and Germany, are constantly increasing.

## Indian Production and Consumption.

CHEMICALS.—Increasing largely. There is a good field open for local manufacturers of aniline and alizarine dyes, the imports of which have doubled in the last five years. Out of 94,782,000 ounces landed last year, 84,318,000 came from the Continent. They are replacing the indigenous dyes, and are said to be preferable to them for common use.

COPPER.—Imports depend on prices and on good harvests, when more copper utensils are made. If the cost of these

could be cheapened the sale would be enormously increased. Belgium is sending yellow sheathing in increasing quantities.

DYES (see Chemicals also), 1890-9.—The normal area under cultivation is about 1,100,000 acres; 50 per cent. in Lower Bengal, and about half and half of the remainder in the North-West Provinces and Madras.

GLASS.—India possesses only one glass factory, and this is in Calcutta. There are a few smaller glass factories, but when they do not use broken imported glass, they turn out goods of an inferior quality. Most of the broken glass that reaches the ports of India is sent to China, where it is worked up by the Chinese glass-workers. Good glass-making materials are to be found in India, and a factory for the manufacture of soda-water bottles alone would find occupation for a large number of operatives. Window-glass is now largely used throughout India where only shutters were used heretofore. It is obtained principally from Belgium, but English soda-water bottles, either from the superior material used in their manufacture, or on account of more careful testing, are found to be better than those of other countries.

PAPER.—Nine mills, of which four are in Bombay, turned out 26,834,692 lbs. of paper in 1891, an increase of 118 per cent. since 1885; ten years ago only 7½ million lbs. were made. The quality of the paper has improved of late years, and there is a large and increasing sale for it. Government professes to encourage local enterprise in every way, and yet they do not get their 127½ million railway tickets in India. Were they to do so, it would keep one mill going for six months. The materials used are: rags, babui and moonj grasses, rice straw, jute and hemp cuttings, old jute bags and cloth.

SALT.—The imports into Bengal and Burma are steadily declining, while the local consumption is increasing; it was 31,355,910 maunds in 1888-89, and 35,051,391 maunds in 1892-93. This is almost entirely due to the extension of railways, as the price of salt is mainly affected by the cost of transport. An increase of consumption is also an indication of greater prosperity. This price of Liverpool salt declined from Rs. 96 per 100 maunds in 1883-84, to Rs. 61·8 in 1892-93, but is rising again.

Imports from	1888-89.	1892-93.	Remarks.
	Tons.	Tons.	
United Kingdom.....	337,087	241,061	Lowest, 222,260 in 1891-92.
Germany.....	7,711	47,391	Highest, 103,403 in 1891-92.
Arabia.....	20,129	25,887	Lowest, 9,840 in 1891-92.
Aden (lowest) ..	17,397	37,282	(Highest.)
Other countries ..	16,484	8,421	Lowest, 4,481 in 1891-92.
Total.....	398,810	359,042	

The following manufactures could also be produced in India, the raw materials and skilled labour being available:—Explosives, earthenware and porcelain, fireworks, glass, leather goods, matches, oil- and floor-cloth, paints and painter's materials, paper and pasteboard, and soap.

## Indian Exports for 1892-93.

Articles.	Rs.	Remarks.
Dyes other than indigo.....	802,013	Rising.
Hides and skins.....	5,591,934	"
Indigo.....	4,141,179	"
Lac.....	784,959	Fluctuates.
Oils.....	595,568	Rising.
Opium.....	9,253,013	Falling.
Sugar.....	597,014	Fluctuates.



*Indian Re-exports in 1892-93.*

Articles.	Rx.	Remarks.
Cum. ds. ....	76,682	Rising.
Copper .....	133,338	"
Glass .....	43,175	"
Gums and resins .....	67,208	Falling.
Sugar (refined) .....	328,980	Rising.

**EARTHNUTS.**—About 40 per cent. of the whole trade is exported from the French Indian ports, being mostly grown in the neighbouring British territory, and from Bombay and Madras. This nut is also called the ground or pea-nut, being *Arachis hypogea*. An average crop yields about 50 bushels per acre. In Europe it is used as a substitute for salad oil, in the manufacture of soap and of lubricants; also as lamp oil and for dressing cloth, and as a constituent of margarine; the consumption being 100,000 tons a year. Formerly the oil was prepared in India, but now the nuts are chiefly sent to Barcelona, Marseilles, and Genoa. It is eaten in India and also largely in the United States, where, however, the Indian product does not find favour for that purpose. The amount exported in 1892-93 was 1,525,000 cwt., having been practically stationary during the last three years. However, the cultivation is extending in Madras. It is extensively used in Marseilles, where the husked nut, as received from India, is ground for soap-making by rather rude machinery. The most modern cotton-seed mills would be most suitable for extracting the oil, with a little additional care, due to the difference in value of the raw product. A steam oil-mill was started in Pondicherry in 1890-92, but from various causes was not a success. There is no reason why this industry should not be successful in India, especially as the by-products are of distinct value. Forty-eight pounds of husked nuts are sufficient wherewith to sow an acre. In the Bombay Presidency a good crop usually yields about 4,800 lbs. of unhusked nuts per acre; the average crop on good land being about 3,500 lbs. The yield of oil in Bombay is often as much as 50 per cent. of the weight of milled seeds; in Madras it is 43 per cent., and in Pondicherry 38 per cent. By cold pressing the quality of the oil is improved, by heat the quantity. The shelled nuts are sent to Europe, the pods to Burmah and the Straits Settlements, oil to these places and Mauritius, and oil-cake to London, Ceylon, and the East. With ground nut-oil, ghee and pure gingelly oil is adulterated. In Southern India the exportation of these nuts is most extensive, and nine-tenths of it is monopolised by the natives. The following figures for 1891-92 will show the proportion raised in each district and the destination of the exported nut.

From	Cwt.	To	Cwt.
Bengal.....	84	United Kingdom....	45,069
Bombay .....	1,041,987	Belgium .....	98,271
Sand .....	138	France.....	1,185,358
Madras .....	187,668	Germany .....	8,974
Burma.....	63	Italy.....	74,845
		Egypt.....	93,097
		Others .....	5,547

**CHEMICALS.**—The exportation of cinchona bark was 3,074,000 lbs. in 1888-89, 2,643,000 lbs. in 1891-92, and 2,814,000 lbs. in 1892-93, besides what was consumed locally. There are large Government plantations in the Darjeeling district, containing nearly  $4\frac{1}{2}$  million cinchona trees, the harvest of the year being 304,000. In similar Government plantations in the Nilgiri Hills the harvest was 119,000 lbs.

**COCONUT PALMS.**—The export of the products of these amounted to Rx. 486,688 from 1891 to 1893. Out of the fibre, or *coir*, is made matting, cordage, rugs, and brooms; of the *oil*, candles and soap are manufactured; of the *oilcake*, manure and food; and of the *toddy*, sugar and spirits. The local consumption of these articles is enormous.

**HIDES.**—There is an increased demand for tanning purposes in India, and also for goat-skins, of which the United States take three-quarters. Gloves and parchment should be manufactured wholesale in India.

**Tanned Hides and Skins** have risen since 1889-90 from 260,716 cwt. and Rx. 2,521,008 to 298,930 cwt. and Rx. 2,956,439. The trade is now established on a sound basis, and should increase enormously in the near future. In Japan, for instance, the natives are producing quantities of fine leather for travelling bags, &c.

**MANURES.**—17,584 tons of bones were exported, which should have been ground and used in the country; also 1,032 tons of other manures, which should also have been used locally.

**OILS.**—If the wealth of oil-producing materials be taken into account, it is passing strange that so little oil is exported. "Oil has been expressed for many centuries by the 'ghanee,' a mortar having a revolving pestle driven by hand or bullock power. The residual cake contains a large quantity of oil, and is fed to the cattle. This mill is in general use, excepting where Europeans have a hand in the production—European machinery, of course, giving better results. Until six or seven years ago, vegetable oils were almost exclusively used for every kind of lubrication in India." A similar mill to that introduced for sugar (which see) would be equally successful.

**OPIMUM.**—75,384 chests were made in 1892-93. The following figures tend to show that, where the opium shops are numerous, there the liquor shops are few, and *vice versa*.

Number of Retail Shops Licensed in 1892-93 for the Sale of	Bengal.	N.W.P.	Punjab.	Burma.	C.P.
Spirits .....	20,887	11,437	1,747	1,413	7,589
Opium and other narcotics.	4,670	4,912	3,401	34	2,180

Number of Retail Shops Licensed in 1892-93 for the Sale of	Assam.	Madras.	Bombay.	Berar.	All India.
Spirits .....	326	31,422	5,068	2,114	82,063
Opium and other narcotics.	1,138	1,276	2,415	962	20,988

**SEEDS.**—*Castor.*—The seeds are exported in increasing numbers for manure cakes, and for inferior qualities of lused cake. It is not recommended for cattle, being a purgative. There were 989,000 cwt. exported in 1892-93, and the quantities are increasing. The oil is used in India as an illuminant and a medicine, and is exported to the United Kingdom and Australia for lubricants.

*Linseed.*—A marketable article has been produced, as an experiment, in India of this fibre. There are 4,000,000 acres under cultivation, producing 2 cwt. an acre. The maximum area is 5,000,000 acres. The seed is used as food to a limited extent; the quantity crushed for oil is still less, such demand as exists being met by importation. Eight million cwt. of the seed are exported now. The seed yields 30 to 35 per cent. of oil, which is used in the manufacture of paint, floorcloth, oil-varnish, and soft soap—all of which should be made in the exporting country. Pure linseed is unknown; the oilcake is of great importance as a cattle food, for which the Indian seed, being richer than the Russian, is preferred.

**Poppy.**—The seed is used as food in India, and the oil for cooking and lighting. Belgium and France take the bulk of the exports, using the oil for soap, for adulterating olive oil, and for painters' colours. The amount exported in 1892-93 was 502,000 cwt., and is decreasing.

**SODA WATER.**—This is made everywhere; three years ago there were 72 factories; the carbonic acid is employed in the manufacture of spurious sparkling wines. As most of the water used is anything but nice, the taste for this product is quite an acquired one. In Karachi, before the Mulleer water was brought in, it was made of brackish water; there is, therefore, not much likelihood of an export trade springing up.

**SUGAR.**—The export trade, which is carried on mostly from Madras, is struggling for existence—although it should be thriving, if the cultivation of cane were encouraged. At present the production of the country is not sufficient for local consumption; the quantity imported to supplement local supplies was more than double the quantity exported—viz., 836,234 cwt., in 1892-93.—*The Indian Engineer.*

### BOARD OF TRADE RETURNS.

#### SUMMARY OF IMPORTS.

Articles.	Month ending 31st October	
	1893.	1894.
	£	£
Metals.....	1,973,508	1,575,963
Chemicals and dyestuffs.....	470,759	416,482
Oils.....	579,112	733,159
Raw materials for non-textile industries.....	4,459,104	4,536,262
Total value of all imports....	35,357,297	35,068,385

#### SUMMARY OF EXPORTS.

Articles.	Month ending 31st October	
	1893.	1894.
	£	£
Metals (other than machinery) ....	2,591,241	2,505,735
Chemicals and medicines .....	644,944	725,772
Miscellaneous articles.....	2,399,247	2,617,506
Total value of all exports.....	18,179,792	19,147,996

#### IMPORTS OF OILS FOR MONTH ENDING 31ST OCTOBER.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Cocoa-nut..... Cwt.	7,040	52,782	8,650	63,921
Olive .....	559	2,588	21,953	86,616
Palm .....	94,542	81,342	116,212	86,474
Petroleum .....	15,957,618	18,498,358	239,424	259,398
Seed .....	2,366	3,546	58,100	76,060
Train, &c.....	1,495	2,616	29,141	43,158
Turpentine .....	38,991	22,542	35,728	22,979
Other articles .. Value £	..	..	69,904	103,550
Total value of oils ...	..	..	579,112	733,159

#### IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDING 31ST OCTOBER.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Bark, Peruvian .. Cwt.	2,435	3,218	4,621	7,287
Bristles.....	292,698	441,477	58,947	68,971
Caoutchouc..... Cwt.	25,666	27,658	272,551	307,184
Gum:—				
Arabic.....	3,173	6,185	8,567	14,444
Lac, &c.....	4,557	5,892	21,525	30,175
Gutta-percha ....	4,883	2,953	15,945	18,828
Hides, raw:—				
Dry.....	50,584	25,332	81,673	59,146
Wet .....	80,262	72,450	168,850	153,455
Ivory.....	2,259	876	108,586	38,456
Manure:—				
Guano..... Tons	968	1,959	6,486	7,506
Bones.....	2,430	8,345	10,129	38,801
Nitrate of soda...	3,639	13,919	32,741	117,226
Phosphate of lime ..	21,441	25,947	40,443	55,050
Paraffin..... Cwt.	59,112	69,134	61,961	69,143
Linen rags..... Tons	1,498	2,719	12,116	24,536
Esparto.....	14,820	15,593	71,758	71,659
Pulp of wood ....	23,630	22,225	126,578	139,402
Rosin..... Cwt.	90,906	49,916	22,217	10,426
Tallow and stearin ..	167,047	174,915	222,965	222,993
Tar .....	9,284	5,810	4,579	4,322
Wood:—				
Hewn .....	189,868	192,726	377,815	387,142
Sawn .....	645,419	683,679	1,494,258	1,493,509
Staves .....	11,619	14,539	43,654	57,982
Mahogany .....	8,756	11,751	77,151	94,110
Other articles.... Value £	..	..	1,399,218	1,077,118
Total value .....	..	..	4,459,104	4,536,262

Besides the above, drugs to the value of 65,675*l.* were imported, as against 55,317*l.* in October 1893.

#### IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH ENDING 31ST OCTOBER.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Alkali..... Cwt.	9,896	15,148	8,806	7,695
Bark (tanners', &c.) ..	32,274	29,280	11,865	12,002
Brimstone.....	51,933	55,265	10,031	11,854
Chemicals..... Value £	..	..	139,301	105,743
Cochineal .....	535	607	3,173	3,635
Cutch and gambier Tons	2,717	1,711	58,518	36,429
Dyes:—				
Aniline .....	..	..	23,735	42,413
Alizarin .....	..	..	23,783	26,492
Other .....	..	..	1,995	3,059

IMPORTS OF CHEMICALS AND DYE-STUFFS FOR MONTH  
ENDING 31ST OCTOBER—*cont.*

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Indigo ..... Cwt.	2,350	666	£ 12,952	£ 7,776
Nitrate of potash . . .	22,142	23,007	19,201	21,019
Valonia ..... Tons	478	855	6,216	10,761
Other articles... Value £	..	..	131,783	127,563
Total value of chemicals	..	..	470,759	416,432

## IMPORTS OF METALS FOR MONTH ENDING 31ST OCTOBER.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Copper:—			£	£
Ore..... Tons	9,826	7,062	50,502	49,116
Regulus ..... "	12,424	4,587	276,586	120,901
Unwrought .... "	6,472	4,667	292,698	180,287
Iron:—				
Ore..... "	256,389	353,321	172,157	225,554
Bolt, bar, &c. .... "	8,956	7,689	80,743	66,830
Steel, unwrought.. "	1,306	411	15,112	3,038
Lead, pig and sheet .. "	16,051	13,254	162,586	130,670
Pyrites ..... "	41,901	39,699	67,747	71,881
Quicksilver..... Lb.	44,500	28,800	3,875	2,225
Silver ore..... Value £	..	..	319,042	184,325
Tin..... Cwt.	65,070	73,883	260,693	236,491
Zinc ..... Tons	4,888	6,499	85,350	104,759
Other articles ... Value £	..	..	186,705	198,852
Total value of metals	..	..	1,973,508	1,575,063

EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING  
31ST OCTOBER

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Alkali..... Cwt.	336,622	645,479	119,399	171,190
Bleaching materials ..	96,890	115,653	39,917	56,201
Chemical manures. Tons	21,881	18,475	163,839	136,914
Medicines..... Value £	..	..	86,418	91,655
Other articles ... ..	..	..	235,461	269,812
Total value .....	..	..	614,944	725,772

EXPORTS OF METALS (OTHER THAN MACHINERY) FOR  
MONTH ENDING 31ST OCTOBER.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Brass..... Cwt.	8,482	8,217	£ 34,027	£ 31,170
Copper:—				
Unwrought..... „	29,870	42,419	69,518	92,868
Wrought..... „	33,557	26,564	92,977	70,715
Mixed metal..... „	21,666	30,575	49,696	61,689
Hardware..... Value £	..	..	167,949	171,589
Implements..... „	..	..	193,035	117,936
Iron and steel..... Tons	243,717	241,270	1,608,708	1,750,126
Lead..... „	4,219	3,828	46,363	43,464
Plated wares... Value £	..	..	32,389	31,605
Telegraph wires „	..	..	216,951	22,292
Tin..... Cwt.	13,301	9,168	56,631	33,861
Zinc..... „	17,392	13,735	13,472	9,426
Other articles .. Value £	..	..	70,398	63,924
Total value .....	..	..	2,591,244	2,505,735

EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH  
ENDING 31ST OCTOBER.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Gunpowder..... Lb.	357,700	375,600	£ 8,524	£ 16,127
Military stores.. Value £	..	..	167,888	164,999
Candles..... Lb.	1,611,300	1,950,800	39,378	33,160
Caoutchouc..... Value £	..	..	95,296	112,017
Cement..... Tons	40,185	33,301	66,247	53,574
Products of coal Value £	..	..	110,350	101,844
Earthenware ... "	..	..	139,148	177,782
Stoneware ..... "	..	..	18,017	9,920
Glass:—				
Plate..... Sq. Ft.	110,085	149,064	5,703	7,979
Flint..... Cwt.	7,551	8,458	20,733	21,469
Bottles..... "	51,965	56,052	26,301	27,149
Other kinds.... "	13,957	19,297	11,090	16,328
Leather:—				
Unwrought .... "	12,159	12,782	112,220	112,252
Wrought ..... Value £	..	..	36,141	32,894
Seed oil..... Tons	4,476	4,115	98,567	87,702
Floorcloth ..... Sq. Yds.	1,344,709	1,597,109	52,877	62,216
Painters' materials Val. £	..	..	112,331	116,184
Paper ..... Cwt.	77,992	81,365	125,438	128,718
Bags..... Tons	3,525	4,946	22,565	31,449
Seep..... Cwt.	48,955	52,764	51,478	57,025
Total value .....	..	..	2,399,247	2,617,506

## Monthly Patent List.

\* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### APPLICATIONS.

19,529. G. C. Douglas.—From W. Don and T. Watson, India. Improvements in non-conducting composition for retarding the flow of heat from, or to, boilers, cylinders, refrigerators, and the like. October 15.

19,691. J. Lyle. Improvements in retorts for heating, drying, or destructive distillation. October 16.

19,753. T. Illingworth. Improvements in apparatus for steaming textile fabrics, also applicable for drying fabrics. Complete Specification. October 17.

19,832. T. Cudlipp. Improved hydro-extractor. October 18.

20,061. J. Mitchell. Improvements in or relating to fastening devices for the doors of gas retorts. October 20.

20,175. O. Perrier. Improvements in processes and apparatus for distillation. Complete Specification. October 22.

20,202. J. Entwisle.—From W. L. Wallis, United States. An improvement in gas purifiers for furnaces. Complete Specification. October 23.

20,480. J. J. Meldrum, T. F. Meldrum, and J. W. Meldrum. Improvements in or connected with the treatment of liquids by gases. October 26.

20,874. A. Bloch. Improvements in apparatus for mixing liquids and gases. October 31.

21,298. F. W. Kent. Improvements in and connected with valves for hot or cold water, steam, or other liquids or gases, and improved methods of operating the same. November 6.

21,326. W. P. Thompson.—From J. Furbringer, Austria. Improvements in muffle furnaces. Complete Specification. November 6.

21,372. B. Abeil. Improvements in evaporating apparatus. November 6.

21,527. F. W. Berk. An improved apparatus for cooling acids or other fluids. November 8.

21,615. J. B. Lee. Improvements relating to the construction of thermometers. November 9.

21,631. E. Orval. Improvements in drying kilns. Filed November 9. Date applied for 9 May 1894, being date of application in Belgium.

21,633. H. H. Lake.—From H. See, United States. Improvements in evaporating apparatus. Complete Specification. November 9.

21,778. I. S. McDougall. Improvements in evaporating-pans for the concentration of sulphuric acid and other liquids. Complete Specification. November 12.

21,946. W. Thomson and P. J. Worsley. Improvements in and connected with furnaces for treating chemicals, ores, or the like. November 13.

22,262. J. Benson. Improvements in apparatus for ascertaining the amount of acidity in liquids. November 17.

#### COMPLETE SPECIFICATIONS ACCEPTED.\*

1893.

21,000. P. D. de la Grée. Improvements in and relating to evaporating apparatus. November 7.

21,761. The Manchester Oxygen (Brin's Patent) Co., Ltd., and W. M. Jackson. Improvements in means for utilising the pressure in cylinders or reservoirs containing compressed gases, in the application and use of such gases. October 31.

1894.

24. J. B. Hilliard. Preventing gases from intermingling while passing through liquids. November 7.

358. J. B. Allott. Improvements in apparatus for drying or evaporating viscous semi-solid or other materials at low temperatures. November 21.

1098. H. H. Lake.—From the Actien Gesellschaft für Kohlensäure-Industrie. An improved cap for cylinders or receptacles for containing liquid or gas under pressure. November 21.

9049. E. A. Fehling and A. Steinbart. Improvements in and relating to processes and apparatus for analysing gases and gaseous mixtures. November 14.

17,289. H. E. Newton.—From E. Hart. See Class VII.

17,314. J. S. Detwiler. Improvements in apparatus for treating powdered materials. November 7.

17,315. J. S. Detwiler. Improvements in apparatus for blending powdered materials. November 7.

18,214. H. H. Lake.—From R. Schicht. An improved method of and apparatus for drying substances which are viscous or semi-fluid in the hot state. October 31.

19,169. G. L. F. Edeline. An improved composition for the prevention of incrustation in boilers. November 14.

19,753. T. Illingworth. Improvements in apparatus for steaming textile fabrics. November 21.

### II.—FUEL, GAS, AND LIGHT.

#### APPLICATIONS.

19,689. W. G. Smith. Improvements in the manufacture of night-lights. October 16.

19,692. J. Lyle. Improvements in the manufacture of solid petroleum fuels. October 16.

19,693. J. Lyle. Improvements in the manufacture of asphaltic compounds. October 16.

19,815. H. Tempest. Improvements in the manufacture of fuel-blocks or briquettes. October 18.

20,066. W. E. Price. Improvements in apparatus for carburetting coal gas or other gaseous fluid. October 20.

20,178. R. Schewe and F. Messedat. A composition for improving petroleum and method of producing such composition. October 22.

20,253. P. C. Tennant and J. E. Hall. Improvements in and relating to apparatus for regulating or controlling the supply of fluids, chiefly designed for use in connection with apparatus for carburetting or enriching gas. October 23.

20,375. F. K. Byerley. Improvements in the manufacture of asphalt and the like from petroleum and in apparatus therefor. October 24.

20,414. R. Fegan. Improvements in the manufacture of briquettes for fuel. October 25.

20,502. W. Sander.—From C. F. Winkelmann, Germany. New or improved fire-lighter. October 26.

20,609. W. B. Fitch. Improvements in candles. October 27.

20,941. W. Ritchie. Improvements in or relating to gas-carburetting apparatus. November 1.

20,998. J. Morrison and T. Booth. An improved apparatus for producing and saving gas. November 2.

21,051. J. Ruscoe. Improvements in apparatus for charging gas retorts. November 2.

21,018. S. W. Borchers. A novel or improved process and means for obtaining electrical energy from fuel gases. Complete specification. November 3.

21,222. T. L. Willson. Improvements in the manufacture, distribution, and use of gas for illumination, heating, and other purposes, and in apparatus therefor. November 5.

21,441. R. Denormandie and H. Hennebute. Improvements in the manufacture of petroleum briquettes or blocks. November 7.

21,655. E. J. Duff. Improvements in gas producers. November 10.

21,658. J. J. Atkins. See Class XVII.

22,034. H. Schon. Improvements in apparatus for the manufacture of coke. November 14.

22,036. W. H. Biggs and R. R. Greenhow. Improvements in the manufacture of artificial fuel. November 14.

22,170. A. E. Tavernier and W. L. Maleolmson. A new household briquette fuel. November 16.

22,184. C. W. Pinkney. Improvements in or connected with apparatus for the manufacture of gas from liquid hydrocarbons. Complete specification. November 16.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

21,926. F. E. Baron and C. C. Fry. Improvements in liquid fuel, and in means or apparatus employed in the manufacture thereof. October 24.

22,169. F. McNamee. Improved fuel for domestic and steam purposes. November 21.

22,378. E. de Pass.—From L. J. Baptiste. Improvements in machines for the manufacture of candles. October 31.

24,422. E. E. Dulier. An improvement in apparatus for destroying smoke. October 24.

24,856. J. Young. Improvements in obtaining cyanides and ammonia direct from the air or other gases containing nitrogen. November 7.

24,955. B. J. B. Mills.—From La Compagnie Internationale pour l'Exploitation des procédés A. Seigle. See Class XII.

1894.

4283. O. Imray.—From La Société Anonyme des Moteurs Thermique Gardie. Process for the manufacture of combustible gas. November 14.

12,888. J. C. Fell.—From M. M. Armstrong. Improvements in the method of burning fuel to obtain complete and smokeless combustion and in the furnaces used therefor. September 26.

16,395. W. F. Browne. Improvements in the manufacture of gas and in apparatus therefor, and in the utilisation of the waste heat produced therein for the generation of steam. November 14.

17,484. E. Kirbis. Method of manufacturing artificial fuel. November 7.

17,679. F. Bremme. Improved mode of treatment and apparatus for the carburisation of gases. November 14.

19,127. J. T. Donovan and H. L. Gardner. A new and improved process for the production of ozone. November 14.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

#### COMPLETE SPECIFICATION ACCEPTED.

1894.

736. J. H. W. Stringfellow. Improvements in the treatment of tar and in obtaining useful products therefrom. November 21.

### IV.—COLOURING MATTERS AND DYES.

#### APPLICATIONS.

19,712. G. W. Johnson.—From The Chemische Fabrik, late Goldenberg, Geromont, and Co., Germany. A process for obtaining lactyl compounds derived from methyl aniline, ethyl-aniline, *p*-anisidine, and *p*-phenetidine. October 16.

19,885. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of colouring matters of the induline series. October 18.

19,970. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of direct dyeing colouring matter. October 19.

19,971. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of direct-dyeing colouring matter. October 19.

19,972. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of direct-dyeing colouring matter. October 19.

20,086. A. Levinstein. Improvements relating to the manufacture of cellulose from the waste produced in the extraction of tanning and dyeing materials. October 20.

21,227. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of stable diazo-compounds in concentrated liquid or solid form. November 5.

21,786. A. G. Green and R. Jansen. Production of dehydrothioniline sulphonic acid and of colouring matters therefrom. November 12.

21,787. A. G. Green and R. Jansen. Production of a new basic yellow colouring matter. November 12.

21,788. A. G. Green and R. Jansen. Improvements in the manufacture of red direct cotton colouring matters. November 12.

22,174. H. S. Wilkinson. Improved dyewood extractor. November 16.

22,273. S. Pitt.—From L. Cassella and Co., Germany. The production of mixed disazo dyestuffs from delta-amidonaphthol. November 17.

22,276. W. W. Horn.—From W. Maybury, United States. Improvements in and relating to thickeners or vehicles for colours, mordants, and the like. November 17.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

20,605. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik. The manufacture and production of new compounds suited for use in the preparation of diazo-compounds and their application in the production of dyes. October 31.

24,365. P. R. J. Willis.—From W. J. Matheson. Improved process for making solid and friable colouring matters from logwood extract and other dyewood extracts. October 24.

24,543. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik. The manufacture of colouring matters and of intermediate products related to the rhodamine series. October 24.

24,714. S. Pitt.—From L. Cassella and Co. The production of azo-dyestuffs derived from  $\alpha_1 \alpha_1$  azimidonaphthalene. October 31.

24,715. S. Pitt.—From L. Cassella and Co. The production of dyestuffs from di-azo safranines and amidonaphthols. October 31.

24,870. C. Dreyfus. The manufacture and production of new colouring matters. October 31.

25,074. H. E. Newton.—From the Farbenfabriken vormals F. Bayer and Co. The manufacture or production of sulpho acids of the naphthalene series derived from naphthalene  $\beta_1 \beta_2$  disulpho acid or naphthalene  $\beta_2 \beta_3$  disulpho acid. November 7.

25,071A. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of  $\beta_1 \beta_1$  dihydroxy-naphthalene  $\beta_1 \beta_1$  disulphonic acid. November 7.

1894.

406. O. Imray. — From The Farbwerke vormals Meister, Lucius, and Brünig. Improvements in the production of sulphurous aromatic bases and of their sulphonic acids. November 14.

515. J. V. Johnson. — From Kalle and Co. Improvements in and relating to the manufacture of azo dyestuffs. November 14.

973. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of anthraquinone derivatives. November 21.

974. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of colouring matters derived from anthraquinone. November 21.

975. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of hydroxyanthraquinones. November 21.

1062. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of dyestuffs. November 21.

1063. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of  $\beta_1$ -naphthol,  $\alpha_1 \beta_1$ -disulpho acid, and  $\beta_1$ -naphthylamine,  $\alpha_1 \beta_1$ -disulpho acid. November 21.

## V.—TEXTILES, COTTON, WOOL, SILK, Etc.

### APPLICATIONS.

19,722. T. Illingworth. Improvements in the process or method of rendering certain woollen and worsted fabrics unspottable, unshrinkable, and impermeable. October 17.

20,033. J. L. Byrom and B. F. Byrom. Improvements in the manufacture of yarns made from wool and other fibre or fibres combined, and in apparatus employed therefor. October 20.

20,121. J. V. Johnson. — From L. F. Gros, France. Improvements in and relating to the manufacture of cotton thread. October 22.

20,122. J. V. Johnson. — From L. F. Gros, France. Improvements in the manufacture of printed woven fabrics. October 22.

20,133. R. M. Harvey. An improvement in the manufacture of inlaid floor-cloth. October 22.

20,341. G. C. Franklin. Improvements in broad silk pieces, and in the manufacture of the same. Complete Specification. October 24.

20,763. T. Burrows and D. E. Radclyffe. Improvements in machines for combing vegetable or animal fibres, such as ramie, flax, wool, or silk waste, and preparing same for spinning. October 30.

21,029. W. G. Thomsen. Improvements in the method of and apparatus for manufacturing inlaid linoleums and other floor-cloths. November 2.

21,519. T. H. Copley and D. Scott. Improvements in the treatment of waste from textile manufactures in order to recover fibres therefrom. November 8.

22,004. T. R. Hanmer. Improvements in finishing textile or woven fabrics. November 14.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

21,060. B. Barraclough. Improvements in certain textile fabrics. October 21.

21,976. W. L. Hays. — From E. Gochley. See Class XII.

21,998. Com. H. de Clavelonnet. Improvements in the manufacture of artificial silk. November 21.

1894.

1089. J. Smith and P. W. Nicolle. Improvements in the treatment of vegetable fibrous matters to obtain fibres therefrom. November 11.

17,625. A. Stephenson. Improvements in apparatus for scouring and washing wool and the like. October 24.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

### APPLICATIONS.

20,308. W. E. Kay and The Thornliebank Co., Ltd. Improvements in and relating to the printing and mangle-creasing of cotton fabrics. October 21.

21,119. T. Lye and W. T. Lye. Improvements in the bleaching or dyeing of "chip," "straw" and "chip plait," and "straw plait." November 3.

21,120. T. Lye and W. T. Lye. Improvements in the printing or colouring of "chip" and "straw" in plait and otherwise, and in means or apparatus employed therein. November 3.

21,287. J. A. O'Loughlin. Improvements in and connected with the production of patterns on indigo-dyed cotton fabrics. November 6.

21,324. F. Scott and R. J. Flintoff. Improvements in the printing of aniline black. November 6.

21,481. W. L. Hiffe, J. J. H. Sturmer, and J. Ward. New or improved method of and means for printing colours and the like on textile fabrics. November 8.

22,221. J. Clapham, W. W. L. Lishman, and M. Jefferson. Improvements in machinery for dyeing and bleaching yarns and fabrics. November 17.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

15,829. O. Imray. — From F. Erbau and L. Specht. Process for dyeing textile material in cops or spools with alizarine and other mordant dyes. October 24.

23,750. M. Williams. Improvements in or appertaining to the bleaching of fibrous or textile materials. November 14.

1894.

98. A. H. Leggif. Improvements in machines or apparatus for applying bronze or other powders to the surface of paper, cardboard, cloth, and such like. November 7.

6887. C. F. Cross and E. J. Bevan. — From C. Bealle. A process for marking indelibly on paper, fabrics, and like receptive material. November 7.

19,226. W. H. Thorpe. Improvements in machinery for mordanting, dyeing, washing, and scouring hanks of yarn. November 14.

19,235. T. Ingham. Improvements in dyeing mixed woven or knitted fabrics. November 14.

## VII.—ACIDS, ALKALIS, AND SALTS.

## APPLICATIONS.

19,584. V. L. Eley.—From The United States Smokeless Powder Co., United States. Preparation of ammonium chromates. Complete Specification. October 16.

19,768. J. E. Riley. Improvements in the process of manufacturing or recovering cyanogen compounds. October 17.

19,769. J. E. Riley. Improvements in the manufacture of sulphuric acid. October 17.

19,770. J. E. Riley. Improvements in the production or manufacture of ammonia. October 17.

19,876. J. Armstrong. Improvements in concentrating and storing acids, alkalis, and other chemicals, and furnaces and apparatus therefor. October 18.

20,168. W. L. Wise.—From Deutsche Solvay-Werke Actien-Gesellschaft, Germany. Improvements in the manufacture or production of chromates. October 22.

20,169. W. L. Wise.—From Deutsche Solvay-Werke Actien-Gesellschaft, Germany. Improvements in the manufacture or production of manganates and stannates. October 22.

20,622. P. Bateson and F. K. Muspratt. Improvements in the manufacture of carbonate of soda. October 27.

20,961. M. Guthrie. Improvements in or relating to the decomposition of alkaline chlorides. November 1.

21,147. J. Y. Johnson.—From N. Basset and W. de Baranoff, France. Improvements in the treatment of alkaline salts or the salts of alkaline earths. November 7.

21,529. E. Andreoli. Production of vinegar by means of ozone. Complete Specification. Filed November 8. Date applied for 20 July 1894, being date of application in Belgium.

21,557. A. Walker. A new process for the purification and utilisation of the chemical by-product known as sulphate of lime sludge. November 9.

21,732. H. Y. Castner. Improvements in the manufacture of alkali cyanides. November 10.

21,774. M. N. d'Andria. Improvements in and connected with the production of caustic soda, caustic potash, and barium and strontium hydroxides. November 12.

21,778. I. S. McDougall. See Class I.

21,945. W. Thomson and B. J. Worsley. Improvements in apparatus for use in the decomposition of common salt, in the production of sulphate of soda, or for analogous purposes. November 13.

21,962. W. Wyld and J. H. Cox. A new or improved method of manufacturing persulphate of iron or ferric sulphate. November 14.

21,963. W. Wyld and J. H. Cox. A new or improved method of manufacturing ferric sulphate. November 14.

22,028. L. Gautreau, E. Charbonnier, and E. Serrant. Process of manufacture and employment of humic acid and its compounds. November 14.

22,297. H. H. Lake.—From C. Piejer and C. Fellner, Germany. Improvements in and relating to the manufacture of ammonia and by-products. November 16.

22,251. J. J. Cresfield and G. S. Johnson. New or improved crystalline salts containing soluble compound silicates and method of manufacturing the same. November 17.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

23,889. G. Jarman. Improvements in or appertaining to apparatus for drying bicarbonate of soda. November 21.

23,890. G. Jarman. Improvements in the manufacture of refined bicarbonate of soda. November 21.

24,175. E. J. Hunt. Improvements in treating the residual products of certain manufactures, and in treating certain metallic ores for the manufacture of chlorine. November 14.

24,856. J. Young. See Class II.

25,024. H. Gaskell, jun., V. C. Driffield, A. Carey, and F. W. Wright. Improvements in and connected with the manufacture of bleaching powder. October 31.

25,024. H. Gaskell, jun., and A. Carey. Improvements in or relating to the manufacture of chlorine. October 31.

1894.

60. J. Werlein. An improved method or process of hardening aluminous materials and the product obtained thereby. November 7.

11,905. E. Driesel and J. Lenhoff. Process for the manufacture of neutral sodium carbonate, neutral sodium sulphite, and sodium borates from sodium chloride. November 21.

17,289. H. E. Newton.—From E. Hart. Improved apparatus for the condensation of nitric acid and other vapours. October 24.

17,556. J. Ascongh. Improvements in the manufacture of sodium bichlorate or borax. October 24.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

## APPLICATIONS.

20,050. J. W. S. Holt and S. Hargreaves. Improvements in or relating to the manufacture of crucibles, fire-clay, refractory, and non-conducting bricks and blocks, furnace linings, or the like. October 20.

20,113. H. Cartledge. Improved construction of potters' kilns. October 22.

20,138. E. A. Shaw. Improvements in the manufacture of pottery or like ware. October 22.

20,156. E. W. Crebbin. Improvements in crucibles, "firebrick" articles, and fireproof or refractory composition for lining furnaces and the like. October 22.

20,287. J. Slater and A. Doulton. Improved methods of preparing "transfers" for use in decorating ceramic-ware. October 24.

20,557. W. G. Moore. See Class X.

20,964. F. von Hardtmuth. Improvements in presses for the production of tiles and other ceramic ware. Complete Specification. November 1.

21,091. T. H. Stubbs. Improved method or methods of decorating tiles or the like. Complete Specification. November 3.

21,191. J. Adams and J. Sherwin. An improved method of stamping undercut dovetailing notches or indentations on or in the backs of encaustic and other tiles and other ceramic productions. November 5.

22,008. C. D. d'Enghien, A. D. d'Enghien, and S. D. d'Enghien. Improvements in kilns or ovens for firing terracotta and other like materials. Complete Specification. November 14.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

23,505. R. Y. Foley and F. L. Carslake. Improvements in or relating to the manufacture of clay goods. November 14.

23,753. A. Maw, G. H. Maw, and B. Stuart. An apparatus for applying glazes or enamels to tiles or other like goods. November 14.

24,283. J. E. G. Meran. Improvements relating to the manufacture of pottery. October 31.

24,682. The Worcester Royal Porcelain Co., Lim. E. P. Evans, A. Rushton, and H. Hawker. Improvements in the manufacture of pottery or earthenware. October 24.

1894.

878. E. Bohm. Improvements in the manufacture of enamelled glass letters and other devices. October 24.

17,366. Craven, Dunnill, and Co., Lim., and T. Pitt. Improvements in apparatus for the manufacture of tiles. October 24.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### APPLICATIONS.

19,580. C. Williams. The manufacture of improved fireproof and weather-proof materials or products. October 15.

19,647. W. Owen. Improvements in or relating to the manufacture of artificial stone. October 16.

19,737. J. Wheeler. An improved material for roofing, weathering, and decorative purposes. October 17.

19,829. J. Tulloch. Improvements in tubular bricks or blocks and wall linings for fireproof buildings. October 18.

19,847. N. Davies.—From G. Frederick and S. P. Davies, Australia. An improved composition for use on streets, roads, pavements, and the like, and on the surfaces of wood and metal. October 18.

20,650. L. Kupper. Improvements in gypseous walls. Complete Specification. October 29.

20,848. E. Wilder and H. Holt. Improvements in the process of manufacturing and calcining Portland cement and in the calcining of similar material. October 31.

20,869. G. F. Bowman. Improvements in or relating to fireproof doors, screens, and partitions for buildings and other structures. October 31.

21,173. J. Sheppard and F. Dashwood. Improved fire- and heat-resisting and ventilating floors, ceilings, and other structures. November 5.

21,525. T. H. Lewis. An improved paving and flooring block. November 8.

21,586. M. Williams. Improvement in the manufacture of asphalt blocks and slabs for paving and other purposes. November 9.

21,734. E. Weekwarth and M. Hoeft. Improvements in and relating to the manufacture of cements for making mortars suitable for the production of artificial stone. November 10.

21,735. E. Weekwarth and M. Hoeft. Improvements in and relating to the manufacture of lime mixtures and cements, or mortars suitable for use in the production of artificial stone. November 10.

22,001. W. Youten. Improvements in fireproof construction. November 14.

### COMPLETE SPECIFICATIONS ACCEPTED.

1894.

2247. D. Belloe and E. Benard. Improved process for transforming roasted cements into powder, and in apparatus therefor. October 24.

10,585. C. Geige. A new or improved method or process for the manufacture of artificial wood. November 14.

12,350. W. Bruch. Improvements in the utilisation of precipitated sewage matters (for the manufacture of cement). October 24.

17,049. J. H. Amies and J. L. Robertson. Improvements in pavements. October 24.

17,221. J. R. Ben. Improvements in or relating to fireproof chimneys. October 4.

19,184. C. A. Repl and L. P. S. Wright. Improvements in and relating to fireproof floors and ceilings. November 14.

## X.—METALLURGY, MINING, &c.

### APPLICATIONS.

19,797. The Cowper-Coles Galvanising Syndicate, Lim., and S. O. Cowper-Coles. Improvements in or connected with the manufacture or production of zinc coated wire and other zinc-coated metallic articles. October 17.

20,067. F. Stubbs. A method of carburetting the surfaces of armour plates. October 20.

20,071. L. Bangert. Process for coating metals with tin. October 20.

20,247. D. W. Sugg. An improved metallic alloy. Complete Specification. October 23.

20,365. W. S. Rawson and R. Heathfield. A process for recovery of zinc from scrap galvanised iron and from hard spelter and other zinc alloys. October 24.

20,422. W. P. Thompson.—From F. Bertrand and O. Thiel, Austria. Improvements in the manufacture of steel, steady iron, or homogeneous metal. October 25.

20,557. W. G. Moore. Improvements in furnaces for heating and annealing steel, iron, brass, castings, glass, earthenware, and the like. October 27.

20,615. J. C. Fell.—From F. A. Gooch and L. Waldo, United States. Improvements in processes for reducing aluminium. Complete Specification. October 27.

20,616. J. C. Fell.—From F. A. Gooch and L. Waldo, United States. Improvements in processes for reducing aluminium. Complete Specification. October 27.

20,636. A. Ryrie. Improvements for the protection from corrosion and preservation of iron, steel, and other metallic surfaces. October 29.

20,814. C. F. Claus, C. Goepner, and C. Wichmann. Improvements in the treatment of argentiferous and auriferous complex sulphuretted ores. October 29.

20,815. E. A. L. Langlois and P. F. de Susini. A metallic composition or alloy. October 30.

20,872. P. de Wilde. A method for the extraction of gold. Complete Specification. October 31.

20,955. A. E. Morgans. Improvements in the extraction of precious metals from their ores. November 1.

21,025. H. R. Angel. A process for extracting gold from antimony ore. November 2.

21,069. P. Rogers and J. Player. Improvements in machinery or apparatus for coating metal plates or sheets with tin, lead, or other metals or alloys. November 2.

21,213. P. Huth. Improvements in casting metals. November 5.

21,245. R. Fegan. Improvements in the smelting of ores. November 5.

21,520. A. H. Smith. Improvements in armour-plates. November 12.

22,023. H. Moissan. Improvements in the manufacture of alloys. November 14.

22,051. J. C. Quinn and R. McDougall. Improvements in the extraction of gold and other precious metals from their ores. November 15.

22,066. T. Hampton. Improvements in and relating to the manufacture of compound armour plates. November 15.



22,195. The Cowper-Coles Galvanising Syndicate, Lim., and S. O. Cowper-Coles. Improvements in and apparatus for applying zinc or other metallic coatings to iron, steel, or other surfaces. November 16.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

20,480. W. J. Clapp and S. D. Williams. Improvements in the manufacture and purification of iron and in means or apparatus employed therein. November 7.

21,530. H. F. Taylor. Improvements in apparatus for coating with tin, lead, or other metals or alloy with same. November 14.

21,823. G. Kynech and Co., Lim., and D. Clerk. Improvements in the manufacture of steel shells. October 24.

23,770. E. G. N. North. Process for the elimination and recovery of arsenic from copper precipitates. October 24.

23,930. R. Pearson. A new or improved process and means for treating quartz containing gold, silver, and other metals. November 21.

24,230. C. J. L. Leffler. The manufacture of ferro boron. November 7.

24,231. C. J. L. Leffler. Improvements in the manufacture of steel. October 24.

1894.

3956. W. F. Reid. A new or improved process and material for the production of articles of metal. November 14.

4533. T. Hampton. Improved method of and moulds for casting compounds, ingots for armour plates, and other plates. November 7.

8586. C. M. Allen. Processes of and converters for smelting and refining ores. November 7.

18,919. B. Talbot. Improvements in the manufacture of steel. November 14.

### XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

#### APPLICATIONS.

19,702. G. E. Heyl. Improvements in accumulators. October 16.

19,868. W. A. Thoms and W. H. Burgam. An improvement in electro deposition of an alloy of zinc and cadmium. October 18.

20,059. P. Scheidt and J. W. Tideman. Casting by means of electric current. October 20.

20,127. M. M. Bair. Improved porous pot for primary batteries. October 22.

20,359. C. Kellner. Improvements in electrolytic apparatus for decomposing metallic salts. October 23.

20,354. P. Marino. Improvements in and connected with the electro-deposition of aluminium and aluminium alloys. Filed October 24. Date applied for May 31, 1894, being date of application in France.

21,080. C. Langier. Improvements in secondary batteries. November 2.

21,323. W. P. Thompson.—From C. L. Coffin, United States. Improvements in apparatus for welding electrically. Complete Specification. November 6.

21,330. G. Hübner. Improvements in galvanic batteries. Complete Specification. November 6.

21,555. L. A. Timmés. Improvements in secondary batteries. November 9.

21,822. J. C. Mewburn.—From F. Horaig and W. Borchers, Germany. A vessel for the electrolytic separation and alloying of metals in the molten state. November 12.

21,838. W. Rein. New or improved process for electrolyzing chemical combinations and winning the products of decomposition formed by the electrolysis. Complete Specification. November 12.

21,845. R. Myers. Improvements in dry batteries. November 12.

22,070. J. Hargreaves and T. Bird. An improved process and apparatus for the electrolytic decomposition of metallic salts, and for treating products therefrom. November 13.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

21,120. W. H. Longsdorf.—From G. Hewett. Improvements in primary batteries. November 14.

21,631. W. H. Caldwell. Improvements in electrolytic apparatus. November 7.

21,276. H. Guthrie. Improvements in apparatus for electrolytic purposes. October 24.

24,353. C. K. Mills.—From C. J. Barbier. A new or improved preparation of active material for electric accumulators. October 24.

21,389. A. Lehmann. Improvements in electrode plates for secondary batteries. October 24.

24,442. C. K. Mills.—From C. J. Barbier. A new or improved electrical accumulator plate. October 24.

1894.

646. W. Walker, jun., F. R. Wilkins, and J. Lones. Improvements in primary voltaic batteries. November 7.

9975. W. P. Thompson.—From C. L. Coffin. Improvements in the method of and apparatus for heating, welding, or working metals by electricity. October 31.

13,499. A. Sinding-Larsen. Improvements in process and apparatus for electrolytical decomposition of alkali salts. October 31.

18,288. C. Rordon. Improvements in secondary or storage batteries. October 31.

### XII.—FATS, OILS, AND SOAP MANUFACTURE.

#### APPLICATIONS.

19,968. H. Whitbread. An improved sapaceous compound. October 19.

20,158. M. Perls. Improvements in materials for and in the manufacture of soft soap. October 22.

20,974. H. A. Leverett. See Class XVII.

21,131. A. L. G. Dehne. Process for the separation of oils and fats from water. November 3.

22,205. J. Wigglesworth and C. H. Simpson. An improved soap tablet. November 16.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

21,976. W. E. Heys.—From E. Godehanx. Improvements in compounds for oiling and similarly treating wool. November 21.

24,955. B. J. B. Mills.—From La Compagnie Internationale pour l'Exploitation des procédés A. Seigle. Improvements in the composition of oils for feeding vapour-burning lamps. October 31.

25,001. H. W. Taylor. An improved soap. November 7.

1894.

1088. A. Warwick, J. Smith, and P. W. Nicolle. Improvements in the manufacture of soap and washing compounds. November 21.

### XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

#### APPLICATIONS.

19,535. T. Coppock, jun. Improved varnish applicable to mixing with the bronze powder colours, and for decorative painting purposes. October 15.

19,858. A. J. Boulton. — From G. Philippe, Belgium. Impermeable paint for mirrors. Complete Specification. October 18.

20,023. J. Round. An improved adhesive composition for use upon india-rubber surfaces. October 20.

20,250. J. Armstrong. Improvements in the manufacture of paint varnish and the like. October 23.

20,373. J. P. Lacy and A. J. Ash. Improvements in the production of zinc compounds for use as pigments and for other purposes. October 24.

22,020. A. Tolhurst. A new or improved composition for the preservation of iron, steel, wood, and stone from rust, corrosion, and scaling, and for preventing the fouling of ships' bottoms. November 14.

22,100. A. J. Boulton. — From G. Philippe, Belgium. Improvements in or relating to damp-proof paints or the like. November 15.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

22,730. A. Buecher. Improvements relating to rust-preventive paints. November 21.

1894.

958. J. Hickisson. Improvements in marking-ink pencils. November 21.

5440. E. Herisse. An elastic composition for moulds. October 24.

9883. L. L. B. Meyer. New or improved manufacture of varnish or of a substance for use in making varnish. October 24.

17,630. K. Ammann. Improvements in washing blue. October 24.

17,662. R. Hingston and J. P. Wernegreen. An improved anti-fouling composition for the prevention of marine growth in or on wood or iron. October 24.

### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

#### APPLICATIONS.

21,550. J. Y. Johnson. — From F. Kornacher and Diesel and Weise and Co., Germany. Improvements in tanning. November 8.

22,260. R. H. Evans. Improvements in the manufacture of size. November 17.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1894.

12,941. P. C. Hewitt. Improvements in glue making machinery. October 31.

13,195. T. H. L. Bake and H. A. Leverett. Improvements in tanning skins and hides. November 21.

### XV.—AGRICULTURE AND MANURES, Etc.

#### APPLICATIONS.

20,971. C. H. Thompson. The manufacture of improved fertilised and fertilising materials for promoting the growth of plants. Complete Specification. November 1.

21,658. J. J. Adkins. Treating brewers' spent hops, wet and dry, for the production of manure and fuel. Complete Specification. November 10.

#### COMPLETE SPECIFICATION ACCEPTED.

1893.

21,931. J. T. Dinsdale. Improvements in the treatment of sewage sludge to obtain a useful manurial product therefrom. November 14.

### XVI.—SUGARS, STARCHES, GUMS, Etc.

#### APPLICATIONS.

20,534. A. Young. Improvements in apparatus for the dissolving of saccharum. October 26.

21,344. S. Briggs. — From H. Vynsteke, Belgium. Improved means and apparatus for treating cereals and amylaceous substances. November 6.

21,708. C. H. Meyer. See Class XVII.

21,837. T. Moore, J. A. Sanders, and W. H. Percy. Improvements in apparatus for boiling sugar and the like. November 12.

22,115. A. Harrison. Improvements in preparing starch for laundry purposes. November 15.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

20,397. C. Steffen. Process for purifying and decolorising saccharine juices and sugar solutions. October 31.

1894.

1146. C. H. Meyer. Improvements in the manufacture from starch or starchy substances of a material suitable for use in brewing and for other purposes. November 21.

14,842. H. H. Leigh. — From G. Cambray. Improved process for the treatment of the juice of sugar-canes. October 31.

### XVII.—BREWING, WINES, SPIRITS, Etc.

#### APPLICATIONS.

19,662. T. R. Timby. Mechanism for ageing liquors. Complete Specification. October 16.

20,022. J. Y. Johnson. — From E. L. Doyen, France. Improvements in the manufacture of sparkling wines, cider, and other effervescent beverages. October 20.

20,987. F. N. Mackay. Improvements in apparatus for cooling wort. October 20.

20,796. P. Kröpf and A. Gruell. An improved method of manufacturing beer. Complete Specification. October 30.

20,974. H. A. Leverett. Improvements in the maturing of alcoholic liquors, vegetable oils, tobacco, and other substances, and in apparatus connected therewith, partly applicable to other purposes. November 1.

20,988. J. T. Poplawsky. Apparatus for cooling beer. Complete Specification. November 1.

21,033. E. Larsen. Improvements in pneumatic malting and apparatus used for same. November 2.

21,658. J. J. Adkins. Treating brewers' spent hops, wet and dry, for the production of manure and fuel. Complete Specification. November 10.

21,708. C. H. Meyer. An improved method of manufacturing from starch or starchy substances a material suitable for use in brewing and for other purposes. November 10.

22,270. P. L. F. E. Vignier. Improvements in and relating to distilling apparatus, chiefly designed for the manufacture of pure grape spirit and of whiskies free from fusel oil. Complete Specification. November 17.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1894.

1146. C. H. Meyer. See Class XVI.

17,277. J. Takamine. Improvements in and in the production of diastatic and alcoholic ferments, and the utilisation of the by-products. November 21.

### XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

#### APPLICATIONS.

##### A.—Chemistry of Foods.

19,987. J. Escuyer. An improved alimentary product and process of manufacturing the same. October 19.

20,319. J. A. Wilson. Improvements in methods of treating meat and like foodstuffs in the raw condition, in order to arrest or retard decay. October 21.

20,428. F. Gaillaume and E. Goltstein. Improvements in and apparatus for sterilising milk. October 25.

20,185. W. F. MacLaren. Improvements in and relating to condensed milk products. October 26.

21,224. W. Schwartz. Improved manufacture of cattle food. November 5.

21,710. C. F. Hope. Improvements in the manufacture of cheese. November 10.

22,018. J. H. Theberath and A. de Kunwald. Improvements in the manufacture of margarine and in apparatus therefor. November 14.

22,103. G. Bergman. Improvements in the method of producing fish meal from fish-offal, fresh fish, or other sea animals. November 15.

##### B.—Sanitary Chemistry

20,447. W. Michalik. A process for the combustion of soot. Complete Specification. October 25.

20,744. E. W. Ives. An improved system and apparatus for purifying sewage or other foul or waste waters. Complete Specification. October 30.

20,790. C. D. Abel.—From A. Schlumberger, France. Improved compounds for filtering and purifying liquids. October 30.

21,635. W. E. Adeney and W. K. Parry. Improvements relating to the precipitation of sewage and waste liquids, and the utilisation of the products. November 9.

21,921. W. Smethurst and J. Van der Crab. Improvements in the process of and apparatus for destroying town and other refuse. November 13.

##### C.—Disinfectants.

20,556. C. Gates. Improvements in and relating to disinfecting compounds. October 27.

20,852. E. Burton. An improved disinfectant compound. October 31.

#### COMPLETE SPECIFICATIONS ACCEPTED.

##### A.—Chemistry of Foods.

1893.

23,266. E. Krog. Malted provender for horses, cattle, and sheep. November 14.

1894.

10,106. A. Berstein. Improvements in the production of a peptonised beverage from milk. November 21.

18,143. W. Paasch. Improvements in pasteurising apparatus for milk, cream, or the like. November 7.

19,309. T. T. Chard. A new and improved phosphate food for horses. November 21.

##### B.—Sanitary Chemistry.

1893.

21,082. C. Raleigh. Improvements in separating solid from liquid matter more especially relating to the treatment of slimes, finely-divided ore, precipitates, and sewage. November 14.

21,931. T. J. Dinsdale. See Class XV.

25,040. H. B. Sharp and J. B. Petrie. Improvements in the preparation of compositions adapted for use in the treatment of sewage and foul waters. November 7.

1894.

17,62. C. Salzberger. Improvements in or relating to the disinfection or purification of water. November 21.

##### C.—Disinfectants.

1894.

1415. I. Levinstein. Improvements in and connected with iron compounds for use in precipitating sewage, purifying waste liquors, colouring, and the like purposes. October 31.

### XIX.—PAPER, PASTEBOARD, Etc.

#### APPLICATIONS.

20,051. W. P. Thompson.—From M. C. Stone, United States. Improvements in or relating to the treatment of paper or other tubes. Complete Specification. October 20.

20,221. Henderson, Craig, and Co., Lim., and E. Rutley. Improved means for watermarking paper. October 23.

20,358. H. H. Lake.—From Arndt and Trost, Germany. Improvements relating to the manufacture of photographic printing paper. October 24.

21,520. T. H. Cobley and D. Scott. Treatment of waste fibrous matter for the production of material suitable as a substitute for mill-board, papier-maché, and the like. November 8.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

21,503. J. Y. Johnson.—From P. Sohege. Improvements in the manufacture of cellulose pulp. October 21.

21,669. J. Craig. Improvements in machinery or apparatus for manufacturing paper. October 31.

### XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

#### APPLICATIONS.

19,869. J. E. T. Woods and W. J. Birnie. Improvements in the manufacture of camphor substitutes. October 18.

20,521. C. D. Abel.—From The Fabriques de Produits Chimiques de Thann et Mulhouse, Germany. Improvements in the manufacture of artificial musk. October 26.

21,730. B. Willeox.—From M. Otto and A. Verli, France. Improvements in the manufacture and production of perfumes. November 10.

21,815. A. B. F. Wegemund. Process for the manufacture of a parasite tincture. Complete Specification. November 12.

22,028. L. Gautreau, E. Charbonnier, and E. Serrant. See Class VII.

22,190. O. Inray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of casein compounds. November 16.

22,191. O. Inray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of a new solid silver compound. November 16.

22,251. J. J. Crosfield and G. S. Johnson. See Class VII.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

24,177. S. Pitt.—From the Chemische Fabrik auf Actien vormals E. Schering. Improvements in the manufacture of aromatic sulphuryl derivatives of protocatechuic aldehyde and their conversion into vanillin. October 24.

24,178. S. Pitt.—From The Chemische Fabrik auf Actien vormals E. Schering. Improvements in the manufacture of vanillin. October 24.

24,179. S. Pitt.—From The Chemische Fabrik auf Actien vormals E. Schering. The preparation of a crystalline polymeric base suitable for medicinal use from amido-acetic aldehyde. October 24.

1894.

18,509. M. Otto and A. Verley. Manufacture of ethers of the alcohols contained in natural essences and of their corresponding alcohols. November 7.

### XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.

#### APPLICATIONS.

20,358. H. H. Lake.—From Arndt and Frost, Germany. See Class XIV.

22,013. C. E. Hearson. Improvements in apparatus for imparting a predetermined temperature to liquids for use in developing photographic plates and for other purposes. November 11.

22,189. F. Davis. An improved developing film or plate holder. November 16.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

21,663. J. Packham. Improvements in producing photographic prints. October 31.

25,002. M. Andresen. Improvements relating to the development of photographic pictures. October 31.

1894.

1109. T. Maloni. Improvements in and connected with lighting apparatus for taking photographs at night or in dull weather. November 7.

### XXII.—EXPLOSIVES, MATCHES, ETC.

#### APPLICATIONS.

19,684. V. I. Efeny.—From the United States Smokeless Powder Co., United States. See Class VI.

20,281. R. Thieme. An improved process for producing nitropentaerythrite and a smokeless explosive therewith. Complete Specification. October 23.

20,593. G. Schinko.—From A. Thümann, Austria. Improvements in or relating to "lucifer" matches and the like. October 27.

20,786. E. B. Beeches and J. P. Wright. Improvements in and relating to machines for making matches. Complete Specification. October 30.

21,331. R. C. Schupphaus. Improvements in pyroxylin compounds and in the processes for manufacturing the same. Complete Specification. November 6.

21,729. J. W. Swan. Improvements in and apparatus for the manufacture of nitro-cellulose or gun-cotton. November 10.

22,019. F. Greening, jun. Improvements in and connected with the manufacture of nitro-cellulose or pyroxylin compounds. November 14.

22,192. Siemens Bros. and Co., Lim., and A. S. Schloemer. An electric exploder. Complete Specification. November 16.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

24,425. H. Boyd. A new explosive. November 7.

1894.

1074. M. von Forster. Improvements in or relating to the manufacture of smokeless powder. November 14.

18,682. H. Maxim. Improvements in fulminating compounds. November 7.

18,683. R. C. Schupphaus. Improvements in the process of nitrating cellulose. November 7.

19,098. A. G. Brookes.—From W. J. Smith. Improvements in exploders or fuses for projectiles. November 14.

19,299. G. Hübner. Improvements in smokeless explosives. November 21.

## XXIII.—ANALYTICAL CHEMISTRY.

## APPLICATION.

21,092. R. H. Wilkinson. Improved process for rapidly estimating water in butter, butter substitutes, lard, or other solid fats; also improved apparatus employed in such process. November 3.

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## PATENT UNCLASSIFIABLE.

## APPLICATION.

21,913. C. D. Ekman and C. Christian. Improvements in the treatment of locust beans in order to obtain useful products therefrom. November 13.

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# THE JOURNAL

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### NOTICES.

Notice is hereby given that, in accordance with the alterations in Rule 27 sanctioned at the Annual General Meeting in Edinburgh, all new members elected after January 1st, 1895, and also those elected since the last Annual General Meeting, who do not pay subscription for 1894, will be required to pay an entrance fee of one guinea. In accordance with the same rule the Life Composition Fee will be 20*l.*, instead of 15*l.* as heretofore, after the 1st January 1895.

#### BANKERS' ORDERS.

For the convenience of Members, the Treasurer has arranged with the Bankers of the Society that they shall collect subscriptions from Bankers in town and country; and Members, who have not already done so, are invited to

fill up and sign the Banker's Order enclosed with the December 1893 number of the Journal, which should then be sent to the Honorary Treasurer, Mr. E. Rider Cook, East London Soap Works, Bow, E.

Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 41 of the bye-laws, the Society has the right of priority of publication for three months of all such papers. Infringement of this bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. EYRE and SPOTTISWOODE, the Society's printers and publishers, to whom all communications respecting them should be addressed.

Notice is hereby given that the next List of Members will contain the professions or business occupations of the Members of the Society. Any Member who desires to confirm or alter his description as given before election should communicate with the General Secretary.

Members are reminded that the subscription of 25s. for 1895, payable on January 1st, should be sent in good time to the Treasurer, in order to ensure continuity in the receipt of the Society's Journal. Any changes of address to appear in the new List of Members now in course of preparation, should reach the General Secretary not later than January 15th, 1895.

#### LIST OF MEMBERS ELECTED 1st DECEMBER 1894.

Appleton, H. A. c/o Dr. Hess, Oil Works, Newlay, near Leeds, analytical chemist.

Ashworth, Arthur, Ferrihill Chemical Works, Bury, Lancashire, chemical manufacturer.

Cook, W. Martyn, 29, Highbury Hill, London, N., analytical chemist.

Emanuel, J. F., 36, West 91st Street, New York, U.S.A., varnish manufacturer.

Enquist, Jno., Richmond Hill, Long Island, N.Y., U.S.A., manager of chemical company.

Frank, Jerome W., 29, Broadway, New York, U.S.A., chemist.

Greaves, Wm., Powell Duffryn Collieries, Aberaman, Aberdare, chemical engineer.

Griffiths, Thos., The Cedars, Clapham Common, S.W., manufacturing chemist.

Healy, A. Augustus, 90, Gold Street, New York, U.S.A., tanner.

Heyl, J. Edward, 134, Fenchurch Street, E.C., chemical and electrical engineer.

Hutchison, Jno. F., 22, St. Enoch Square, Glasgow, chemical manufacturer.

Kerr, Jas., c/o Linlithgow Oil Company, Limited, Linlithgow, N.B., works chemist.

Laing, Wm., 224, Swan Arcade, Bradford, Yorks, oil merchant.

Lidgey, Cecil R., 43, Marmora Road, Honor Oak, S.E., analyst.

Machalski, F. J., 159, North Fifth Street, Brooklyn, N.Y., U.S.A., technical chemist.

Macmillan, Arch., Chemical Laboratory, 29, Chambers Street, Edinburgh, analytical chemist.

Pureell, M. F., 13, Victoria Street, Westminster, S.W., consulting chemical engineer.

Smclair, Dr. W., Young's Oil Company, Limited, Uphall, N.B., chemist.

Sutherland, G., Croft Cottage, Bonhill, N.B., chemist.

Thomson, Jno., The Grange, Buckton Vale, Stalybridge, works manager.

Toch, Maximilian, 2364, Seventh Avenue, New York, U.S.A., colour merchant.

Todd, Herbert C., 18, Bread Street Hill, E.C., ink and sealing wax manufacturer.

Urquhart, D., 17, Victoria Street, Westminster, S.W., electrical engineer.

White, Wm., Burmah Oil Works, Dunneadow, Rangoon, Burmah, chemist.

#### CHANGES OF ADDRESS.

Barbour, T. F., 10 Robertson Street: 204, George Street, Glasgow.

Beanes, Edw.; Journals to 119, Ashley Gardens, London, S.W.

Bloomfield, R., 10 Tirhoot; Telhara Factory, via Dhaka Chumparan, Bengal, India.

Brown, Reginald B., 10 Knighton Park Road; 16, Melbourne Road, Leicester.

Butterfield, J. C.; Journals to 13, Victoria Street, Westminster, S.W.

Caldecott, W. A.; Journals to Box 101, Barberton, S.A.B.

Cox, Robt.; Journals to c/o Messrs. J. and G. Cox, Gorgie, Edinburgh.

Crawford, D.; Journals to Langdale's Chemical Manure Co., Limited, Mushroom, Newcastle-on-Tyne.

Crow, Dr. J. K., 10 West Drayton; 23, Vanbrugh Hill, Blackheath, S.E.

Glover, Wm., 10 16; 26, Bella Vista, Minas de Rio Tinto, Huelva, Spain.

Hall, Edgar, 10 Brishane; Rivertree, via Tenterfield, New South Wales.

Harlock, E. B., 10 Urnston; Newton Farm, Middlewich, Cheshire.

Jarvis, Talbot, 10 Bedford; c/o Ind, Coope, and Co., Limited, Burton-on-Trent.

King, Walter R., 10 Southend; 55, Sunderland Road, Forest Hill, S.E.

Laidler, T. S.; Journals to c/o United Alkali Company, Limited, Allhusen's Works, Gateshead.

Mackay, G. A. D., 10 Parliament Square; Police Chambers, Edinburgh.

MacKenzie, Dr. G. S., 10 Pyrmont; c/o Sydney Smelting Company, 116, O'Connell Street, Sydney, N.S.W.

Matthews, C. G., 10 Burton; St. John's Lodge, Beckenham, Kent.

Ogata, S.; Journals to Imperial Mint, Okurasho Branch, Tokyo, Japan.

Sedder, F., 10 Balham; Mersey and Irwell Joint Committee, 44, Mosley Street, Manchester.

Shearer, A., 10 Forest Gate; 36, Demesne Road, Alexandra Park, Manchester.

Sykes, Jas., 10 Huddersfield; Springfield Soapworks, Heckmondwike.

Thomson, J. S., 10 Uphall; West Tower Street, Carlisle.

Trechmann, A. O.; Journals to Tinnoth House, Cuxton, Rochester.

Wilson, Jos. A.; Journals to Vale House, Piercy, Newchurch-in-Rosendale.

Wiostanley, W. H. J., 10 Stockport; Hayfield Printing Co., Hayfield, near Stockport.

Wray, O. J. P., 10 Ferpleigh; 17, Old Dover Road, Blackheath, S.E.

## London Section.

THE CHEMICAL SOCIETY'S ROOMS, HURLINGTON HOUSE, W.

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74, North Side, Clapham Common, S.W.

SESSION 1894-95.

Monday, January 7th, 1895:—

Dr. D. H. Attfield, M.A., D.P.H., "An Investigation of the Natural Sodium Sulphate Lakes of Wyoming, U.S.A."

*Meeting held Monday, December 3rd, 1894.*

MR. B. E. R. NEWLANDS IN THE CHAIR.

## ON THE RATIONAL STERILISATION OF ALIMENTARY LIQUIDS.

By E. W. KUHN, OF PARIS.

THE researches of those celebrated masters of the modern science of "Biological Chemistry," Lister, Pasteur, Hansen, and others who have worked in the same direction, have superabundantly proved that the chief modifications occurring in alimentary liquids are originated and caused by the action of micro-organisms,—yeasts, bacteria, and ferments, and that in some cases the pathogenic properties of waters used for drinking purposes must be ascribed to microbes. These researches, most of which have been made during the last 20 years, have shown that under certain circumstances it may be of extreme importance to destroy these organisms, and thus prevent the changes they set up in the liquids themselves and in the organs of those consuming them.

The chief methods adopted for carrying out this idea of destroying the micro-organisms can be classed under three leading categories:

1. Chemical or antiseptic methods.
2. Physical methods, other than the use of heat, *i.e.*, filtration, electricity, intense artificial cold.
3. The use of heat.

What reception has been accorded to these various methods in science and the industries?

The use of antiseptics has been persistently rejected by medical authorities, because the daily ingestion of any chemical substance with food must be regarded as entailing some risk to the health of the recipient. This method is therefore discarded on hygienic grounds.

Filtration, a method of great merit, although capable of rendering great service in some directions, cannot be applied with success to the sterilisation of alimentary liquids. Besides the difficulty of working and constantly superintending the filters, every system of filtration carried far enough to be efficacious bacteriologically has then the disadvantage of retaining and eliminating from the alimentary liquids, besides the organisms, a quantity of other substances finer than these. These being an essential part of the liquid, cannot be eliminated without completely modifying the liquids, and thus diminishing their value.

Thus if wine, milk, beer be filtered through Chamberland's porcelain filter, with a view to their sterilisation, they lose their colour, become decomposed, and assume in appearance having quite as great a resemblance to pure water as to the original liquid sought to be sterilised. This has been shown by a series of very exhaustive experiments made by Dr. Aubry at the Munich Institution for the scientific investigation of brewing.

The use of electricity and of alternating currents, which has been the subject of much investigation at the hands of Mr. de Meritens in France, has not realised the expectation formed in regard to it. These processes, although perhaps of value in certain directions, such as the rapid maturing of fermented liquids, offer no guarantee of homogeneous or efficacious action in the sterilisation of liquids.

As to intense artificial cold, Mr. Raoul Pictet has made some extremely curious experiments at Geneva, and found that microbes are not destroyed, even on being submitted to a temperature exceeding 200° C. below zero, whereby they are imprisoned in solidified atmospheric air, but on the contrary resume their functions as soon as they are restored to normal temperature.

There remains one other method, the intensely destructive effects of which renders it the most efficacious of them all, and that is, the use of heat.

Although up to the present the use of heat has been attended by very serious disadvantages, the necessity of applying some method or other is so general and urgent that this method has already found application to large quantities of milk and beer, which are heated in bottles by the process known as "pasteurisation." All the beer which is exported from Germany, Austria, Scandinavia, and the United States is thus pasteurised. Moreover, the heating of wine by the "appareils continus" is also resorted to to a certain extent, and similar apparatus are coming into use for the sterilisation of water.

But though the use of heat as a sterilising agent for liquids has already yielded enormous results, it must be recognised that its use hitherto has involved very serious disadvantages, which have arrested the development of its application. The methods hitherto devised have only aimed at utilising the useful effects of heat, without simultaneously suppressing the harmful effects. These harmful effects are the more serious because sterilisation by heat is of value chiefly in its application to the alimentary liquids, that is to say, to liquids in which the slightest alteration of their original condition would be so serious as to involve the rejection of any method causing it. The alterations which these liquids undergo are chiefly:

Alterations of flavour, which in the case of beer has been called the "pasteurisation" or caramel flavour; in the case of milk, the flavour of cooking or boiling; in the case of water subjected to this process, the insipid flavour of boiled water. Then comes the loss of gas, which is likewise one of the usual alterations. All these liquids, even water, are gaseous, and these gases play an important part in the taste and in the assimilating, stimulating, and digestive properties of the liquids, so that the loss of these gases modifies them considerably. Lastly, alterations in brilliancy, which nearly always occur when heat has been used by any of the processes hitherto applied. These are very serious in the case of beer, wine, and water, as are also the dissociation and coagulation which occur in milk. Further, if the pasteurisation of beer, wine, milk, &c. be effected in metal receptacles, they invariably become turbid, a fact which has hitherto been attributed to the use of heat.

I have been engaged in seeking to ascertain the causes of these various disadvantages, with a view to devising a system by which they might be overcome, and it is the result of my work in this direction which I have the pleasure of laying before you.

After lengthy research I have found the following to be essential to the solution of the problem:

1. Immediate action.
2. Equal distribution of calorific effect.
3. Absolutely air tight apparatus.
4. Complete reabsorption of the gases and vapours.
5. Elimination of the chemical action of the metal.

I propose reviewing these points seriatim.



### 1. Immediate Action.

I discovered that the biological action of heat precedes in a marked degree its chemical or physical action, a fact which led me to believe that it would be possible to stop the heat action at a point that would secure the desired biological results, while eliminating the disadvantages attending the latter stages.

I ultimately accomplished this by applying heat to such an extent only as is absolutely necessary to bring about the destruction of the living organisms, which of course vary according to the liquid treated. As soon as this physiological effect has been produced a sudden rapid cooling must take place and the liquid must not be allowed to cool gradually, the intermediate temperatures being harmful. This sudden cooling, immediately after sterilisation, renders it possible to limit the action of heat to this effect alone and to prevent any chemical or physical action of decomposition or alteration.

### 2. Equal Distribution of Calorific Effect.

Any portion of the liquid which is over heated is affected in flavour and brilliancy by the coagulation and precipitation of those nitrogenous albuminoids which are usually present in alimentary liquids. The affected portions mix with and ultimately contaminate the whole liquid. On the other hand any portion which is insufficiently heated is not sterilised, and the propagation of the micro-organisms will still proceed rapidly therein, thus infecting the rest of the liquid. It is therefore indispensable that both the heating and cooling should be absolutely even throughout all the parts of the liquid.

This is chiefly attained in my apparatus by the following features: horizontality, small diameter of cylinder as compared with length, and oscillation. I have by these means obtained an absolute evenness of action, such as could not be obtained with any of the older non-oscillating apparatus, fitted with agitators and stuffing-boxes, which could never remain tight when subjected to alternate heating and cooling. All this entailed constant expansion and contraction, which resulted in loss of gases and altered the liquids treated.

### 3. Absolutely Air-tight Apparatus.

The gases contained in the liquids, apart from their influence on taste, are also the vehicles of the chief volatile aromatic principles of the alimentary liquids, such as vapours of ether or vegetable essences, various alcohols, &c. Any escape of gases would entail the loss of some of these indispensable constituents and consequently an alteration of the liquids. It is necessary, therefore, that the entire apparatus should be perfectly air-tight so as to retain the hot gases under high pressure.

This was difficult to attain in view of the constant and successive expansion and contraction above referred to, but the general arrangement, by which complete evenness of action is obtained, also allowed of the construction of an apparatus hermetically closed in every part. In this way I have been able to sterilise water at 120° C. whilst retaining all the gas originally held in solution in it.

### 4. Complete Re-absorption of the Gases and Vapours.

It is not sufficient to retain the gases by hermetically closed apparatus, but the gases and volatile constituents of the heated liquids must be re-absorbed and re-dissolved.

In order to attain this I relied upon the following facts:—

1. The tensions of the gases and vapours vary directly as the temperature. These tensions can never be sufficient to attain the desired object.
2. The liquid treated expands in direct proportion to the temperature.
3. Every degree of temperature through which a liquid is raised when heated in an hermetically closed vessel may be expressed by a definite augmentation of pressure for each degree. A definite co-efficient of solubility of the gas contained in the liquid is also established for every degree through which the liquid is raised.

The pressure due to expansion is the one to which serious attention must be paid; for if the apparatus were entirely filled, and heat applied until the vessel burst, this would only be effected by expansion and not by the pressure of the gases evolved, which would be kept within restraint by reason of the co-efficient of solubility above referred to.

Guided by these facts I have found that by regulating the level to which I fill the apparatus I can cause the gases to become re-dissolved so soon as the pressure of expansion is removed by cooling.

I have found that as a rule 2 per cent. of the entire capacity of the apparatus must be left empty.

### 5. Elimination of the Chemical Action of the Metal.

In experiments made with different alimentary liquids in tinued or copper vessels, I noticed the rapid and intense reactions which take place between these metals and the liquids. These reactions are due chiefly to the action of the organic acids on the metals, producing gallates, malates, &c.; they are also due to the formation of metallic hydroxides by which the albumenoids and nitrogenous substances are precipitated. This accounts for beer, wine, &c., becoming turbid when heated in metal vessels as above mentioned.

Numerous researches and analyses led me to the conclusion that silver is the only metal which is not subject to these reactions, and which can be used in constructing sterilising apparatus.

All other metals employed, even nickel and aluminium, give these reactions. I therefore silver-plated my apparatus internally, and can now heat the liquids without the slightest metallic action.

I have thus been able, by observing the foregoing principles, to ensure the following conditions:—

1. Complete and evenly distributed sterilisation.
2. No change of taste or flavour.
3. No dissociation or alteration of chemical composition.
4. The preservation of all the gases and volatile constituents without alteration of taste.
5. No alteration of brilliancy.

The apparatus I have designed for beer, wine, milk, &c., consists of a cylinder of silver-plated metal the length of which is at least six times its diameter, fitted with a pressure gauge, a dial thermometer, a water gauge, taps, &c.

This cylinder contains the liquid to be sterilised and has internally a set of circulating tubes which communicate with a jacket, which surrounds the apparatus. The entire structure is placed on trunnions so that it can be easily oscillated to such extent that the top and bottom of the cylinder can be reversed. When the apparatus is ready and its hermetical condition has been verified by carefully examining the joints it is first necessary to ascertain that it is perfectly clean and the liquid is then allowed to enter from below. The water gauge is marked to show the extent to which the apparatus may be filled and this corresponds to an empty area of 2 per cent. of the volume capacity of the machine, to allow for the expansion of the heated liquid. When the apparatus has thus been filled the cock by which the liquid enters and that by which the air escapes are carefully closed and hot water at about 102° above the sterilising temperature. This water passes through the tubes, runs into the jacket which surrounds the apparatus and then flows off.

In 10 minutes the sterilising temperature, which is about 65° C. is attained and then the hot water must be stopped. The liquid is left exposed to this temperature for 15 minutes during which the cylinder is repeatedly oscillated by being turned through half a revolution both to the right and to the left. The pressure then rises to 5–6 atmospheres varying with the quantity of gas contained in the liquor. After this period of 15 minutes ordinary cold water is let in through the tap C. This water passes through the same circuit as the hot water and in about 10 minutes brings the liquid back to its original temperature. In the case of lager beer and liquids which it is desired to maintain at a low temperature the ordinary cold water is stopped and is replaced by iced water at 0° C. In 10 minutes the liquid is thereby brought down to 2–2½° C. Thus the total time occupied by an operation is: for heating, 10 minutes; for resting, 15 minutes;

for cooling, 20 minutes; or a total of 15 minutes. Taking into account the time occupied in filling and emptying the apparatus, the operation does not take more than from an hour and a quarter to an hour and a half, so that six or eight operations can be carried out in a working day. A single workman can perfectly manage the apparatus as all that is required is a little general attention, and that the cocks should be properly opened and shut.

After sterilization it is necessary that the liquids should be drawn off in such a manner as not to expose them to the germs contained in the air which can be done by isobarometric decanting. In the same way it is necessary to see that the vessels destined to receive the sterilised liquids are themselves free from all germs which might develop in the liquids. This is secured by means of steam, boiled water, or sulphurous acid.

Water must be heated to 120° C, and this must be done economically. With this object the water is first decanted and filtered by any of the well known methods, and then fed through recuperators into a steriliser, all of which are filled with water. The steriliser is the same as that described above, except that it is of course unnecessary to silver plate the apparatus, and that it does not oscillate. I proceed as described for beer, wine, milk, &c., except that I must use steam to obtain the temperature. The water contained in the recuperators is destined to be used in the second operation, and I avail myself of the steam when spent in the steriliser and generally of all spare heat still available to subject this water to preliminary heating by which a great saving is effected. Pumps in the steriliser and compressed air in the recuperators may be used for transferring the water if sufficient fall cannot be obtained. With a steriliser of 10 cubic meters capacity I can sterilise 100 cubic meters or 22,000 gallons of water per day, double this if working day and night. Thus it will be seen that a battery of these sterilisers could easily deal with the water supply of a city, and the cost entailed would be small. I have submitted my system to a number of eminent experts who have all expressed their satisfaction.

The following is an account of experiments with water carried out at Liège:—

Water taken from the River Meuse was found to contain 16.9 cc. of gas per litre, and when sterilised in my apparatus it was found to contain 16.7 cc. per litre, so that there had been practically no loss of gas. On the other hand, experiments made at the Bacteriological Laboratory showed that the water when first taken from the river gave in cultures on gelatine plates and in peptonised broth, maintained at 20–22°, an average of 2,230 microbe colonies per cubic centimetre. Most of these colonies were bacillus fluorescens, liquefaciens, bacillus subtilis and bacterium coli. This same Meuse water after sterilisation in my apparatus was found to be absolutely free from living germs, nor did cultures on gelatine plates or in peptonised broth show any development of micro organisms, even when maintained at 37°.

Besides this a hectolitre of water sterilised by my method was taken, and to this was added a litre of broth containing a culture of bacillus subtilis. This micro-organism is one of the most resisting to all methods of desirution, and chiefly to heat. It will live in ordinary boiling water, and its tenacity of life can be compared to that of the anthrax bacillus. It is far more resisting than the germs of cholera and typhoid fever. The emulsion was found on examination to contain 7,380 germs of bacillus subtilis per cubic centimetre. Fifteen minutes in my steriliser at a temperature of 120° absolutely killed all these germs.

The conclusion drawn by Dr. E. Malvoz of the Bacteriological Laboratory of the University of Liège, who made these experiments, is that by my system polluted water containing the most resisting germs can be sterilised in a quarter of an hour at a temperature of 120°.

Extensive experiments were made with beer at Brussels by Dr. van Laer. They were of a most exhaustive character, and may be briefly referred to as follows:—

In the first trial, lager beer was submitted during fifteen minutes to a temperature of 65°, and then cooled suddenly to its original temperature of 7°. After pasteurisation the

liquid remained bright. The persons who had assisted at the operation witnessed no appreciable alteration either in the colour, taste, or smell of the pasteurised liquid.

The samples were analysed before and after treatment with the following results:—

#### ORIGINAL GRAVITY OF WOLF 1173 BEER.

	Non-treated Beer.	Treated Beer.
Apparent extract .....	Grammes, 374	Grammes, 374
Real extract .....	5'475	5'475
Alcohol .....	4'63	4'63
Nitrogen per 100 cc. ....	0'77	0'72
Total acidity (cc. NaH <sub>10</sub> N <sub>10</sub> ) ..	24'0	24'0
Carbonic acid in volume per 100 cc. ....	98'5 Degrees.	94'5 Degrees.
Specific rotatory power ..	16'4	16'4
Number of organisms per cc. ....	1240 cells of yeast and microforma.	None.
Remarks .....		Absence of copper and lead.

Samples of this beer were forced during 15 days to a temperature of 30° C. After this the non-pasteurised liquid was examined to find out whether, when submitted to the incubation it had undergone any appreciable alteration in composition.

The following are the results of this examination:—

	Non-treated Beer.	Treated Beer.
Apparent extract .....	Grammes, 372'5	Grammes, 374
Real extract .....	5'325	5'475
Alcohol .....	4'51	4'63
Nitrogen per 100 cc. ....	0'79	0'72
Total acidity (cc. NaH <sub>10</sub> N <sub>10</sub> ) ..	31'8	24'0
Carbonic acid in volume per 100 cc. ....	98'5 Degrees.	94'5 Degrees.
Specific rotatory power ..	15'7	16'4
Number of organisms per cc. ....	Incalculable num- ber of elements.	None
Remarks .....	Beer turned thick deposit, bad taste.	Bright, good taste without deposit.

As the result of these experiments, Dr. van Laer has certified that beer may be kept unaltered in composition, appearance, and flavour for an indefinite period.

My system of sterilising liquids will no doubt be applied to many useful purposes in the various industries. The chief purposes to which it can now be applied seem to be—

1. Preserving beer and preparing it for export.
2. Maturing certain beers.
3. Medicinal beer.
4. Preserving wine and preparing it for export.
5. Sterilising the wine must, previous to placing therein pure races of yeast.
6. Champagne. Avoiding long storage in the cellars and the necessity of removing the ferment of yeast from the bottles.
7. Preserving cider and preparing it for export.
8. Preserving milk and preparing it for export.
9. Sterilising potable waters and water destined for surgical purposes in hospitals.
10. Sterilisation of the wash in distillation in order to introduce pure races of yeast which yield pure alcohol.
11. Sterilising large quantities of meat extracts, and forwarding them in large receptacles.
12. Preserving large quantities of fruits and syrups.
13. Sterilising infusions of coffee, tea, and cocoa.

14. Sterilising the products of the secretions of microbes, in lieu of sterilisation by means of porcelain filters, in the new methods of inoculation.

I have given much attention to these various applications of my methods, but a detailed account of my investigations under each of these heads would lead me beyond the scope of this paper.

#### DISCUSSION.

The CHAIRMAN in inviting discussion desired, at the request of the author, to convey his thanks to Mr. De Mosenhal for his very able translation and adaptation of this most interesting paper.

Mr. A. GORDON SALAMON hoped that the paper would receive the attention not only of brewers but of those who were competent to pronounce an opinion on the merits of Mr. Kuhn's work as applied to the sterilisation of potable waters. It appeared to him that the results obtained—as proved by the analyses submitted—were of the greatest importance from a hygienic standpoint. For the first time, they had a process capable of dealing with great bulks of water so as to leave them absolutely free from disease germs, without expelling the dissolved gases, or impairing the flavour of the fluid. If that were so, Mr. Kuhn had indeed conferred a benefit upon humanity. Educated in his father's brewery at Clermont-Ferrand, where M. Pasteur made nearly the whole of the experiments in connection with his work, "*Etudes sur la Bière*"; and receiving his initiation into the science of Bacteriology from the great Pasteur, Mr. Kuhn had worked hard at this subject for many years past. For more than a year he had experimented on a large scale on the pasteurisation of beer for use in casks; and though he failed at first in securing homogeneity of pasteurisation, he did obtain results which showed that the gases in the beer were not affected or expelled by the process of sterilisation, nor was the taste of the beer altered. Later, he made the important discovery that certain dissolved nitrogenous substances were, under the influence of heat, acted upon by metals or their hydroxides, silver being the only available metal which would withstand this influence and prevent the brilliancy of the beer from being impaired. Then he secured homogeneity of pasteurisation by introducing the principle of oscillation into the apparatus. Now, as the result of his long series of experiments, he came before them with a process ready not only for the brewer, but for all those who were interested in the preparation of beverages. With regard to its possible application in the brewery, he gathered that the process could only be applied to beer already brewed, *i.e.*, that in order to produce a bright beer it was necessary to start with it bright; nor did the process make a bright beer any brighter. At any rate, there would not seem to be any advantage in passing beer through the sterilisation apparatus and then contaminating it with findings. But even with that limitation it would be of the utmost importance to be able to secure the immunity of our beer from disease, and to export it in casks with full assurance that it would arrive at its destination free from false ferments or deposits and as bright as when it was shipped. This would constitute a new era for the export beer trade of this country, and would possibly be the means of enabling it to compete against the lager beer which had done so much harm to our export trade. In the course of his experiments, Mr. Kuhn had made a further discovery of considerable importance with respect to the determining temperatures of the life history of various yeast organisms. The death temperatures for many of these organisms had been already determined; but Dr. Kuhn had submitted them to intermediate temperatures with this remarkable result. He found that the animation or vital functions of the organisms might be suspended, and that the degree of temperature to which they were heated was a factor in determining the time during which the suspension of animation lasted. Thus, if it were known that a certain export beer was going on a voyage of two months duration, it would be possible to suspend the animation of the ferment for that period, with the certainty that it would return to life at the end of that period and give its natural condition to the beer.

Mr. E. G. HOOPER agreed with Mr. Salamon that the subject was of the utmost importance to the brewing industry of this country. English brewers prided themselves on producing a beer which was sound and capable of standing for a considerable period; but one could not be oblivious of the fact that it would be of great advantage if the beer were also absolutely sterile. The process was equally worth the attention of all those industries which were concerned with the production of aerated waters, and so-called temperance beverages. All these industries would, doubtless, avail themselves of the process if it were practicable. He did not gather that the process was applicable to beer *in cask*, as Mr. Salamon's remarks implied.

Mr. SALAMON regretted that he had not made himself clear on that point. He meant that this was the first practicable attempt at dealing with such a bulk of beer as would permit of sterilised beer being placed in cask. Hitherto, brewers had been restricted to pasteurisation in bottle, and they all knew with what result.

Mr. HOOPER, continuing, said there could be no doubt that if the process could be relied on to produce the results laid before them that night, it would advance pasteurisation to a point never before reached. His past experience of pasteurised beer was that it usually showed considerable deposit and was never effectually sterilised; and whenever the pasteurisation had been effected by heat the beer usually had a mawkish flavour. It was a most essential feature of the process before them that the flavour of the liquid treated was not affected by it.

Mr. F. WILSON had carried out several experiments on the sterilisation of beer in champagne bottles some 35 years ago. He had found no difficulty with the gas, because the bottles resisted the pressure well; but the flavour of the beer was so objectionable that the process was abandoned. He would like to know whether tin was among the metals which were to be avoided on account of its action on the liquid. There was no difficulty in keeping pasteurised beer; he had kept it for 10 years sound and satisfactory in every respect except flavour, which was not only caramel-like, but intensely bitter. That objection did not occur in lager beers, because the quantity of hops used in them was much less than in English beers. He was doubtful whether Mr. Kuhn's process could be satisfactorily applied to the heavily hopped beers of England.

Mr. W. LAWRENCE feared that the process of sterilisation, applied as recommended in the paper, would not yield so good a beer as if it were left un-sterilised, as the ripening action of the beer would be arrested before it had got into thoroughly good condition. He had tried apparatus made of various metals, and had found most of them injurious; therefore he considered bottles best. In his opinion, the great question raised that evening was the sterilisation of water. The difficulty was not in sterilisation, but in persuading people that any such process was necessary. With regard to the loss of gas from water so treated, that was not an important question. He had followed the discussion of this question at the Health Congress held last July, and had exhibited a sample of sterilised water before that Congress. The analysis of that water showed that the gases escaped in the following order: nitrogen, oxygen, carbonic acid. The first and last of these were both undesirable in drinking water, and the oxygen was re-absorbed. The great question in regard to water was not so much the sterilisation as taking away the salts of lime which led to gout and rheumatism. That was what his process did. He could sterilise and remove all the hardness from water at the ordinary boiling temperature either in a small apparatus or up to a hundred thousand gallons per hour. But with regard to the sterilisation of other liquids, that could only be done satisfactorily by such a process as Mr. Kuhn's.

Dr. S. RIDEAL asked whether any experiments had been made in the apparatus with reference to spore-bearing and pathogenic organisms.

Mr. G. W. MACINDOE, while admitting the scientific value of the process, thought that the cost was likely to be very high.

Dr. G. HARRIS MORRIS had also had some experience in the sterilisation of English beer in bottle, and could confirm all that Mr. Wilson had said as to the undesirable flavour produced in that way. The same flavour was not noticeable in pasteurised lager beer, probably, partly for the reason given by Mr. Wilson, and partly because it was masked by the flavour peculiar to lager beer. If the process could, as suggested by the author, be applied to beer in cask, it would, no doubt, be a great boon to the trade, but there appeared to him to be practical difficulties in the way. They were told that the beer, after passing through the apparatus, would go into the cask bright, and that no further fermentation would then take place. They knew, however, that English beer rarely contained any great amount of gas at any one time, and it appeared to him that such amount as it did contain when the beer was exported, would be lost during transit, and that the beer would arrive in a flat, unpleasant, and unsaleable state. With regard to the suspended animation, but not destruction, of the yeast cells, owing to the adoption of certain temperatures, that was perfectly correct, and he believed that continental pasteurisers had been aware of, and taken advantage of, it for some years past. The great point in the author's process, as applied to English beer, would be the absence of any flavour due to sterilisation. If that could be done and the beer would subsequently condition well, the process would be of inestimable benefit to the English trade. With regard to the sterilisation of water by this process one point had not been mentioned, namely, whether there would be deposition on the tubes due to the hardness of the water, and if so, whether this would affect the efficiency of the method.

Mr. D. HOWARD thought that interest in the paper was not confined to the brewing trade, there being a vast number of cases, such as the preserving of liquid foods, or the preparation of drugs, wherein the great difficulty to be overcome was the prevention of decomposition. As a rule, the object was attained by antiseptic rather than by septic methods, heavy doses of some germicide often being put into the material to be preserved. In the early days of bacteriology far too little thought was taken whether the macro-organism might not suffer by the means taken to prevent the spread of micro-organisms, whether a thoroughly sterilised milk containing a large dose of antiseptic might not ultimately determine the existence of a baby as well as a microbe. But just as in surgery, septic methods were superior to antiseptic, so a method which opened out the possibility of sterilising solutions before use was superior to a process for disinfecting solutions. He heartily commended the process, therefore, to the attention of pharmacists and those who prepared liquids to be used as tonics. Possibly, also, it might lead to the displacement of alcoholised tinctorial drinks by something more wholesome, if less exhilarating.

Dr. G. LINDSAY JOHNSTON considered that the subject of the paper concerned the medical profession in a special degree, as the use of sterilised water in surgery was becoming more and more important every day. In the most advanced hospitals of Europe the antiseptic method of treatment was entirely obsolete. The object of surgery was to heal wounds by first intention, or primary union of the parts. To this end, it was necessary not only to remove pathogenic germs, but to prevent the irritation of the tissues, which their access to the wound gave rise to. The organism referred to in the paper (*bacillus subtilis*), was comparatively innocuous. Far more important were the streptococci, and it was impossible in dealing with wounds to use an antiseptic which would kill these germs, because of the irritation which it caused to the tissues. Consequently, if surgeons wanted to secure an immediate and perfect union of the parts, it was impossible to use an antiseptic; and such immediate union was, in some cases (such as cataract of the eye) absolutely necessary to ensure a successful operation. It was for this reason that he expected Mr. Kuhn's process to yield valuable results in connection with surgery. One other point in connection with Mr. Kuhn's experiments he would like to refer to. Treatment with boiling water would kill most of the micro-

cocci, but it would not always kill their ova or spores; certainly it would not kill the spores of the *bacillus anthracis*. This could only be successfully dealt with by successive boilings, the germs hatched from the spores during the cooling being killed in the next boiling. By having at the hospitals a supply of sterilised water produced by Mr. Kuhn's process, this difficulty would be easily got over, and at least one department of surgery would thus be brought to a higher state of perfection than it had yet reached.

Mr. W. THOMAS regarded the process as an extremely useful one, but thought it was subject to limitations. The destruction of organic life in liquids was very important, but how were the liquids to be dealt with afterwards? Hitherto, antiseptics had been relied on. Mr. Kuhn's process would avoid that necessity, but there would be the countervailing one of keeping the sterilised liquid in closed vessels, or the protection would be lost. This difficulty applied equally to potable waters and to chemical or medicinal preparations. No doubt sterilised water could be with care preserved for the purpose referred to by Dr. Johnson; but he greatly doubted the practicality of supplying it through the mains for general consumption. Under the ordinary limitations of domestic conditions, two supplies would be untrustworthy, because the most readily accessible tap would always be used, whether it were the "safe" one or not; and the expense of sterilising the whole town's supply of water would be prohibitive. He did not raise these points as objections to the process, but because it was desirable that in considering a new method, its limitations should be taken into account. The same remarks applied to some extent to the sterilisation of beer. Granted that the flavour was not affected, it would be an advantage to have sterilised beer in vessels. Glass was trustworthy for this purpose, but he doubted whether it would be possible to obtain casks which, after months of travelling, would be proof against the access of bacteria.

Mr. O. GUTTMANN said that the remarks of several speakers had led him to make a mental calculation with respect to one of the most important applications of the process, namely, the purification of water for town use. The result was to show that in order to supply sterilised water to a town of, say, 100,000 inhabitants, it would cost 5,000l. per annum for fuel alone. As this would mean an increase in the rates to the extent of 4s. in the £, he had not much faith in the application of the process in that way.

Mr. C. G. CRESSWELL remarked that, though the application of the process to beer, water, foods, and drugs had been discussed, no one had referred to its adoption in the case of wine. He reviewed the experiences and difficulties he had met with in an attempt to so treat the light natural wines of Italy, Spain, and Portugal as to admit of their being imported for consumption in England. The results of those experiments proved that for red wines containing tannin, sterilisation was not necessary, but that for white wines either the removal of the albuminoids upon which the bacteria lived, or the destruction of these latter by sterilisation of the liquid, was necessary, and for that purpose Mr. Kuhn's process appeared to be the most rational method that had yet come to his knowledge.

The CHAIRMAN, referring to Mr. Guttmann's criticism, thought that that gentleman had not taken into account all the conditions of the problem. At any rate, he for one could not agree with his calculation. He would like to know the number of heat units lost in heating a specific quantity of water by the process.

Mr. GUTTMANN observed that he had only made a rough mental calculation, but that he thought it would be found to be within the mark.

Mr. H. DE MOSENTHAL, replying for the author, said he feared that in his efforts to condense the immense amount of material with which Mr. Kuhn had supplied him into a comprehensive form, he had perhaps laid the process open to questions and doubts which would not have arisen had all

the author's work on the subject been before the meeting. It had rightly been said that one of the most important applications of the process in this country would be that of enabling brewers to export their beer in casks, thus permitting English beer to compete with that made in Germany. Mr. Kuhn had actually applied the process to English beer, and the only reason why the analyses of lager beer were quoted was that he (the speaker) had thought it best to choose those certificates which had the most scientific character, *i.e.* in which the analyses bore testimony to investigation. Members might rely, however, that English ales had been tested in the same way, and that their flavour had remained quite unimpaired. He understood that the author attributed this to the reabsorption of the gases and vapours, which he had endeavoured to explain in the paper. He was assured, too, that experiments with English casks had proved them to be quite equal to retaining the liquor in good condition during the time necessary for transport. With regard to the point raised by Dr. Johnson, he would understand how impossible it was to carry out a series of experiments with every individual microbe; therefore, that micro-organism had been taken which was known to be the most resistant, namely, the bacillus subtilis, and it was shown that by treatment at a temperature of 120° C., with six atmospheres of pressure, the spores of this bacterium were killed. As to wine, the experiments carried out in France had been unnumbered, and had given complete satisfaction to the most eminent experts. In fact, in every direction in which it had been tried, the success of the process was attested by the evidence of the highest authorities. He recognised the difficulties of making people appreciate the necessity for sterilising water. On the continent, however, the importance of doing so was beginning to be appreciated. Mr. Kuhn was now making an installation to supply the barracks at Antwerp with sterilised water.

### INDIA-RUBBER.

BY T. CHRISTY, F.R.S.

Mr. T. CHRISTY exhibited specimens of different sorts of rubber, specially with the view of showing that rubber can be extracted by water. In the first instance the Landolphia was shown with the roots and boughs as cut from the living tree, next the stems after they had been boiled; the next stage was the debris of the bark and the rubber still hanging on to one end of a twig which otherwise was perfectly clean and free from any succus; then there was the mass as it fell into the pan with the bark mixed with the gum. It was then shown in different stages of treatment up to the Landolphia rubber as sent into commerce. Another Landolphia was shown from the Congo: this had been wound off direct from the tree into a ball and dried in the course of winding. Another exhibit was Almadina, so-called from the man who discovered it and worked it out in West Africa; it also goes by the name of potato gum. This gum has most interesting properties, which have been fully explained in the scientific papers, especially by Mr. Lascelles Scott. From a series of experiments lasting over four years, it was found that by placing in a box, open to the sun and rain, some of the very best india-rubber and gutta-percha, some pure Almadina, and also Almadina mixed with india-rubber and gutta-percha, at the end of the experiment the best rubber had almost disappeared and was quite worthless, whereas the india-rubber and gutta-percha mixed with Almadina remained perfectly sound and with full elastic properties. He obtained some tons of Almadina, melted it, and added to it a considerable quantity of water and some tannic acid. This was well stirred and it took up a large quantity of water. When the mass was sufficiently kneaded it was put into bags and allowed to cool, and then sent

down to some large india-rubber works, and the proprietor was so pleased with it that he offered to take any quantity at 1s. 6d. to 1s. 10d. per lb. This rubber, of course, had a quantity of water in it, allowance had to be made in charging the weight when it was handed to the railway of at least 20 per cent. As practical manufacturers on a large scale had now admitted its great value, he then met them and told them that he could no longer continue to manufacture this gum, and that he was prepared, if they gave him a sufficient order for the raw material, to give them all the information. Suffice it to say that they gave the order, but the foreman resented it very much and did all he could not to use this rubber, until his place was handed over to another man who thoroughly understood the valuable properties of Almadina. The consequence was that a very much higher class of goods was turned out of these works and large contracts made; this shows how difficult it is to overcome prejudices.

Another gum also obtained by being boiled was chicle gum. This came from Mexico, and was known to Americans as the base for their chewing gum. Small pieces were prepared for those who desired to experiment with it. He had sent a sample to one of his friends, who was certainly one of the most advanced men in the rubber trade, and explained to him the most simple manner of testing it, *viz.* chewing it; he did so, and he wrote back saying that he was delighted with the material and wanted a quantity for experiment, and, if it went down to a certain price, to put his name down for the first supplies. This gum was also found useful in plaster making and pills.

In conclusion there were many other gums which he believed would advantageously yield to the treatment of cutting down the boughs and boiling them in water, finally extracting the rubber as the mass cooled. He had sent a request to several places where the rubber trees were growing wild, suggesting that this plan should be tried.

### ANALGENE (ORTHO-ETHOXY-ANA-MONO-BENZOYLAMIDO-QUINOLINE).

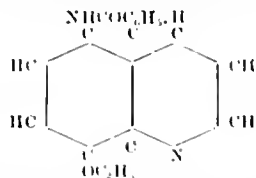
BY E. JAHN.

Mr. E. JAHN exhibited some interesting samples, representing the intermediary products in the manufacture of analgene. Analgene is one of the recent synthetic remedies used chiefly in the treatment of neuralgia and of sundry pains of nervous origin. It has no action on the heart and no unpleasant symptoms are produced by its administration. The colour of the urine of persons having partaken of analgene, however, very often becomes dark red. The red colour is due to the decomposition product of analgene in combination with uric acid. A sample of this substance was among the exhibits, labelled "Muriate of *o*-oxy-ethyl-*a*-amido-quinoline." The solution in water is bright red.

The following is a list of specimens exhibited:—

1. Quinoline-*o*-sulphonic acid, commercial.
2. Quinoline-*o*-sulphonic acid, pure.
3. *o*-Oxyquinoline, commercial.
4. *o*-Oxyquinoline crystals.
5. *o*-Oxyquinoline, sublimed.
6. *o*-Oxyquinoline ethyl-ether.
7. Sulphuric-*o*-oxyquinoline ethyl-ether.
8. Nitrate of *a*-Nitro-*o*-ethoxyquinoline.
9. *a*-Nitro-*o*-ethoxy-quinoline, commercial.
10. *a*-Nitro-*o*-ethoxyquinoline, commercial.
11. Hydrochloride of *o*-oxyethyl *a*-amido-quinoline.
12. *o*-Oxyethyl-*a*-amidoquinoline, commercial.
13. *o*-Oxyethyl-*a*-amidoquinoline, pure.
14. Analgene, crude.
15. Analgene, pure.
16. Hydrochloride of *o*-Oxyethyl-*a*-amidoquinoline.

Analgene is a derivative of quinoline, and has the following formula:—



It is prepared by introducing into the quinoline molecule consecutively an ethoxy-group in the ortho-position and a substituted (acid radicle) amido group in the para-position.

On treating oxyquinoline with caustic soda and ethylbromide, ethoxyquinoline is obtained, which by the action of nitric acid is converted into ethoxynitroquinoline, and this substance is then reduced to the amido form in the usual way. The amido derivative is treated with benzoylchloride and soda in aqueous solution, and thus yields the product in question. This crude analgene is thoroughly purified by repeated crystallisation from alcohol, and in its pure state forms a white crystalline powder insoluble in water, soluble in acids, readily soluble in hot and sparingly so in cold alcohol. As a criterion of its purity, it melts at 208° C. and leaves no residue when ignited on platinum foil.

#### *o*-Oxyethyl- $\alpha$ -Amidoquinoline Hydrochloride.

It has been proved that in the human system analgene is either destroyed entirely, or separation of the benzoyl group takes place with complete oxidation of the residue. Where a separation of the benzoyl group occurs, the urine is always of a dark colour, caused by the action of the free amido product on the uric acid. This may be proved in two ways:—

1. By adding oxyethylamidoquinoline to urine in its normal state. After shaking, and, if necessary, warming, the intense coloration takes place, the intensity varying according to the quantity added from a luminous red to a dark brown red. This coloration is caused by the combination of oxyethylamidoquinoline with uric acid, which produces exactly the same colour when in solution.

2. By isolating the amido compound from the urine after the latter has absorbed the benzoyl product. When soda is added, the colour which is at first red is changed to yellow. On extracting with ether and subsequent evaporation and drying of the extract, the amido compound is obtained not in a pure state, but its presence is unmistakable. The residue when taken up with water and mixed with a solution of chloride of lime yields the light rose colour characteristic of ethoxyamidoquinoline. It disappears gradually when left standing, but instantly on heating. Another reaction proving the presence of ethoxyamidoquinoline in urine may be made as follows:—The urine is acidified with hydrochloric acid, this renders the colour brighter, then some ice and a few grains of nitrite of soda are added, whereupon the liquid turns to a light yellow. On adding a solution of  $\beta$ -naphthol disulphonate of soda, the colour becomes a dark cherry red. When heated with ferric chloride, the red urine turns to a deep red colour.

## PHOSPHOR COPPER AND BRONZE.

BY C. MCCOMBIE.

THE more free from pores and the more homogeneous an alloy is, the stronger, tougher, and more elastic is the casting from it.

Bronze and other alloys of copper in the liquid state contain oxygen, and the casting therefrom is porous in proportion to the volume of oxygen. But if phosphorus be introduced into the liquid mass it eliminates the oxygen by combining with it, and thus prevents the production of porous castings.

The tin contained in alloys is not easily crystallised, except on the introduction of phosphorus into the mass. Two crystallised metals form a more homogeneous alloy than two metals, one of which does not crystallise. Accordingly phosphor-bronze is far more homogeneous than bronze without phosphorus.

Phosphorus furthermore renders alloys highly fusible. Thus the molten mass will fill the very minutest parts of the mould and produce most exact castings.

Phosphorus also reduces the loss in castings, and lessens the expense in making bronze.

The introduction of phosphorus into a molten mass of metal has not heretofore been accomplished without considerable loss of that substance, and up to the present it has not been found possible to introduce into the copper more than 10 per cent.

Mr. Schenckler has, however, quite recently discovered a process by which he gets 20 per cent. of phosphorus into the copper, this being the highest percentage that copper will take.

This phosphor-copper imparts to any alloys all the qualities enumerated above, but it may also most advantageously be used for the renovation of old bronze, brass, and scrap gun metal.

Alloys hardened by an addition of tin lose in toughness and are rendered brittle in proportion to the percentage of tin used. The new phosphor-copper when added imparts to the alloy any desired degree of hardness, and not only produces that effect, but at the same time enhances the strength, toughness and elasticity of the casting.

Finally, alloys containing phosphor-copper have the advantage of not being liable to oxidation.

This phosphor-copper is cast in plates that may readily be broken up into small pieces of equal weight. On adding it to the liquid mass it at once thoroughly mixes with it without developing those stifling vapours so obnoxious to foundrymen.

The samples submitted contain—

	Per Cent.
Phosphorus .....	10
" .....	12½
" .....	15
" .....	18
" .....	20

**Phosphor Bronze.**—The degree of hardness imparted to phosphor-bronze is dependent on the quality of phosphor-copper added to the alloy. It naturally varies with the purpose the casting is intended for.

## ERRATUM.

(This Journal, November 1894, p. 1023.)

TABULAR SCHEME FOR IRON ORE ANALYSIS.

BY J. J. MORGAN.

In column No. 1, under sub-heading *Residue*, for HCl read HF.

## Liverpool Section.

UNIVERSITY COLLEGE, BROWNLOW STREET.

Chairman: E. Carey.

Vice-Chairman: H. Brunner.

Committee:

J. Campbell Brown.	G. Schack-Sommer.
Jos. C. Gamble.	E. Scott.
C. L. Higgins.	A. Smetham.
C. A. Kohn.	Frank Tate.
E. K. Muspratt.	H. Tate, jun.
W. Norris Jones.	

Hon. Treasurer: W. P. Thompson.

Hon. Local Secretary:

Dr. Lewis Bailey, University College, Liverpool.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

SESSION 1894-95.

January 9th.—Mr. Frank Tate. "Modern Improvements in Refrigeration."

February 6th:—

Mr. A. Smetham. "Reversion of Superphosphate."

Mr. J. Walwyn White. "Portable Railways for Works."

Meeting held Wednesday, 5th December 1894.

MR. EUSTACE CAREY IN THE CHAIR.

# ON THE CALCULATION AND COMPOSITION OF BLAST FURNACE CHARGES AS APPLIED TO THE SMELTING OF LEAD AND SILVER ORES.

BY ERIC F. WATSON, F.I.C.

In the process of working ores in blast furnaces we can in general distinguish between two distinct constituents in the ore apart from those which are volatile.

The metal to be recovered and that non-volatile component part accompanying the metal and which is to be slagged.

It sometimes happens that the constituents of an ore to be slagged consist of such a variety and quantity of such elements that the temperature they are submitted to is in itself sufficient to cause the charge to melt, thus reducing it without the addition of fluxes. This of course is the most economical method.

In most cases, however, the ores are of such a composition that they do not admit of being reduced directly, but only after the addition of the corresponding fluxes. In the latter case the metallurgist will (taking due consideration of the properties of the metal to be reduced and the nature of the constituents to be slagged) avail himself of such fluxes as best suit his purpose, or whenever practicable (and which is the best method of procedure) classify his different ores in such a manner that when mixed the one replaces in the other the necessary slagging material and thus gives to the whole the requisite fusibility.

In working lead ores in blast furnaces whether they have to be previously roasted or not the metallurgist will always bear in mind the volatility and ready fusibility of lead, and try to produce a slag, melting at the lowest possible temperature in order on the one hand to increase the output of metal, and on the other to run through within the shortest possible time the greatest amount of ore with the minimum expenditure of fuel.

Now before we can produce a slag of the proper composition so as to admit of a complete reduction of the metal, it is before all things absolutely essential that the chemical composition of the ore be known quantitatively; then only can one learn the amount of elements to be slagged.

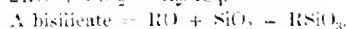
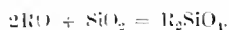
To obtain such an analysis is now-a-days no hard matter, although in a great many cases and especially in large works where ores of various composition are being treated, it becomes not only complicated but almost impossible to analyse each separate batch of ore, simply because the time and cost would make the final process of treatment too expensive.

In such cases the metallurgist is obliged to ascertain the nature and quantity of the constituents to be slagged from previous analyses, or other data well known to him, so that he may be in a position to calculate the most suitable composition to give to the charge.

The ores to be smelted at the works I have at present in my mind, and which I shall take as a practical illustration of my paper, are of the most varied and always changing composition, they contain besides gold, silver, lead, and copper, silica which is most predominant and iron in the form of iron sulphide ( $\text{FeS}_2$ ) as well as manganese, aluminium, calcium, and magnesium in varying quantities. The lead, copper, and a greater portion of the silver occur in combination with sulphur, whilst the gold is to a large extent in the metallic state, only a small portion of it appearing to exist in combination with other metals. The usual method of procedure is to first roast in reverberatory furnaces and then smelt with the addition of the necessary fluxes in the blast furnace obtaining as the principle product argentiferous and auriferous pig lead.

The resulting slag is to be a ferrous silicate and is to have as nearly as possible the composition of a mono silicate, but far more economical purposes is calculated on an oxygen basis of 1:1.5 making it  $\frac{1}{2}$  mono and  $\frac{1}{2}$  bisilicate.

I will explain what I mean by this. A monosilicate has the composition—



R = a metal combined with one atom of oxygen.

In these two slags the oxygen in the base is in proportion to the oxygen in the acid.

In the monosilicate as 1 : 1

„ bisilicate as - 1 : 2

By adding the amounts of oxygen together we get  $2 : 3$  or  $\frac{2}{3} = 1 : \frac{3}{2} = 1.5$ , and it is more economical to do this for the simple reason that we require a less amount of base or iron flux. The base to be used as fluxing material is composed for the greater part of ferrous oxide. According to the analysis and nature of the ores the first thing we should proceed to do would be to classify them into groups.

A convenient classification is the following:—

Under the *First Group* come all dry silver ores (i.e., low grade silver ores) containing no pyrites, or at least pyrites only in small quantities.

Under the *Second Group*. All pyritic ores including matte; and under the *Third Group* all lead ores.

It may be well for me to mention here that when I speak of matte I am referring to roasted iron pyrites  $\text{FeS}$ , i.e., the amount of matte the pyrites is capable of producing, since this is the chief constituent in our ores next to silica and provides us at once with a most suitable means for slagging off a portion of the silica. (I shall work out an example later on so as to show you more clearly what I mean by the “calculation of fluxes.”)

The analysis of group one have shown by practice that the greatest proportion of silica is 80 per cent, whilst the smallest amount of base is equal to 20 per cent. lime ( $\text{CaO}$ ) which substituted in the form of slagging material is equivalent to 20 per cent. of iron.

If it is intended to make the dry silver ores smelt readily it is better to have a small excess of ferrous flux rather than too little and for this reason the assumption is made that the assay gives 80 per cent. silica and only 20 per cent. calcium oxide = 20 per cent. iron.

Likewise in the pyritic ores of the second group, in order to ascertain whether they are readily fusible or if fluxing material is wanting, a determination of the iron and silica must be made.

In fact, in all the pyritic ores it is necessary, by means of assaying, to ascertain the amount of matte they are capable of producing, since this gives us the basis upon which to calculate the contents in iron of the ore available for slagging purposes.

It has been found, according to all the matte and iron assays that have been made, that there exists a constant proportion between the matte assay and actual amount of iron present in the pyritic ores, from one and the same mine, for which reason all pyritic ores have also been classed into four groups as the result of a good many assays and iron determinations made:—

	Per cent. Fe.
In the first group ores containing.....	72
„ second „ „ „ .....	58
„ third „ „ „ .....	51
„ fourth „ „ „ .....	37

of the matte contents.

According to this subdivision, therefore, the amount of iron is calculated by multiplying the amount of matte by that number in per-centages in the pyritic ore in which it was contained. This may at first sight not seem quite clear. I will therefore give an example.

Suppose we have made an assay of these pyritic ores and obtained as a result 40 per cent. of matte.

Now say this ore has come out of the 4th group in which every 100 parts of matte yield 37 parts of iron, then we get the following proportion:—

$$\frac{37 \times 40}{100} = 14.80 \text{ per cent. Fe.}$$



i.e., the above ore giving 40 per cent. of matte contains 11.80 per cent. of iron.

The amount of silica contained in the pyritic ores is then ascertained as follows:—

Since the iron is contained in the ores as  $\text{FeS}_2$ , the equivalent amount of sulphur corresponding to the iron must be first calculated, and the amount of  $\text{FeS}_2$  thus found subtracted from the dry weight. The remainder is then taken as silica and other earthy bases and put down (as in the case of the low grade silver ores) as consisting of 80 per cent.  $\text{SiO}_2$  and 20 per cent.  $\text{CaO}$ .

By this means we arrive at the amount of silica, earthy bases, and by roasting the pyritic ores we also ascertain the quantity of the  $\text{FeO}$ .

If we find the amount of base thus calculated sufficient to make the silica present fusible (i.e., by the formation of the desired silicate) then the addition of an iron flux is not necessary. On the other hand, if the amount of base is in excess then ore may be added, but if there is too much silica and too little base present then the necessary amount of iron flux must be added in order that the ore may melt effectively.

The lead ores we should treat in precisely the same manner. A determination of the lead would be made so as to ascertain if the addition of an iron flux is necessary before they will melt or not.

This estimation of the lead serves only to ascertain whether the lead occurs as oxide or as sulphide in case analysis of the ore should not be to hand. Its equivalent of oxygen or sulphur is first calculated, and the difference between  $\text{PbO}$  or  $\text{PbS}$  and dry weight taken as silica. The necessary amount of  $\text{FeO}$  being then calculated corresponding to the excess of  $\text{SiO}_2$ .

In practice, however, when one is continually smelting the same ore it is customary to draw out once and for all a set of tables which show at a glance the amount of iron and silica required for a certain percentage of lead and matte contained in the ore, thus giving directly the quantity of ferrous flux to be added in order to make the lead ores fusible. These tables are of course founded on the result of a series of assays.

The iron flux is used in the form of brown iron ore (limonite) or in other words hydrated oxide of iron which contains on an average 50 per cent. of iron and is considered an excellent fluxing material.

On these bases every calculation is made, and one can depend upon it that if the calculation has been properly carried out, the charge will melt in the reverberatory as well as in the blast furnace.

In works I have visited where such a class of ores as I have mentioned is treated, this method of calculation and fluxing is resorted to, and it has proved itself to be the best and most economical, and enabled a set of blast furnaces to be worked for a period of two years without any stoppages whatsoever for overhauling. If we look at this method of calculating the charge from an economical point of view, we may still be led to raise the objection that too much silica has been taken into account necessitating thereby the use of an extra amount of iron ore. But this objection does not appear to be sufficient since the presence of a small excess of  $\text{FeO}$  would prevent the danger and assure us of a ready fusibility of the charge, and in case the resulting slag shows itself to be too liquid (a matter readily ascertained as soon as the first charges have been down) the excess of  $\text{FeO}$  could always be neutralised by the addition of dry silver ores always at hand.

And now let me work out for you here an example of an average ore to be smelted, and show how we should proceed to calculate out the fluxes in the ordinary course of events. Let us suppose it is required to calculate the furnace charge, tonnage of bullion and matte and assay composition. Also analyses and percentage of copper in matte for a furnace of daily capacity of 150 tons under the following conditions:—

## Analysis of Ore.

## Excesses in Fluxes.

	Per Cent.	
$\text{SiO}_2$ .....	28.00	Limestone ( $\text{CaO}$ ) 50 per cent. excess.
$\text{Fe}_2\text{O}_3$ .....	33.00	Iron ore ( $\text{FeO}$ ) 60 per cent. excess.
$\text{CaO}$ .....	6.00	
$\text{Cu}$ .....	17.00	Loss of Au, Ag, and Cu in smelting
$\text{Al}_2\text{O}_3$ .....	6.00	3 per cent. each.
S .....	10.00	(of which 8 per cent. goes in matte found
		by practice and analysis of matte).

In silver the ore runs 15 oz., and in gold  $\frac{1}{10}$  of an oz.

Here we have both a bullion and matting problem. The first question to consider is that sulphur has more affinity for copper than for iron.

The loss in copper must therefore first be deducted before calculating the matte, i.e., 3 per cent. of 17 = 0.5 leaves 16.5 per cent. Cu. Next how much sulphur does 16.5 per cent. Cu require in matte formation.

According to our matte tables calculated on the formula  $\text{Cu}_2\text{S}$ .

16.5 Cu require 4.186 per cent. S.

We are given 8 per cent. available sulphur, therefore  $8 - 4.186$  leaves 3.814 per cent. S to combine with iron in matte formation. This 3.814 per cent. S we find requires 6.82 per cent. Fe.

The composition of matte therefore per 100 tons of ore is as follows:—

Cu .....	16.5
Fe .....	6.82
S .....	3.94
	<hr/> 31.32

The next consideration is the combination of Fe and  $\text{SiO}_2$ , and as we require a ferrous silicate all the iron must be calculated on the basis of  $\text{FeO}$ . (For simplicity, I have finally calculated as pure mono silicate  $\text{FeO} \cdot \text{CaO} \cdot \text{SiO}_2$ , and not as a mixture of mono and bisilicate as previously stated.) First then deduct amount of iron taken by the sulphur from the amount in ore analysis:—

33 per cent. $\text{Fe}_2\text{O}_3$ = 20.70 $\text{FeO}$ .
6.82 Fe = 8.56 $\text{FeO}$ .

$20.70 - 8.56 = 12.14$  = the amount of iron in the ore available for slagging purposes.

We find next the—

28 per cent. $\text{SiO}_2$ require 33.60 per cent. $\text{FeO}$ .
Available $\text{FeO}$ = 20.94

Therefore amount to	= 12.66 $\text{FeO}$ .
be fluxed in	

Next to calculate from flux analysis the amount of iron ore required for 100 ton charge of this ore.

Since the iron ore contains 60 per cent. excess we get the following simple proportion  $60:100::12.64:x = 21.1$  = tons of iron ore necessary for fluxing 100 ton of the ore to be smelted.

Similarly take the estimation of lime equivalent in ore—

CaO, .....	= 6 per cent.
6 per cent. $\text{Al}_2\text{O}_3$ equivalent to CaO = 9.88 per cent.	

Therefore  $15.88 = \text{lime value in ore}$ .

As in the case with iron, so here we must also determine the amount of lime required by 28 per cent.  $\text{SiO}_2$ , and we find that the lime required by the  $\text{SiO}_2$  in ore to be smelted

= 26.32 per cent. $\text{CaO}$ .
Available lime in ore = 15.88 per cent. $\text{CaO}$ .

Therefore the amount	= 10.44
to be supplied by flux	

Since the limestone flux contains 50 per cent. excess  $\text{CaO}$  we need only multiply 10.44 by 2 to get the amount necessary for 100 tons = 20.88.



The tonnage of fluxes for 100 tons of ore are therefore :—

Iron Ore.....	21.10
Limestone.....	20.48
	<hr/>
	41.58
Ore .....	100.00 tons,
	<hr/>
	141.58 tons.

All these calculations have now to be readjusted since the capacity of furnace charge is 150 tons, and we have so far only calculated to 100 tons plant.

The following proportion you will find gives us the true composition of our charge—

141.58:150::100::x = 105.94 tons of ore in 150 ton charge.
141.58:150::21.10::x = 22.35 " " iron flux " "
141.58:150::20.48::x = 21.69 " " limestone " "

149.78 = approximately 150 tons.

If we readjust the 31.32 tons of matte made on 100 tons of ore to absolute ore charge, i.e., 150 tons, we get  $31.32 \times \frac{150}{100} = 31.77$  tons of matte.

To find the percentage of copper in matte; in 31.32 tons matte we have 16.5 per cent. Cu = 52.68 per cent. copper.

The amount of bullion produced is calculated as follows:—  
105.94 tons of ore  $\times 45$  oz. = 4767.3 oz. = gross amount of silver in ore. Loss = 3 per cent. = 143.01; therefore 4767.3 oz. - 143.01 leaves 4623.29 = nett amount of silver in ore.

The gold per ton = 0.2 oz.; this on 105.94 tons = 21.18 oz. = gross amount of Au in ore; loss = 3 per cent. = 0.635 oz.; therefore 21.180 oz. Au. - 0.635 = 20.545 oz. = nett amount of gold in ore.

In the analysis of the ore to be smelted I have not shown any lead. In order therefore to complete the problem let us assume that there is 12 per cent. PbO. This does not to any great extent affect the calculation since the metal is to be reduced, though the amount of elements to be slagged would be slightly less. 12 per cent. PbO = 11.12 per cent. Pb = tons of lead in 100 tons of ore or 11.78 tons of lead readjusted to absolute ore charge of 105.94 (roughly 106 tons).

Deducting 3 per cent. for loss we get as the nett amount of lead produced 11.43 tons.

This completes the problem as far as the calculation of the fluxes goes. Of course I could go much further by giving you a statement showing the financial considerations of this problem, but I have not deemed it necessary. It only remains now to insure a thorough mixing of the whole, and for this purpose layers of ore and flux are laid alternately on top of one another, and cut vertically when required, the cutting showing a stratified appearance.

The best practical evidence we can then have as to the right composition of the charge is its behaviour on roasting in the reverberatory furnace. If it already begins to melt on roasting we can take it for granted it will melt still more readily when charged into the blast furnaces. In contradistinction to what I have been saying, I might give you an instance I heard of of unsuccessful rule of thumb smelting where the works had to be closed up and abandoned simply because about 30 per cent. of the metals were being lost in the slags, and this was proved by the fact that some more competent smelters came along, bought up these old slags and actually made a profit out of them.

In conclusion let me admit that I am fully aware how delicate a matter it is for me to pretend to give any opinion as to the organisation of smelting works to such men as yourselves who have much more practical experience than myself, but I have ventured to say this much in order to draw more attention to what certainly lies at the root of all successful smelting, viz., systematic assaying and analysis, and the giving of the proper composition to the charge so as to combine all the useful elements in such due proportion as to extract the greatest amount of metal.

## Manchester Section.

ROOMS OF THE CHEMICAL CLUB, VICTORIA HOTEL.

Chairman: R. Forbes Carpenter.

Vice-Chairman: Edw. Schunck.

Committee:

G. H. Bailey.  
F. H. Bowman.  
G. E. Davis.  
C. Dreyfus.  
H. Grimshaw.  
P. Hart.

J. M. Irving.  
E. Knecht.  
I. Levinstein.  
W. H. Perkin, jun.  
Sir H. E. Roscoe, M.P.  
W. Thomson.

Hon. Local Secretary:

J. Carter Bell.

Bank House, The Cliff, Higher Broughton, Manchester.

SESSION 1894-95.

Friday, January 4th, 1895:—

Mr. R. Le Neve Foster. "The Prevention of Smoke arising from Intermittent Firing in Various Chemical and Allied Processes."

Mr. R. J. Flintoff. "The Formation of Mazonia in developing Aniline Black on Cotton."

Meeting held Friday, 7th December 1894.

MR. R. FORBES CARPENTER IN THE CHAIR.

In the half hour before the reading of papers which is allotted to discussion Mr. Levinstein referred to several laboratory experiments which were made by Behring previous to the introduction of antitoxin in hospitals. In the first instance a small measured quantity of the diphtheric poison from Löffler's diphtheria bacillus was injected into a guinea-pig, when death followed; then a calculated quantity of antitoxin was injected into another guinea-pig, and a quarter of an hour afterwards the same quantity of the diphtheric poison which previously proved a fatal dose, was injected, when no symptoms of diphtheria developed. Finally a number of cc. of the diphtheric poison was poured into a test tube, to this were added so and so many cc.'s of antitoxin. This mixture was injected into another animal when again no symptoms of diphtheria or any other symptoms were noticeable. These experiments prove without doubt in Mr. Levinstein's opinion that the antitoxin neutralises and makes the true diphtheric poison of Löffler's bacillus innocuous. How far these remarkable results obtained with animals will be confirmed in treating human beings suffering from true diphtheria, and how far no other ill effects may follow such treatment, only time can definitely decide. Several chemists are at present engaged in the investigation of the chemical constitution of antitoxin, and if they should be successful in their labour, there is no reason why antitoxin may not be one day synthetically produced without the intermediate use of animals. Mr. Levinstein then showed three bottles, each containing about 10 cc. of fluid-healing serum, bottle (1) with 600 immunity units; bottle (2) with 1,000; bottle (3) with 1,500 units. The serum had been prepared by the Farbwerke Hoechst a M. under the personal supervision of Professor Behring and Professor Ehrlich.

PIPE COLUMNS FOR SAVING CHAMBER SPACE,  
IN THE MANUFACTURE OF SULPHURIC ACID.

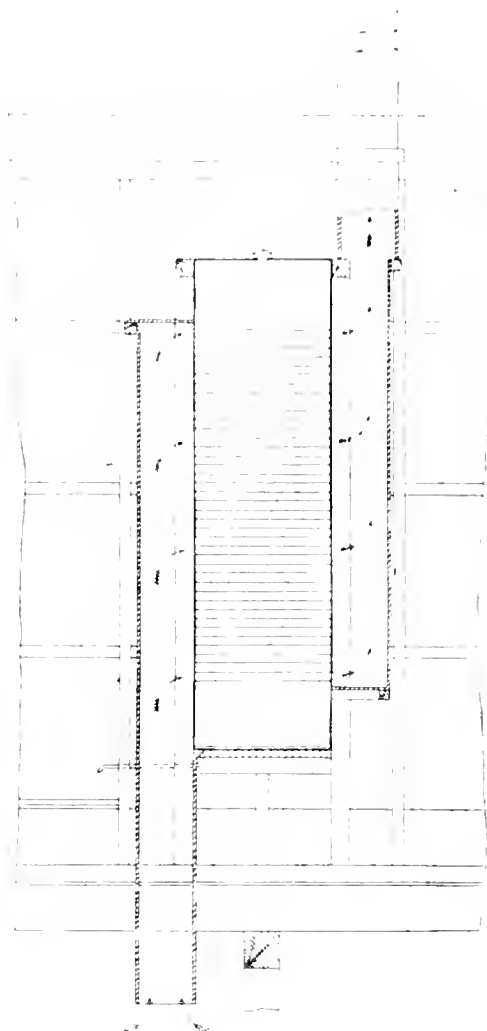
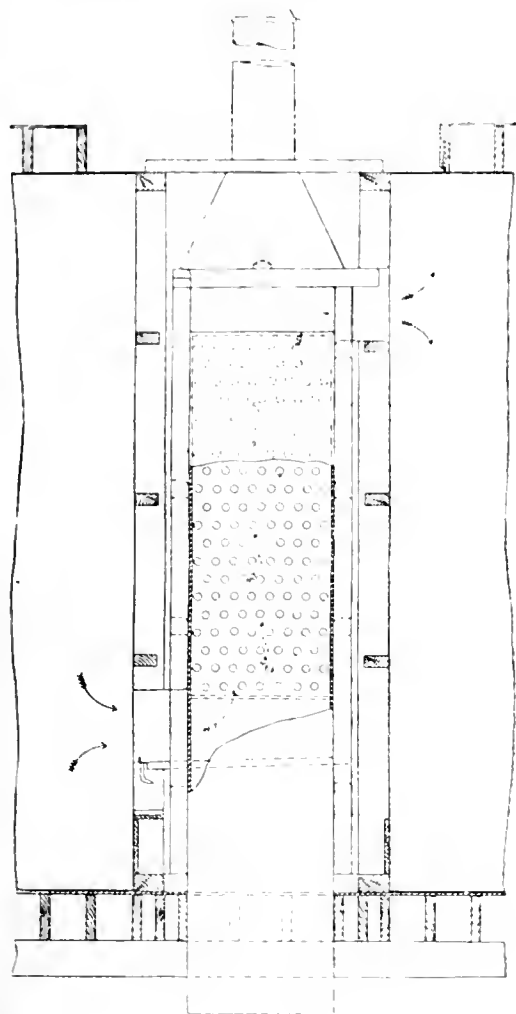
BY PETER S. GILCHRIST.

In these days of severe competition, manufacturers have to look very closely into the cost of production, to see that each article is produced at the lowest possible cost. In no branch of business has competition been felt more keenly than in the chemical industries. Manufacturers re-model their plants to maintain efficiency, so as to cope with newer plants that are erected from time to time, and which com-

prise the latest modern improvements, for the advantages in manufacturing certainly lie with the latter. In this branch of industry, nothing plays so important a part as sulphuric acid, and it is to the manufacture of this I confine myself, but more especially to the chamber process.

The construction of chambers is the same in general, but these differ materially in their relative sizes, usually being

long, so as to get the maximum amount of capacity from a minimum consumption of lead, the longest chambers being generally at the first part of the system, and reducing towards the end, the last one usually being of small capacity. Chambers are now built in America, with a width of 30 ft., and a height of 20 ft., this being wider than is generally found in English works.



NO. 1. PIPE COLUMN.

As to the length of chambers, there are wide differences of opinion, personally I prefer short chambers. The longest one the writer has had experience with was in Baltimore Md., being 225 ft. long, but it was found difficult to obtain uniform working throughout.

In a chamber, say 60 ft. in length, there is no doubt a higher yield of acid proportionally obtained, in comparison with a longer chamber, for the gases in traversing the chamber naturally become weaker, owing to condensation of the gases in progress: this is shown by the drips and also by an analysis of the gases.

You are all acquainted with Professor Lunge and Dr. Naef's experiments, showing how unequal the formation of acid is in the chambers, and how the curve of acid formation rises to a certain point, (in the first chamber about 50 ft. from the front), and then takes almost a horizontal move-

ment until it enters the next chamber, when the curve again rises, and so on.

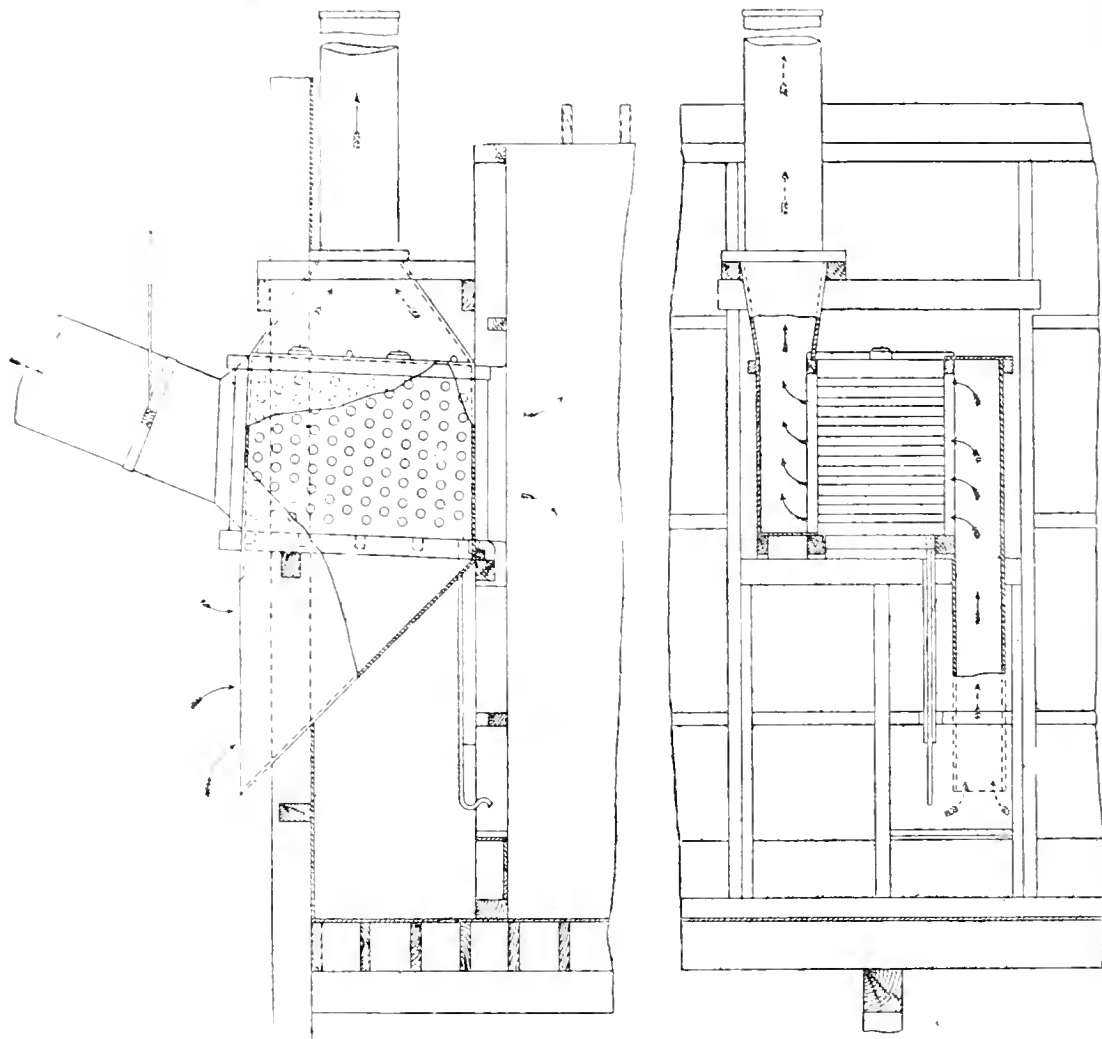
Anyone watching closely the passage of the gases from one chamber to another must see that it causes a great reaction to set in, consequently followed by an increased production of sulphuric acid.

The reasons for this are obvious, the vapours strike against the surfaces of the chamber ends, and the sides of the connection, causing the particles forming to interact more quickly. Again the gases in passing through the small aperture, are better mixed, and are cooled by radiation from the surfaces of the lead.

With small chambers this result is repeated oftener than with large ones, so the work accomplished in a corresponding amount of chamber capacity must necessarily be in favour of the small chambers.

The oftener this increased reaction takes place in the system, the more work can a given chamber space accomplish, and it is with this in view we aim to diminish

the chamber space, and probably eventually to do without any.



NO. 2. PIPE CASE BETWEEN GLOVER AND FIRST CHAMBER.

To this end the writer and F. B. Hacker have devised the "Pipe Column," it is a small tower 4 ft. by 3 ft. 6 ins. by 13 ft. 6 ins. high, with horizontal lead pipes burnt to the sides, and so placed as to continually break the course of the gases, as they pass through. See sketch No. 1.

The column is cased in on two opposite sides, facing the ends of the open pipes, one side being left open at the bottom, and the other at the top, which is connected with a stack, by this means a strong current of air is continually drawn through the pipes, or artificially cold air can be forced through.

Acid lites are put on the top, for acid to pass down, or steam may be introduced into the tower, along with the inlet gases.

The column sits in a strong wooden frame, the bottom of the tower being carried on four spiral springs, of such resistance as just to take up the expansion of the lead, as it usually expands in the same proportion as the chamber side sheets, this prevents any wrinkling or cracking of the lead connections to the chambers.

The casing around the tower which forms the flues, is made of one inch and a quarter tongued and grooved

narrow flooring boards; the stack of light galvanized iron. The tower is constructed so that it can easily be got at, all burning being outside, the pipes are also flanged and burnt on the outside, making a strong and compact tower.

The weight of lead used can be varied according to the amount of work the column has to do, 10 pound per square foot, at the first part of the system, and eight pounds per foot towards the end.

Sometimes we use a pipe case in the place of a column, this is simply a square or rectangular horizontal connection, with pipes as in the column, see sketch No. 2.

The pipe columns can be placed between the Glover and the first chamber, between the several chambers, and between the last chamber and Gay-Lussac tower, if necessary, but it is only essential in very warm climates.

The gases in passing through the column become very thoroughly mixed, by coming in contact with each successive layer of pipes, causing the particles forming to interact very rapidly, and the moist surfaces upon which the gases impinge are conducive to a great chemical reaction, owing to the very rapid formation of sulphuric acid.

The heat of this reaction is carried off by means of cold air passing through the pipes, which is very important.

The gases, in coming in contact with the surfaces of the pipes, are attracted momentarily (partially due to the moisture present, and also to the cooler surfaces exposed), give up the heat due to the reaction caused by the formation of sulphuric acid, and the liberation of nitrous acid gas, also lowering the temperature of the main body of gases.

This in turn causes a repellant movement, which communicates to the numerous particles in their upward passage through the column, so that a more complete mixing is obtained than by simple obstruction.

Sufficient moisture must be present to make the re-action efficient, and it is essential that it be applied so as to cover the surfaces of the pipes.

The simplest way is by introducing steam along with the inlet gases, say at the top of the chamber, about 10 feet

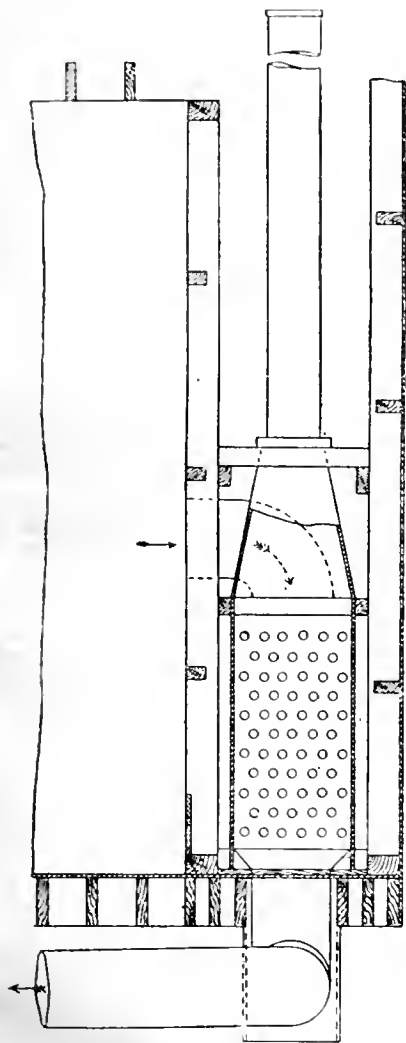
from the column, the nozzle of the pipe, pointing towards the inlet; this has worked well in practice.

Weak acid could be passed down the tower, which would absorb some of the heat, by passing vapour into the next chamber, and thus it would assist the cooling, but against this is the cost of pumping and attention. At the latter end of the system, steam is the best medium for introducing moisture to the columns.

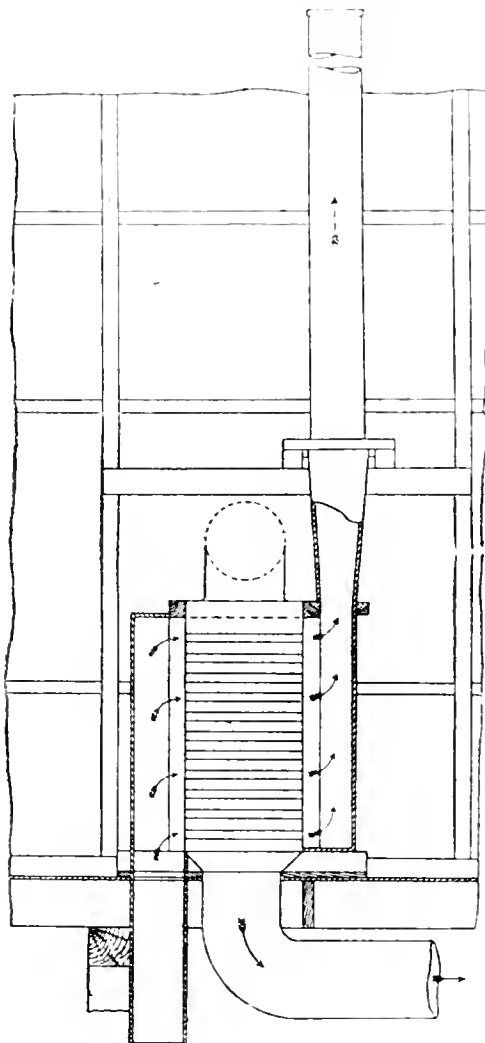
So many obstructions to the gases as the numerous pipes present, suggest to the practical mind one of the greatest evils in chamber working, namely, bad draught; but such is not the case.

The columns average 14 square feet in area; less than half of this is taken up by the pipes, and after allowing for friction, it still leaves ample passage-way.

In text-books on the working of chambers, we are told in draughting-chambers, to let the first one work with



NO. 3. PIPE CASE BETWEEN LAST CHAMBER AND GAY-LUSSAC.



a good pressure, two-thirds of the way, or thereabout, normal, and the last chamber a good suction.

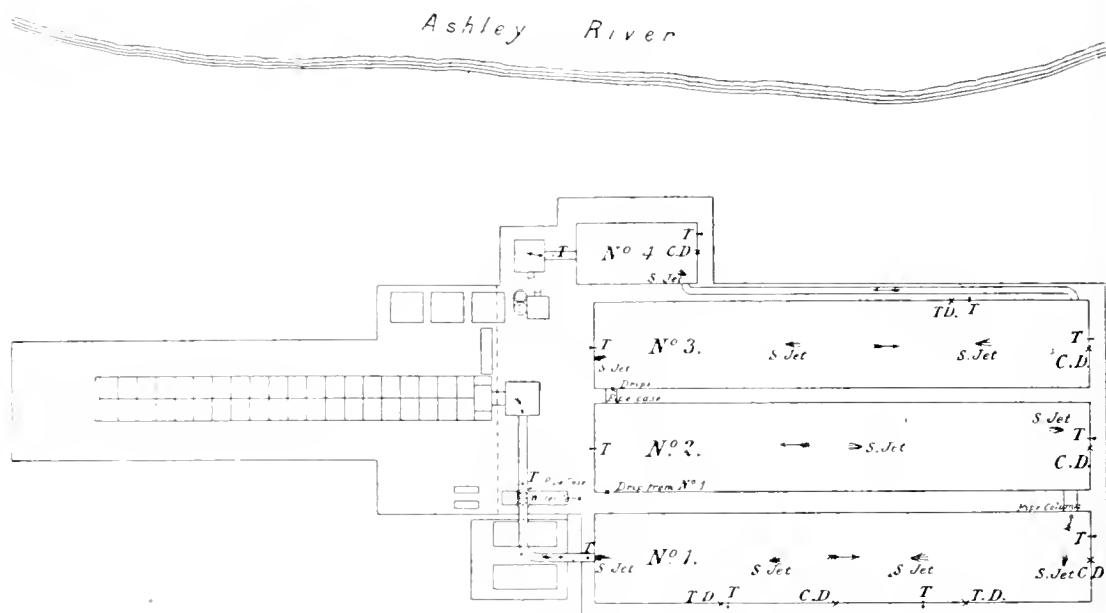
To do this is simply a question of construction, for if the connections are larger, we cannot attain it.

This has been the case with the pipe columns in combination with short chambers, a strong pressure being on all of them, and at one of the works where they are in operation, even a strong pressure at the lutes on the Gay-Lussac tower.

This suggested to the writer the desirability of complete control of the escape gases, when under such conditions as are here presented. A wind storm coming up suddenly would play great havoc with the chamber workings, especially in America, when the exit pipe is direct from the top of the Gay-Lussac tower, draughting into a large chimney being seldom used; so to prevent such a condition occurring, an automatic damper is placed in the exit pipe from the Gay-Lussac tower.

This prevents the sudden withdrawal of any large body of gas, even with a wind travelling at fifty to sixty miles an hour, as the valve in the damper reduces the outlet as

necessary. The percentage of oxygen in the escape gases varies very slightly by this arrangement. The first application of the pipe columns was in November 1892, at the



NO. 4. PLAN OF THE WANDO PHOSPHATE COMPANY'S CHAMBERS.

works of the Wando Phosphate Company at Charleston, S.C. They were added on a small scale to an existing plant of 246,000 cubic feet, consisting of four chambers, the first three being each 160 feet long, the last 40 feet (Glover and Gay-Lussac towers); using lump pyrites, see sketch No. 4.

There was a small pipe case introduced between the Glover and the first chamber,  $3'6'' \times 4'0'' \times 6'6''$  with 66,  $2\frac{1}{2}$  inch pipes.

Between the first and second chamber a pipe column,  $3'10'' \times 4'0'' \times 13'6''$ , with 110 pipes.

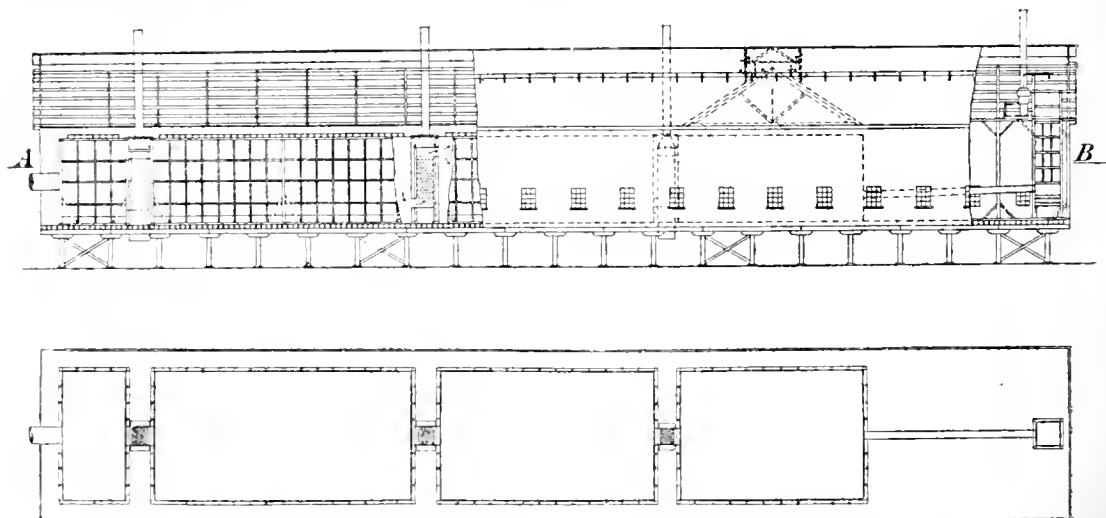
Between the second and third chambers a pipe case,  $3'4'' \times 4'0'' \times 5'6''$ , with 56 pipes. And on re-building their second Gay-Lussac tower, they added a pipe case,

$3'0'' \times 3'0'' \times 5'0''$ , with 70 pipes in the down pipe between the two towers, their object being to cool the gases completely before going into the final tower.

A test over a long period gave a cubic capacity of a little under 17 cubic feet per 1 lb. sulphur per 24 hours, using 3.6 per cent. nitre, and giving a yield of 4.8 lbs. of 50° Beaumé acid per 1 lb. of sulphur.

There is but little acid made in the last two chambers, no steam being used in these.

The chambers will stand more pyrites furnaces being added, but in lieu of this, when increasing they will probably reduce the chamber space, using part for a set of fines furnaces.



NO. 5. PLAN OF THE DARLINGTON PHOSPHATE COMPANY'S CHAMBERS.

The following are the temperatures:—

Entrance to first pipe-case, 230° F.

No. of Chamber.	Front.	Back.
	° F.	° F.
1	205	200
2	165	163
3	125	120
4	100	90

Atmospheric temperature, 83° F.

The acid leaving the bottom of the Glover tower is 325° F. I mention this as it will be of interest to some of you, showing what high temperatures are sometimes got in warm climates.

Oxygen, 5.5 per cent. and .5 grains sulphuric anhydride per cubic foot in the escape gases. In this case the pipe columns were too far apart to work to good advantage.

At the time the pipe columns were added, they changed from sulphur to pyrites when burning sulphur; the chamber pace was 21.15 cubic feet per 1 lb. of sulphur per 24 hours, nitre 4.29 per cent. on the sulphur, yield 4.4 lbs. of 50° acid per 1 lb. of sulphur.

This shows an increase of 29.1 per cent. in the yield of acid made, not taking into consideration the change from sulphur to pyrites. It also shows a reduction in the consumption of nitre of 16.1 per cent.

These tests were taken in the corresponding months during the warm weather.

These columns are doing excellent work to day, without any appreciable wear, for on a recent examination of the pipes, they were found in excellent condition; and though the gases enter the first pipe case at 230° F. and sometimes above, there has been no repairs upon it whatever.

The second application of the columns was at the works of the Darlington Phosphate Company at Darlington, S.C., the August cyclone of 1893 having blown down all the chambers.

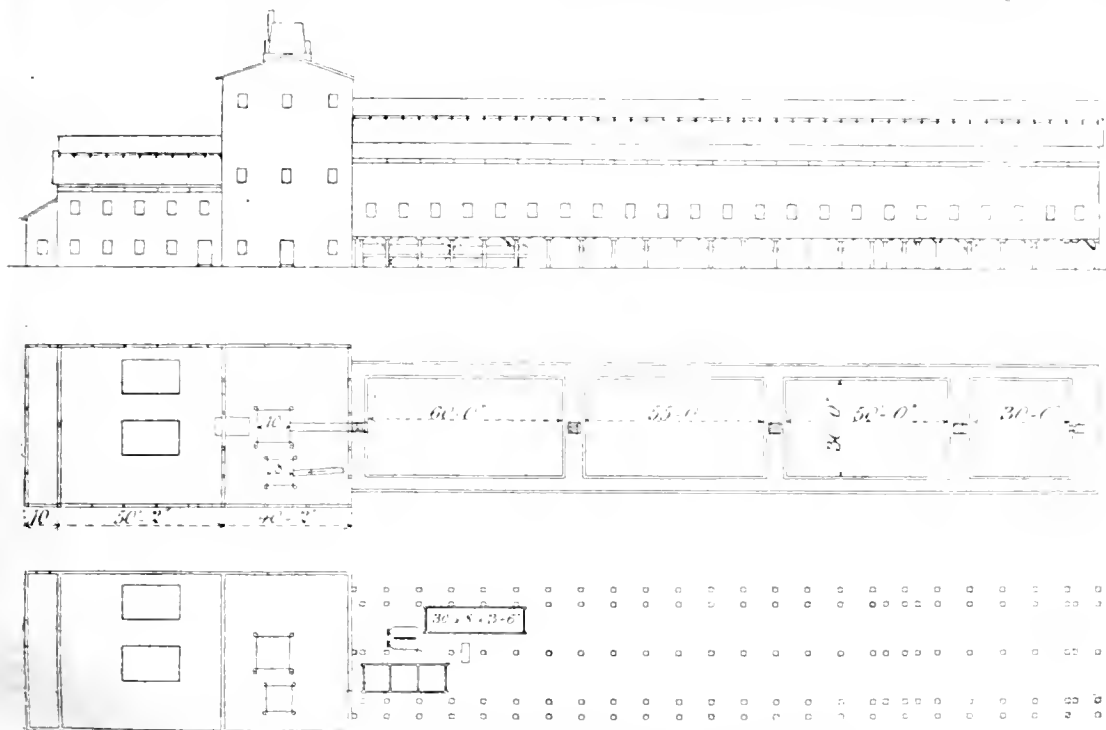
The original plant consisted of three chambers, having a total capacity of 129,500 cubic feet, with a small coke tower in the rear, using Johnson's mechanical fines furnaces, with no nitre recovery, making at the outside 11 tons of 50° Beaumé acid per day.

The present plant consists of four small chambers, the first one only 15 feet long, its object being to catch the dust; the second 60 feet, the third 50 feet, and the fourth 13 feet, having a total capacity of 100,800 cubic feet, with coke tower in the rear, using the same furnaces and no nitre recovery. See sketch No. 5.

Between each chamber there is a pipe column, averaging 3' 10" × 3' 5" × 13' 6", with 150 2½ pipes between the last chamber and the coke tower, a small pipe case, 3' 6" × 1' 10" × 6' 0" long, with 56 pipes 1' 10" long.

A test for 81 days gave the following:—

Pyrites burnt per day 14,304 lbs., averaging 11.6 per cent. sulphur, two-thirds being Virginian ore and one-third Spanish, actually yielding 12.12 per cent. sulphur. This is an equivalent of 6,025 lbs. of sulphur, being 16.5 cubic



NO. 6. PLAN OF THE SAVANNAH GUANO COMPANY'S CHAMBERS.

feet of chamber space per 1 lb. of sulphur per 24 hours, and in the cooler months was under 15 cubic feet, nitre consumption 13 per cent.

The average yield of acid for this period was 14 tons of 50° Beaumé acid (2,000 lbs. per ton) per day, or 1.65 lbs. of 50° Beaumé acid per 1 lb. of sulphur.

As this means an increase over the total chamber area of seven-tenths of an inch, calculated as 50° acid, one can readily appreciate the amount of work done in the system.

The average temperature of the chambers were as follows:—

No. of Chamber.	Front.	Back.
	° F.	° F.
1	220	222
2	178	178
3	156	154
4	123	102

This is after passing the pipe case.

Atmospheric temperature 73° F. Average oxygen in the escape gases 6.6 per cent. Average acidity in the escape gases 0.81 grains sulphuric anhydride per cubic foot.

The result of the application of the pipe column over the original plant is as follows :—

Reduction in the size of the chambers 21.84 per cent. Increase in the yield of sulphuric acid made 28.18 per cent., equal to a total increase of 50.02 per cent.

The last to put in these columns was the Savannah Guano Company, of Savannah, Ga. These works were also destroyed by the same cyclone, just leaving the furnaces standing, and these were much injured.

The original plant consisted of three chambers, having a total capacity of 126,000 cubic feet, with Glover and Gay-Lussac towers, and two Spence fires-furnaces, making 80 tons of 50° Beaumé acid per week, using 5 per cent. nitrate of soda.

The plant now consists of four chambers, the first 60 feet long, the second 55 feet, the third 50, and the last 30 feet, having a total capacity of 117,000 cubic feet, with Glover and Gay-Lussac towers, and the two Spence furnaces.

Between the Glover tower and the first chamber a pipe case 3' 3" x 4' 6" x 6' 0" with 68 2½-inch pipes, between the several chambers a pipe column 3' 6" x 4' 0" x 13' 6" with 150 pipes, and a case 3' 0" x 3' 0" x 5' 0" with 60 pipes, between the last chamber and the Gay-Lussac tower. The latter was placed to dry the gases before entering the absorption tower, but it has not been found necessary.



No. 7. SECTION OF IMPROVED PIPES.

For a time this plant worked below 15 cubic feet, when the atmospheric temperature in the shade reached 96° F., but it was found that the two Spence furnaces could not maintain this heavy work, so they have had to cut down till the present production is only 112 tons per week, which gives an increase of 40 per cent., added to a reduction in chamber space of 7.7 per cent., or a total gain of 47.7 per cent.

The company may add another furnace later so as to get the full advantages of the chambers.

The temperatures run as follows :—

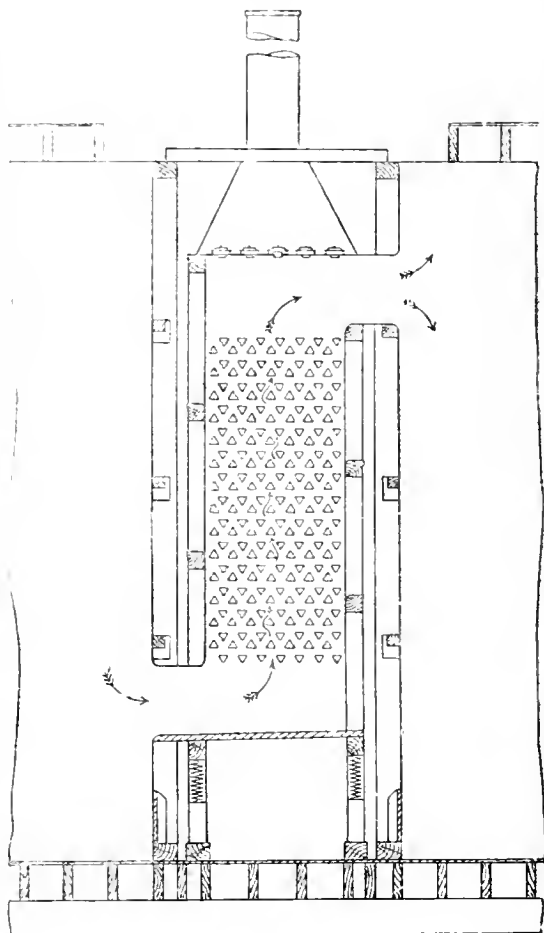
No. of Chamber.	Front.	Back.
	° F.	° F.
1	268	210
2	176	173
3	132	120
4	101	100

Lowest atmospheric temperature 71° F., highest 97° F. Oxygen 5.5 per cent. Nitre, 3.5 per cent. Escape gases, 63 grains sulphuric anhydride per cubic foot.

The amount of acid made in the first and last pipe case could not be measured, the pipe column between the first and second chamber at normal work showed 1,231.08 pounds of 50° Beaumé acid per 24 hours. The column between the second and third chambers, 1,125 pounds of 50° acid, and the one between the third and fourth chamber 472.1 lbs. 50° acid, or a total of 2,831.18 lbs.

The total capacity of the three columns is 583.2 cubic feet, which gives a yield of 8.85 per cent. of the total make of acid, and on a basis of 21 cubic feet per pound of sulphur, equals 226 times as much chamber space as the towers occupy, without taking into consideration the acid that is produced in the Glover tower.

But this is not the only results obtained by the columns, for the gases as they leave the columns have become so thoroughly mixed, that a stronger reaction has set in, so that the yield of acid in the chamber following is much above the average, being plainly shown by the yield of acid at drips, corresponding with drips of the same capacity and location in ordinary chambers.



No. 8. IMPROVED PIPE COLUMN.

The chamber working is much simpler with the columns, being less liable to oscillations, for a disturbance in any part of the chambers is generally neutralised by the gases simply passing through one of the columns.

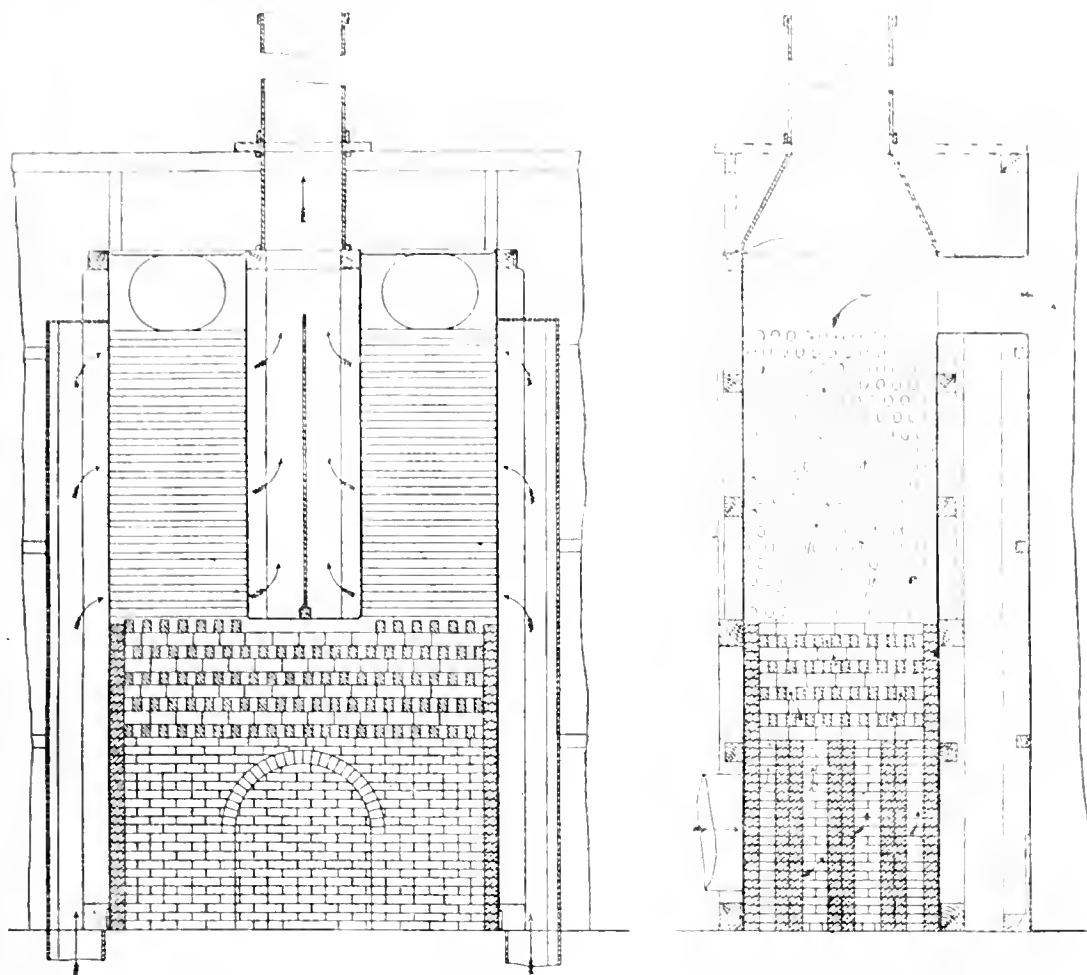
They require no special attention, especially when steam is used as a means of introducing moisture, as was the case in the foregoing citations, the ordinary drips of the chambers being all that is necessary to regulate them, even when the steam is introduced from the top of the chamber, the nozzle pointing towards the inlet, so that the steam enters along with the gases, for this affects the drip at the end of the chamber, and as long as this is kept steady the column is also, for it bears a corresponding relation to the drip.

As we usually use a reducing valve for the steam, so as to always get a normal pressure, it was found on one of the sets of chambers on this system, that it ran for four consecutive weeks without any of the steam valves being touched, the drips scarcely varying one degree.

In giving the temperatures of the various chambers in the Darlington and Savannah citations, they are all taken at the same relative positions at a height of 5 feet from the floor, and 6 inches in the chamber.

As the passage ways, separating the chambers, are at the outside 7 feet wide, it is interesting to note how great a change takes place in this short distance, it shows that the pipe columns effect a large amount of cooling.

All of the plants mentioned have not been tested to their fullest capacity, as the furnaces have been the limit in all instances, and especially as it so at the Charleston and Savannah works.



No. 9. IMPROVED PIPE COLUMN TO BE PLACED BETWEEN THE GLOVER AND THE FIRST CHAMBER.

So far I have only put one column between each chamber, and a small pipe case between the Glover tower and the first chamber, these at the most having only 150 pipes, with smooth surfaces.

Further recent improvements consist in using corrugated pipes, in lieu of plain pipes, with a strengthening rib in the centre, as they present relatively larger surfaces to the gases, than can be got by plain pipes. See Fig. 7.

Again, on the top of each pipe, when burnt to the side of the column, a saucer is formed, the object being to retain a greater amount of moisture, or rather weak acid, the pressure of which will still improve the reaction going on in the column. In looking at section of pipe No. 1 in sketch 7, it is apparent that a larger area of moisture is presented than if plain, upon which the gases impinge, and again the ribs on the sides of the pipes form a rough surface, which will enable to attain a better mixture of the gases.

Now I propose to put two pipe columns, as shown in sketch No. 8, side by side, between the chambers, still retaining the same size, but embodying the new improvements, using section of pipe No. 1 which has 16 per cent. more surface than the plain pipes used, increasing the number of pipes in each column 35 per cent., or a total increased exposed surface of 56 per cent., this would make the increase by using two towers 3.32 times as much.

Instead of a small pipe case between the Glover tower and the first chamber, I propose to substitute a double column as shown in sketch No. 9, using section of pipes No. 2 or No. 3, so that in case of much dust coming over from the Glover it would not remain on the pipes, this would increase the amount of work done 5.93 times as much as formerly.

A little of the nitrous acid passed down this tower would be of advantage, care of course being taken that denitration is fairly complete, as the more nitrous gas present the more work can be accomplished.

It is not only important to do as much work as possible whilst the gases pass from one chamber to another, but to do it as early in the process as possible; and as the columns will always have proportionally, a larger percentage of oxygen and nitrous gas than in the surrounding chamber space, it becomes an important factor in the chamber arrangements.

The limit of reduction of chamber space by this system, stands in the ratio to the number of columns used, ten cubic feet of chamber space per one pound of sulphur per twenty-four hours, can easily be obtained with a limited number of columns. How much lower than this by using additional columns I cannot say, but it is most probable that it would be very much lower.



The important question in all diversions from usual methods, is costs? When land is dear, a small structure is advisable, but in any case under this system, the cost is much less than when ordinary chambers are erected, especially is it so when climatic changes render it necessary to have expensive buildings, so as to protect the chambers from excessive heat and cold, as is necessary in America, and on the continent.

These pipe columns can be readily added to existing plants, so that increased production can be obtained without additional chamber space.

#### DISCUSSION.

Mr. GEORGE E. DAVIS said he had listened with very great interest to the paper, but was strongly of opinion that the inventors had not made out their case. Some months since he had carefully studied some figures that appeared from the pen of Mr. Gilchrist in the journals of the American Chemical Society for November 1893 and July 1894, and they appeared to be identical with those given in the paper that night with the exception of those of the Savannah Guano Company. He did not think it right to compare a new mode of manufacture with such antiquated methods as working without nitre recovery; the latest and most improved installations should be used for comparison, and they had in this country many vitriol works (working without the aid of pipe columns), giving results that would evidently astonish the inventors of these appliances. In the old-fashioned days in which he began his career as a vitriol maker (working without nitre recovery), 24 cubic feet per lb. of sulphur per 24 hours were considered the normal working space requisite, and if any less than this was provided, the nitre consumption was abnormal, while, on the other hand, if the space was increased, very little (if any) good was experienced. It was, moreover, well known at that time that the more nitrous the chambers were kept, the more work could be done in a given space. For instance, in works A, six chambers, 34 cubic feet per lb. sulphur nitre consumption, 8.2 per cent.; works B, six chambers, 22 cubic feet per lb., 8.4 per cent. nitre; works C, 20 cubic feet per lb., 9 per cent. nitre. Each of these was the average of six months working. According to the present paper the new works of the Darlington Phosphate Company appeared to have no system of nitre recovery, and to consume 13 per cent. of nitre when working with about 16 cubic feet per lb. of sulphur. As to the results of the working of the Savannah Guano Company, where they had pipe columns and Glover and Gay-Lussac towers, the description seemed a little ambiguous; the author appeared to say "for a time the plant worked below 15 cubic feet," and the nitre consumption was 3.5 per cent. To his mind there was nothing startling in the foregoing work, for to his own knowledge there was a works in Widnes in 1876, using as little as 2.9 per cent. of nitre; but as he could not be sure of the exact chamber space he refrained from quoting it further. Of one works, however, he could speak, wherein 63 tons of pyrites were burned weekly in three chambers working with 14 cubic feet of chamber space per lb. of sulphur, wherein the nitre consumption was only 1.8 per cent. This was not accomplished by the aid of pipe columns, but by the provision of ample absorbing space which was the real solution of the problem of how much acid could be made in a given chamber space. He had designed and re-organized a good many vitriol plants during the past 20 years, and there was no difficulty in working with a normal amount of nitre in as low a chamber space as 8 cubic feet per lb. of sulphur, and without pipe columns, a result twice as good as the lowest capacity mentioned in the paper that night. Of course, starting with a normal chamber space, the per-centage of nitre necessary would increase as they overloaded the chambers with work, and of this he had had very good experience. In one works with three chambers, the per-centage of nitre used, over a period of three months, when working with 15 cubic feet per lb. sulphur was 3.4 per cent. For a further period, when the space was reduced to 10 cubic feet, the nitre consumption was 5 per cent.; while when the pyrites was further increased so that the space was reduced to 8 cubic feet per

lb. of sulphur, the nitre consumption ran up to 7.1 per cent. If the absorbing space had been increased as the chamber space was reduced, the nitre consumption would have remained nearly normal. But it must not be forgotten that as the chamber space was reduced so the per-centage of oxygen in the waste gases increased, and the problem of absorbing the nitrous gases from a weaker mixture had to be encountered, and this was not, even at the present moment, sufficiently recognised.

He verily believed that the importance of the pipe column had been over estimated, the crux being the complete absorption of all the nitrous gases that it was possible to absorb. If this were done the amount of vitriol that could be made in a given chamber space was simply astonishing to practitioners of the old school.

Every vitriol maker knew that if the chambers were underworked, a very pale straw colour only was requisite in the final chamber, while when the chambers were "pressed" it was necessary to work them much darker in colour; in fact, working with a normal chamber space, nitrous gases equal to 200 lbs. of nitrate of soda had to be sent into the chambers for every ton of sulphur entering at the same time. In a plant working without nitre recovery, this quantity must all be "potted," but where absorbing and denitrating towers exist, the 200 lbs., say  $x$ , was made up of two quantities  $y$ , the amount potted, and  $z$ , that introduced through the medium of the Gay-Lussac acid. With an abnormal chamber space, a factor  $n$  would readily let them see what should be potted, and how much should be introduced into the Glover tower, as nitro-sulphuric acid. If  $n$  equalled that factor, and  $x$ , the total nitre to be introduced, then— $nx = y + z$ , when  $n = 1$ ,  $x = 200$ .

The paper made some reference to the necessary draught for chambers. It was, however, quite clear that no constant formula could be laid down for an abnormal chamber space; still, with a normal chamber space, he firmly believed no better rule could be followed than the old fashioned one of working the damper so that the gases in the last chamber "scarcely knew their own mind," i.e., were almost in equal balance with the atmospheric pressure.

The CHAIRMAN said that Mr. Gilchrist appeared to have been aware of the rocks on which previous application of the principle of mixing and scrubbing the gases on their passage from chamber to chamber had broken down. In the Thyss tower, the grids being too close together, the draught of the burners was obstructed too much, and the heat developed caused great wear and tear of lead. Both these drawbacks were overcome in Mr. Gilchrist's pipe column. Their importance in saving chamber space did not seem to be due so much to the work they themselves performed in percentage of acid made, as in scrubbing out the mist of nitro-sulphonic acid, decomposing this with the steam present, and in liberating nitrous acid again for fresh work in the next chamber. Mr. Gilchrist, at his request, made arrangements for measuring the acid, and running from the three towers at the Savannah Guano Company's works, the latest plant erected, and this amounted to 8.5 per cent. of the total make made in 583 cubic feet of space. He also, at his request, gave the figures of air temperature in summer working, the time of most unfavourable conditions for acid-making even in England, and much more so in the Colton belt, where in this case the temperature ranged from 71° to 97° F. As regarded the similar piece of apparatus of Professor Lunge, the Platt tower tried at Widnes, the results were not encouraging as to the amount of work done by the tower itself (*cide* Journal, 1893, p. 991), for Mr. Carey reported that the difference in total acidity between inlet and outlet was only about 2 grains  $\text{SO}_3$  per cubic foot, 30 inlet, 28 outlet, and of the 30, 14 grains only existed as  $\text{SO}_3$ , the remainder being in the form of mist as nitro-sulphonic acid. There was, moreover, no apparent effect in the reduction of chamber space necessary, as in Mr. Gilchrist's results, or as in the results Professor Lunge gave of the working of the Platt towers at a Bohemian works placed between a set of two chambers, where the second chamber men had next to nothing to do; it made no acid, serving as a drying chamber before Gay-Lussac's columns. He considered the result of low-chamber space combined with the nitre

economy, when working under such trying atmospheric conditions, bore remarkable testimony to the efficiency of the apparatus Mr. Gilchrist had described, and in adapting which to a set of new chambers he had carried out Professor Lange's recommendation to have chambers about half the present length of 120 feet, though the discussion of the validity of the reasons which Professor Lange gave for adopting this form would lead them too far that night.

## ON A NEW CLASS OF LAKE PIGMENTS.

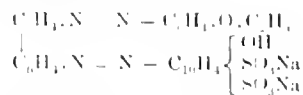
BY CARL OTTO WEBER, PH.D., F.C.S.

IN A paper published some time ago\* I discussed the question of producing fast shades on cotton with the benzidine dyes in particular and the acid dyes in general. I pointed out that this problem would be solved by the discovery of some compound which would be as satisfactory a lake former for the sulpho group, as tannic acid is for the amido group, or as alumina or chromic oxide for the orthodihydroxyl group. There are, of course, already compounds known which, in many cases, are capable to act as lake formers in the sulpho group, barium in the form of barium chloride is very extensively used for this purpose in the manufacture of lake pigments. But the chemical behaviour of these barium lakes, simply being that of barium salts of colour acids, and the attempts of converting these lakes into compound lakes like the tannin-antimony or alumina-lime lakes so far having proved fruitless, it is evident that the fastness of these barium lakes against acids and alkalis can be a slight one only, so that they are quite unsuitable for textile purposes. The fact that all sulphonated dyes, without exception, are wool dyes, forming, as I have shown in a former paper (this Journ., 1894, 120), lakes with the substance of the wool itself unmistakably indicates the class of compounds, amongst which we may expect to find a lake former generally applicable for all sulphonated dyes.† If we remember that lanuginic acid, a product of the decomposition of some of the proteids with baryta, is capable of forming excellent lakes with carboxylated and sulphonated dyes, there is little room for doubting that a lake former generally suitable for lake formation with all acid dye stuffs may be found amongst the large class of compounds classified under the head of proteids. The whole of our experience regarding the dyeing of wool and silk endorses this view, which, in the face of the remarkable lake-forming properties of lanuginic acid, may even be extended to the amido acids resulting from the decomposition of the proteids by acids and bases. It must, however, not be assumed that lanuginic acid already answers all our requirements for a lake-former in the sulpho group. In the first instance there is the difficulty of producing this compound sufficiently cheap for practical purposes, and further the vastness of the lanuginic lakes still leaves very much to be desired.

In the paper above referred to, I ventured the opinion that the remarkable fastness of the benzidine dyes when printed on calico with albumin thickening, the goods being steamed afterwards, could not very well be explained by the assumption of the coagulated albumin acting as a protective cover for the dye, but that a true chemical action (lake-formation) between the albumin and the dye was highly probable. It is even possible that the steaming of the albumin colour causes a deeper alteration of the albumin than its mere coagulation, and that the products of such an alteration then act as lake-formers. Indeed I have shown in the paper already referred to that conversion products of albumin, such as peptones, possess the property of forming well defined and very beautiful lakes with the

sulphonated dyes. I therefore considered this subject of sufficient theoretical and practical interest to examine the lake-forming properties, first of the best known of all proteids, of egg albumin, which I may add were found to be practically identical with those of blood albumin.

The series of experiments was begun with the study of the behaviour of one of the substantive dyes against albumin. The dye used was Cassella's diamine scarlet B., the product of the combination of tetrazodiphenyl with phenol and  $\beta$ -naphthol-7-disulphonic acid, the phenol-hydroxyl of this compound being finally ethylated. The formula of this dye is therefore—



This dye was selected first on account of its great solubility in water, so that fairly concentrated solutions remain perfectly clear even in the cold, and further on account of its not forming a precipitate on the addition of dilute acids. These properties of the dye obviously remove any liability to mistake the cause and nature of a subsequently observed precipitate. Moreover, there are in this dye besides the sulpho groups no other lake-forming groups, since the isolated hydroxyl group, as I showed in a paper read before this Society (this Journal, 1893, 659) some time ago, has no lake-forming properties whatever.

On adding a solution of diamine scarlet to a solution of egg or blood albumin, a precipitate occurs either at once or on standing, according to the concentration of the solutions. This precipitate is rather slimy, and therefore very difficult to filtrate. Neither is the complete precipitation of the dye possible without using an enormous excess of albumin; but by heating the incompletely precipitated mixture to 80° C. on the water bath, complete precipitation of the dye takes place, and the albumin lake separates out in the form of bright scarlet flakes. This lake can be easily filtered and washed, the filtrate, even when using boiling water for washing, being perfectly colourless. As might be expected, the fastness to soap of this lake is rather unsatisfactory. Treatment with soap solution (10:1000) at 50° C. slowly decomposes it. Equally sensitive is this lake against the action of caustic alkalies, ammonia, and alkaline carbonates. On addition of acetic acid, however, to these solutions the lake is again precipitated with all its original properties. On drying this lake on the water-bath or in an air-bath, or in vacuo at ordinary temperature, it diminishes greatly in volume, and is eventually obtained as a dark red horny mass, which, however, in the mortar is easily reduced to a fine powder of a very deep and brilliant scarlet colour. When suspended in water the dried lake, even on prolonged boiling, shows no sign of swelling, neither does the water show any coloration. Soap solution at 80° C. scarcely affects the lake, only on prolonged boiling the soap solution exhibits a very slowly-increasing coloration. Dilute solutions of the alkaline carbonates, even at boiling point, are without any appreciable action, but hot solutions of the caustic alkalies dissolve the lake rather quickly.

It must, however, be observed that this scarlet lake prepared as above described, cannot be a uniform body. According to the amount of the excess of albumin used, this lake evidently represents a mixture, and probably not even a homogeneous one, of the lake proper with coagulated albumin. It appears that the precipitation above described takes place in two stages. In the first instance, the lake proper separates out, which is later on enveloped by the coagulating albumin. This, no doubt, is the reason of the depth of coloration of the lake entirely depending upon the quantity of albumin used for precipitation; but this being so it is of course possible that the albumin lake I obtained differs in its properties more or less from these lakes in the pure state, and it might further be objected that the precipitates I obtain, in the manner above described, are no lakes at all, but merely occlusions of colouring matter in the coagulated albumin. It was, therefore, important to devise a method for the preparation of these lakes in a state of purity. This is easily done by adding an excess of

\* Chem. Zeit. XVIII., 1894, pp. 528, 563.

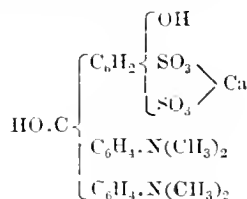
† Barium chloride is not so applicable, many of the sulphonated dyes not being precipitated by it.

acetic acid to the mixture of the solutions of albumin and dye. On the addition of the acid the scarlet lake is immediately precipitated, and the precipitation, even when working in the cold, is nearly complete. Neither the scarlet nor albumin being precipitated from their solutions by acetic acid, there can no longer be any doubt as to the precipitate formed under these conditions being an albumin lake. On heating the acidified mixture on the water-bath, the precipitate which at first is rather curdy, becomes increasingly finer in texture, and forms eventually a mass of exceedingly fine particles. At this stage every trace of the colouring matter has been precipitated. Providing a sufficient quantity of acetic acid having been used, this precipitate cannot contain any free coagulated albumin, and it must therefore represent the albumin lake in a practically pure state. The precipitate is now filtered and washed neutral, first with warm then with boiling water. The precipitate upon the filter does not possess any sliminess, and has no tendency to clog or cake as do the precipitates containing free albumin. On drying this lake, it also shows, however, the property to shrink very considerably in volume, and when perfectly dry to form horny pieces which are, however, distinguished from the lakes containing free albumin by their much deeper and at the same time brighter colour. The physical condition of many lakes in the dry state depending very much upon the nature of the liquid evaporating from them during the drying process, it appeared feasible that by expelling the water from the lake, instead of by evaporation by displacement, first with absolute alcohol, and then with ether, better results might be obtained. But this treatment was found not to have the desired result, and it seems that after the removal of the extraneous water the lake represents the conditions of a hydrate which, on subsequent drying, loses its water of hydration, and is converted into an anhydride, a process which, in the case of these lakes as in other cases with which we are more familiar,\* is accompanied by a very considerable shrinkage in volume. The horny pieces of pure albumin lake are easily pounded into an almost impalpable powder possessing a very strong and brilliant scarlet colour. The chemical behaviour of this pure lake is practically identical with that of the lake containing free albumins.

The behaviour of albumin against diamine scarlet is typical of its behaviour against all sulphonated or carboxylated dye stuffs, whether they belong to the benzidine series or not. Consequently the sulphonated basic dyes, the numerous azo dyes, and all sulphonated nitro colouring matters without exception can be converted into albumin lakes in the above described manner. This is very much more than any of the lake formers for acid dye stuffs known at the present time will do, and this method of lake formation will no doubt attain to some importance in the future, especially for the manufacture of paste colours for stained paper, and wall paper, and it will, I believe, also be of considerable interest to calico printers for the production of some very useful styles, ranging between the present mordant and pigment styles. The fastness to light of these albumin lakes far surpasses that of the corresponding barium lakes, and all those acid dyes which like crystal ponceau, patent blue, naphthol yellow, naphthol green, and scores of others are not precipitated at all by barium chloride, and for this reason are either totally unavailable at present for the purposes of lake making, or require the use of circuitous and expensive methods, leading after all to at best only moderately satisfactory results, all these dyes form readily excellent lakes with albumin, by the above described treatment. One of the most valuable features of these albumin lakes is the fact that the shade of the lake obtained with any acid dye exactly corresponds to the shade the same dye produces on wool or silk. Barium lakes many a time exhibit a disappointing difference in this respect, which in numerous cases rendered some of the finest dye stuffs quite useless for the purpose of the lake manufacturer.

It appeared now interesting to examine the process of lake formation in the sulpho group by means of albumin, also with such colouring matters which contain other lake forming groups besides the sulphur group. I showed in a former paper (this Journal 1893, 657) that dyes containing different lake forming groups in the molecule, in order to form satisfactory lakes, require lake formation in every different lake forming group. This is the reason why the precipitation of the sulphonated basic dyes with barium chloride is either impossible (acid magenta, Thioearmine, &c.) or yields indifferent results only. If, however, we effect lake formation in the amido group of these dyes, by means of tannic acid, at the same time as in the sulphur group by means of barium chloride, we obtain lakes which are in every respect satisfactory. But on the other hand I showed that in dyeing wool with sulphonated basic dyes, neither simultaneous nor subsequent treatment with tannic acid is required, in fact the dyed wool is utterly indifferent against it. From this I concluded that in the dyed wool, both the lake-forming groups of these dyes have undergone lake formation with the substance of the wool itself. Albumin should therefore be expected to behave similarly against this class of dyes.

On adding to a solution of Patent Blue B (Meister,



Lucius and Brining), a solution of albumin, it is found impossible to effect even an approximately complete precipitation of the coloring matter; indeed, on filtrating this mixture it is found that, although it has become rather turbid, almost the whole of the dye and the albumin pass together through the filter. On adding acetic acid in excess to this turbid mixture it becomes again quite clear, but on heating it on the water bath to 90° C. the beautiful albumin lake of patent blue B separates out. Only traces of the dye escape precipitation.

As we see from the above formula, patent blue B, besides the two lake-forming sulpho groups, contains also a lake-forming amido group, and the question arises as to whether both the different lake-forming groups, or only one of them, and which, contributed to the formation of the lake. Taking into account the observations made in a former paper (this Journal, 1893, 656), regarding lake formation with dyes containing several heterogenous lake-forming groups in the molecule, it is highly probable that the two sulpho groups, as well as the amido group of the patent blue, have undergone lake formation in the albumin lake. An experiment easily demonstrates the fact that neither barium chloride, nor tannic acid will precipitate patent blue, although a mixture of these precipitants will do this readily. This experiment, therefore, seems sufficiently convincing to assume that in the albumin lake both the sulpho groups and the amido group have suffered lake formation. This conclusion is essentially supported by the fact that tannic acid is without the slightest effect upon this lake, whereas the presence of a free amido group in it would, according to numerous former experiences, cause a material alteration of the shade of the lake. If, on the other hand, we assume for the moment the amido group of the dye to have undergone lake formation, but the sulpho groups still being in the free state, the absolute insolubility of such a lake would be quite inexplicable in the face of the fact that even such an energetic lake former in the amido group as tannic acid by itself absolutely fails to precipitate this dye. It will be seen, therefore, that albumin, like wool, must possess the capability to fix sulphonated basic dyes through lake formation in their sulpho group as well as in their amido group.

This, of course, involves that albumin must be capable of also forming lakes with ordinary basic dyes, not containing the sulpho group, just as we have already proved

\* Dehydration of aluminate or ferric hydrates. It is interesting to note that these hydrates which dry very much in the same way as the above described albumin lakes are capable to exist, like albumin, in a colloidal state.

its capability to form lakes with simply sulphonated dyes not containing any other lake-forming groups. To test this important point a solution of 10 grms. of egg albumin in 1000 cc. of water was mixed with a solution of 0.5 grms. of benzaldehyde green, 5 grms. of sodium acetate, and 5 cc. of acetic acid (80 per cent.) in 100 cc. of water. The mixture remained perfectly clear, but on heating it at 86° C. on the water-bath a precipitate rapidly increasing in quantity soon begins to form, and eventually the whole of the dye is precipitated in the form of a most brilliantly-tinted green lake. The behaviour of the other basic dyes is analogous, though it must be observed that the readiness with which the precipitation is initiated varies considerably for the different basic dyes, and the same is to be said of the completeness of the precipitation. This is a perfect analogy to the varying readiness and completeness with which the different basic dyes are taken up by wool. These lakes of the basic dyes with albumen exhibit a remarkable resistance against dilute inorganic and organic acids. Decomposition only takes place on prolonged boiling of the lakes with dilute sulphuric, hydrochloric, or nitric acid. The fastness against alkalis of these lakes surpasses that of the lakes of the sulphonated dyes; they are particularly resistant to soap solution.

The above offers conclusive proof of the existence of albumin lakes of the basic dyes and further furnishes additional evidence of the striking analogy between the lake-forming properties of albumin and wool and silk. We can, therefore, no longer doubt that in the albumin lakes of sulphonated basic dyes the lake formation extends to both the sulphur group and the amido group. The capability of albumin to form lakes with basic dyes cannot be considered to be for itself of any practical importance in view of the excellent qualities of tannic acid for this purpose. The fastness of the albumin lakes of basic dyes is not superior to that of the corresponding tannin lakes and while the latter on treatment with antimony salts are converted into compound lakes of considerable fastness, no similar treatment and conversion is at present possible for albumin lakes. Nevertheless there might under certain circumstances an albumin lake be preferable to a tannin lake, owing to the oftentimes surprisingly great difference between the shade of the albumin lake and the tannin lake of the same dye, which difference is identical with that exhibited by the same dye when applied to cotton and wool or silk.

Of the colouring matters the lake-forming properties of which are merely a function of hydroxyl groups, the eosin group in its tinctorial behaviour stand nearest to the acid dyes proper. On adding to a solution of 10 grms. of albumin in 500 cc. of water a solution of 1 gm. of eosin GGF (Casella) in 100 cc. of water no precipitate appears, and although addition of acetic acid causes a slight turbidity, the mixture passes completely through a filter. On heating in the water bath a distinct precipitate appears at 85° C. and at 90° C. the eosin is completely precipitated. This illustrates in a particularly striking manner the remarkable lake forming power of albumin as, excepting the salts of the heavy metals, the precipitation of eosin by means of inorganic salts is either very incomplete or fails altogether.

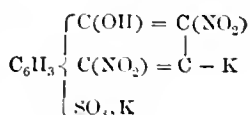
I have repeatedly mentioned that the isolated hydroxyl group is a most indifferent lake former. For lake formation it is practically available only in the nitro-colouring matters, and even in these cases it is rather doubtful whether indeed the hydroxyl group is the lake-forming group and not a hydrogen atom to which the proximity of the nitro group imparted acid functions similar to those possessed by the hydrogen atoms of the halogen acids. This appears highly probable if we remember that the acidifying tendency of the nitro group is strong enough to convert so unquestionable a base like diphenylamine into an equally unquestionable acid like hexanitrodiphenylamine. A similar phenomenon appears in the fact that naphthol yellow S

is not as might be expected a monobasic acid, but a strong dibasic acid. The nitro colouring matters have so far proved of small importance for the manufacture of lake-pigments, owing to the lack of a satisfactory precipitant. All the more remarkable is therefore the fact that naphthol yellow S which in this respect proves particularly refractory, easily yields most beautiful lakes with albumin. The mixture of the solutions of albumin and naphthol yellow remains perfectly clear, but already on addition of acetic acid, an abundant precipitate begins to form. Heating on the waterbath for a short time completes the reaction. It is interesting to observe that the shade of this lake is again identical with the one produced by naphthol yellow on wool, whereas the inorganic lakes of this dye are conspicuous by their disagreeable brown or orange shade.

We now come to that interesting class of dye stuffs which besides the sulfo group contain the orthodihydroxyl group. That the sulfo group of these dyes will be capable to enter into lake combination with albumin appears practically certain, but the question as to the behaviour of the orthodihydroxyl group itself, and also its possible influence upon the lake-formation of the sulfo group requires examination. The incapability of ordinary alizarin to dye un mordanted wool renders the possibility of lake formation between the orthodihydroxyl group and albumin also rather improbable, but it nevertheless appears worth while to establish this fact beyond doubt by direct experiment. On account of its insolubility in water, the use of ordinary alizarin paste in this experiment will have to be avoided as conducive to errors, and our first task is therefore to find a means to bring the alizarin into aqueous acid solution, the acid of course again merely serving the purpose of preventing coagulation of the albumin. Very dilute solutions of this description are obtained on acidifying alkaline, preferably ammoniacal, solutions of alizarin with acetic acid. Better results are, however, obtained by acidifying with acetic acid solutions of alizarin in borax. By this means strongly acid solutions of alizarin of considerable concentration can be prepared. On adding such a solution of alizarin to a solution of albumin no precipitate appears, nor does the heating of this mixture to 96° C. even when prolonged for hours, produce the looked forward to result. It is therefore evident that the orthodihydroxyl group does not carry the property of forming lakes with albumin. In the face of this fact the behaviour of alizarin sulphonic acid, alizarin S, against albumin appears particularly interesting. The sulfo group of this dye places the possibility of the formation of an albumin lake in this group of the dye beyond doubt; but such a lake would still contain the free orthodihydroxyl group, owing to the above shown incapability of this group to form an albumin lake. An albumin lake of Alizarin S would therefore possess all the properties of ordinary alizarin.

On adding a solution of alizarin S to a solution of albumin, no precipitate occurs, but the mixture assumes a deep crimson colouration which, however, on addition of acetic acid changes into brown. Many hours heating of this mixture on the water-bath does not produce the slightest trace of a precipitate; but on the addition of alumina sulphate or acetate, a copious Turkey red-coloured precipitate is thrown down. This precipitate is the albumin-alumina lake of alizarin S. The fastness of this lake against alkalis and soap exceeds that of the previously described albumin lakes, but is much inferior to that of alizarin S dyed upon wool.

This behaviour of alizarin S against albumin is particularly interesting, as it furnishes a striking confirmation of the result I arrived at in a former paper (this Journal, 1893, 636), namely that lake-formation in dyes containing heterogeneous lake-formers in the molecule is only possible by providing for each of the different lake-forming groups according to their specific lake-forming tendencies. Thus I showed that sulphonated basic dyes which should be able to form lakes with either barium or tannic acid do neither, but are readily converted into most beautiful lakes on simultaneous treatment with these two lake-formers. Similarly we find now that the prospective capability of



alizarin S to form lakes with either albumin\* or albumin† cannot be realised, but that these two lake-formers combined at once produce the intended result. Albumin being as I showed not only a lake-former for the sulpho group, but also for the amido group is therefore by itself able to precipitate sulphonated basic dyes in spite of their heterogeneous lake-forming groups. But against alizarin S albumin behaves exactly as barium chloride against sulphonated basic dyes; no precipitate occurs until the lake-forming tendencies of the coexisting heterogeneous group, in this case the orthodihydroxyl group are satisfied.

The observations made in the above experiments already contain the material for the elucidation and explanation of a number of facts in connection with wool and silk dyeing. But before entering upon this subject it will be advisable to extend this investigation to some other members of the proteid class, an intention which I hope to put into operation before long.

### DISCUSSION.

Dr. WEBER, in reply to a question raised by Mr. Flintoff, as to whether the lakes produced in the manner referred to in the paper were fugitive or not, said that in a former paper he had pointed out that it was not the dye alone which determined the fastness to light, but the lake-former with which it was combined. In dyeing Diamine fast red on cotton, an extremely fugitive shade was obtained, whereas if it were dyed on wool one got one of the fastest scarlets it was possible to dye. As a matter of fact, on the Continent, fast red on wool was considered to be almost as fast as alizarin. In dyeing diamine or benzidine scarlets on cotton, one always got more or less bright shades which were more or less fugitive; but, if on the contrary, one dyed these scarlets on wool one got a fast colour. It was an absolute truth that the fastness of the dye was not an inherent quality of the dye alone, but depended very much upon the way in which the latter was applied. In other words, unless the dye had a chance to enter into lake-formation one could not expect a satisfactory degree of fastness. This was a fact very clearly borne out by Hummel's researches "On the fastness of artificial dye stuffs," and in most cases the fastness of any dye that could be applied to wool was immensely greater than the fastness of the same dye on cotton. The albumen lakes were also at least four times as fast to light as the corresponding barium lakes. The reason for this it was difficult to state, but the fastness was there. This relation between the fastness of a dye and its lake-formation was strikingly shown by what he had termed "half-lakes," as, for instance, the barium lake of a sulphonated basic dye. Such a lake was exceedingly fugitive, owing to the lake-forming amido group being left in the free state. If one added to such a half-lake tannic acid, this amido group underwent lake-formation, and the fugitive half-lake was now converted into a fast compound-lake.

\* In the sulphonic group. † In the orthodihydroxyl group.

## Newcastle Section.

Chairman: G. T. France.  
Vice-Chairman: Alf. Allhusen.

### Committee:

P. P. Bedson.	B. S. Proctor.
T. W. Hogg.	W. W. Proctor.
T. W. Lowibond.	W. L. Kennoldson.
W. Martyn.	W. A. Rowell.
John Morrison.	T. W. Stuart.
John Pattinson.	John Watson.

Hon. Local Secretary and Treasurer:

Saville Shaw, Durham College of Science, Newcastle-on-Tyne.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

Meeting held in the Durham College of Science,  
Newcastle-on-Tyne, on Saturday, November 24th, 1894.

MR. GEO. T. FRANCE IN THE CHAIR.

### PRESENTATION TO DR. J. T. DUNN.

DR. DUNN, who has occupied the position of Honorary Secretary and Treasurer to the Newcastle Section since its formation, has been obliged to resign that office in consequence of his appointment to the Headmastership of the Plymouth Technical Schools.

After letters of regret for non-attendance had been read from Sir Lowthian Bell and Mr. Alfred Allhusen—

The CHAIRMAN said that they were met together to do honour to Dr. Dunn. There were few members then present who were present at the founding of the older Newcastle Chemical Society, to which for three years before and 11 years after its amalgamation with the Society of Chemical Industry Dr. Dunn had acted as Secretary. During these 14 years nothing had ever transpired to mar the complete confidence they felt in him, and they all had had occasion to admire the thorough but quiet and unostentatious way in which the duties of the secretaryship had been performed. He would call upon the Secretary to read a letter which had just been received from the General Secretary on behalf of the President and Council of the Society. The Council wished to place on record their appreciation of the valuable services Dr. Dunn had rendered to the Society and to join with the Newcastle Section in their expressions of goodwill and gratitude towards him.

He had much pleasure in handing to Dr. Dunn, on behalf of the subscribers, a silver kettle, together with a purse of gold as a small token of the esteem in which he was held amongst them.

The kettle bore the following inscription:—

"Presented to J. T. DUNN, D.Sc. (together with a purse of gold), by the Members of the Newcastle Section of the Society of Chemical Industry, as an indication of the high esteem in which he is held and in recognition of his valuable services as Honorary Secretary during the past 14 years, 24th November 1894."

MR. JOHN PATTINSON had been associated with Mr. Dunn both during his connection with the old Society and since it was merged into the Society of Chemical Industry. No friction had ever arisen in any part of Dr. Dunn's work and the Committee had always felt that they could place perfect reliance upon his judgment and prudence. The spontaneous way in which the Members of the Section, which was not a large one, had contributed towards the testimonial fund, showed how they all valued his work.

He (Mr. Pattinson) wished to express his personal regret at losing Dr. Dunn from the North, though the fact that he was leaving them to fill an important educational post was

some slight compensation. He trusted that he would find Plymouth as pleasant a place as Newcastle had been to him.

Prof. P. P. BENSON, D.Sc., though he felt that he inadequately represented that body, would yet like to say a word on behalf of the Durham College of Science, of which Dr. Dunn was one of the most distinguished students as he also was the most distinguished graduate in science of Durham University. Dr. Dunn was a colleague of his for eighteen months, and during that short time he formed a high opinion of his powers which a friendship of nearly twelve years only served to confirm. He entertained a high opinion of Dr. Dunn's work in the educational world, and was glad to think that he was to carry the name and fame of the College into southern climes.

Dr. Dunn, in reply, said that it was very gratifying to him to receive such a testimonial from the Members of the Section with all of whom his relations had always been pleasant. Since the time, (exactly 14 years ago) when he first joined the old Society it had undergone many changes. In the old days the Society at Newcastle edited and published its own transactions, and he was selected to act as its secretary on account of his combining a knowledge of chemistry and of shorthand. The work had been always to him a pleasure and it was a very great pleasure to him to think that his efforts had been appreciated not only by the local members, who had given him such a valuable token of their esteem, but also by the Council of the Society. He could not but think that they had spoken of his work in terms beyond his deserts. That all had gone smoothly was really due to the good feeling existing amongst those with whom he worked. Though he hoped to find Plymouth the pleasant place it had been pictured to him, there was no danger of his ever forgetting the city of his birth, Newcastle, where so many years of his life had been spent.

## Nottingham Section.

### UNIVERSITY COLLEGE, NOTTINGHAM.

Chairman: J. M. C. Paton.

Vice-Chairman: C. Spackman.

#### Committee:

L. Archbutt.

F. J. R. Carulla.

F. Clowes.

J. B. Coleman.

H. Forth.

J. F. Kemyson.

T. Mason.

J. O'Sullivan.

S. J. Pentecost.

C. Taylor.

G. J. Ward.

Treasurer: J. T. Wood.

Hon. Local Secretary:

R. L. Whiteley, University College, Nottingham.

#### SESSION 1894-95.

January 1895.—Mr. Jas. O. Sullivan: "Note on the Estimation of Cane Sugar."

Meeting held at University College, Nottingham,  
on Wednesday, November 21st, 1894.

MR. J. M. C. PATON IN THE CHAIR.

### THE MINIMUM PROPORTIONS OF CARBON-DIOXIDE AND OF NITROGEN IN AIR WHICH ARE EXTINCTIVE OF DIFFERENT FLAMES.

(ABSTRACT.)

PROF. CLOWES gave an account of a series of experiments carried out by himself and M. E. Feilmann, B.Sc., with the object of ascertaining the minimum proportions of

carbon dioxide and of nitrogen, which, when mingled with air were extinctive to the flames of various combustible liquids and gases. He explained the method of experiment, which consisted in mixing the measured volumes of air and of the gas by the motion of a light ball, so as to avoid the solvent action of water on carbon-dioxide. After proving that the method yielded mixtures accurately in the desired proportions, the trials were made by introducing the experimental flame into mixtures of air and gas, which were varied in proportion until the flame was immediately extinguished, but continued burning for a short time in a mixture containing 1 per cent. less of the extinctive gas. The influence of the products of combustion was by this procedure eliminated. It was found that the size of the flame had no influence on the extinctive proportions.

The table on page 1156 summarises the average results obtained.

The results obtained show:—

(1) That carbon dioxide exerts a uniformly more powerful extinctive effect than nitrogen does. This may possibly be partly due to its high density, which renders the movement of air to feed the flame sluggish.

(2) That the different wick-fed flames are extinguished by a strikingly uniform proportion of the extinctive gas. These flames were noticed to gradually diminish in size and die down, evidently owing to the supply of combustible material to the flame being diminished in amount by the reduction of temperature in the flame.

(3) That the extinctive proportion for the various gas-fed flames varied widely, and did not appear to be deducible from the composition and chemical nature of the gas which was burnt. The extinctive proportion for a coal-gas flame was much larger than that for the wick-fed flames; whilst the extinctive proportion for the hydrogen flame was extraordinarily high.

The gas-fed flames increased in size during the process of extinction, instead of diminishing as in the case of the wick-fed flames. This appeared to be due to the fact that the supply of combustible was maintained independently of the flame itself, and that the flame had to increase its surface in order to obtain the supply of oxygen necessary for its combustion in air containing a diminished proportion of that gas.

Attention was directed to practical advantage derivable from the large proportion of carbon dioxide (58 per cent.) necessary to extinguish the hydrogen flame. The author had described an ordinary miner's safety-lamp, in which a hydrogen flame could be burnt at will instead of the oil flame for the purposes of delicate and accurate gas-testing. It is now found that by burning the hydrogen flame beside the oil-flame in parts of the mine where the atmosphere might contain large amounts of carbon dioxide, the extinction of the oil-flame was not attended with "loss of flame" in the lamp; since the hydrogen flame never suffered extinction and served to rekindle the wick in fresh air, after the oil-flame had been extinguished by a foul atmosphere. This had proved to be a great convenience to exploring parties in the mine after a fire or explosion; and it was the direct consequence of the resistance of the hydrogen flame to extinction by carbon dioxide.

Recent experiments (J. R. Wilson: American Journ. Pharm. 50, No. 12) made upon rabbits had thrown considerable doubt on the statement that the proportion of carbon dioxide in the air which extinguished an oil-flame or a candle-flame was irrespirable; this was a matter to be decided by physiological experiments. But it might be taken as finally established by the experiments described in the present paper that about 15 per cent. of carbon dioxide was at once extinctive to ordinary illuminating wick-fed flames; although 33 per cent. was requisite to at once extinguish a flame of coal-gas; whilst a hydrogen-flame did not suffer extinction until 58 per cent. of carbon dioxide was present in the air.

#### DISCUSSION.

MR. G. J. WARD said that they were in the habit of burning under their boilers waste gas from the blast furnaces. Roughly speaking the gas contains 30 per cent.

Combustible Substances Burnt.	Extinctive Proportion of Carbon Dioxide added to Air.			Extinctive Proportion of Nitrogen added to Air.		
	Percentage added.	Percentage Composition of Mixture, O : (N + CO <sub>2</sub> ).		Percentage added.	Percentage Composition of Mixture, O : N	
Air, dried, absolute .....	14	18.1	81.9	21	16.6	83.4
Alcohol, methylated .....	13	18.3	81.7	18	17.2	82.8
Paraffin, ordinary lamp oil .....	15	17.9	82.1	23	16.2	83.8
Colza oil with equal volume of petroleum .....	16	17.6	82.4	22	16.4	83.6
Candle .....	14	18.1	81.9	22	16.4	83.6
Hydrogen .....	58	8.8	91.2	70	6.3	93.7
Carbon monoxide .....	24	16.0	84.0	28	15.1	84.9
Methane .....	10	18.9	81.1	17	17.4	82.6
Ethylene .....	26	15.5	84.5	37	13.2	86.8
Coal-gas .....	33	14.1	85.9	46	11.3	88.7

of carbon monoxide, 6 per cent. carbon dioxide, 4 per cent. of hydrogen and hydrocarbons, and 60 per cent. of nitrogen. Approximately 85 per cent. of air is required to burn the mixed gases. After the addition of air the mixture would contain about 89 volumes of CO<sub>2</sub> and 11 volumes of oxygen in every 100 of non-combustible gases, a mixture which was extinctive for carbon monoxide according to Dr. Clowes' experiments, and yet combustion took place under their boilers. Could Dr. Clowes explain this?

Mr. R. L. WHITELEY did not agree with the argument that, because a rabbit could live in air which contained a certain large percentage of carbon dioxide, therefore a human being could also. He thought that animals accustomed to live above ground should be experimented upon if anything like satisfactory conclusions were to be arrived at.

Dr. STAFFORD said that there seemed to be some considerable difference of opinion with regard to this matter. Recent experiments tended to show that they had been accustomed to over estimate the dangerous qualities of carbonic acid gas as a constituent of respiratory air. Considerable quantities of CO<sub>2</sub> might be added to air before it becomes fatal to an animal breathing it. In this connection Dr. Whitelegge speaks of CO<sub>2</sub> in respiratory air as the "harmless index." It would appear that the use of oil-stoves for warming rooms—in place of open fires—is not so objectionable as might appear. Providing that the combustion of the oil is complete—the CO<sub>2</sub> produced does not appear to be harmful—fairly adequate ventilation being allowed for. Recent research seems to show that the deleterious properties of expired air lie more in the volatile organic material given off than in the CO<sub>2</sub> expired.

Mr. F. J. R. CARULLA referred to the power of the lamp for keeping a flame alight in atmospheres where the oil-flame went out, which seemed to the speaker an extremely valuable discovery. As bearing on that subject he would mention a case that he had read of. Somebody had fallen into a well, into which it appeared impossible for anybody to descend for the purpose of rescue, as candles were tried, but they went out on reaching a certain depth. Seeing this, one of the onlookers suddenly asked for an umbrella, much to the surprise of the others. This was supplied, and to the astonishment of the people around, one person opened it, tied a string to it, let it down into the well, brought it up and repeated the operation several times. The result was that the foul gas was displaced by the introduction of fresh air, and the atmosphere was rendered respirable. He was reminded of the incident when he saw Dr. Clowes mixing the air in the glass tube with a ball.

Dr. CLOWES, in reply, said that the conditions under which the furnace gas, mentioned by Mr. Ward, was burnt, were sufficiently different from those of his own experiments to account for the difference in the result. Part of the extinctive gases was mixed beforehand with the combustible gas, and the combustion of hot gas took place in a heated space, which would tend to maintain the flame. He remarked also that hydrogen was present as well as carbon monoxide. The conditions were therefore entirely different from those he had worked under, and the conclusions arrived at in the paper were not applicable to Mr. Ward's case.

Mr. FEILMANN stated that unpublished experiments, which were now in progress, proved that under different conditions the extinctive proportion was subject to variation.

The CHAIRMAN said that, besides in coalmines, men were overcome by irrespirable gases in distilleries, breweries, and other chemical works. The men were dragged out and brought round, but they had no means of knowing the composition of the atmosphere in which the man had been. He said, too, that it would not do for workmen to know that air which contained sufficient carbonic acid to extinguish flame did not necessarily destroy life. It would lead to their incurring carelessly unnecessary risks, and consequently there would be more lives lost through accidents.

## Yorkshire Section.

Chairman: C. F. Tetley.

Vice-Chairman: J. J. Hummel.

### Committee:

J. E. Bedford.	A. G. Perkin.
F. Branson.	C. Rawson.
J. B. Cohen.	F. W. Richardson.
T. Fairley.	G. W. Slatter.
W. Leach.	Thorp Whitaker.
W. McD. Mackey.	T. B. Wilkinson.

Hon. Local Secretary:

H. R. Procter, Yorkshire College, Leeds.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.



Meeting held at the Yorkshire College, Leeds, on Monday,  
November 5th, 1894.

PROFESSOR A. SMITHIELLS IN THE CHAIR

## THE BACTERIOLOGICAL ANALYSIS OF WATER. PART I.

BY F. W. RICHARDSON, F.R.S., F.R.M.S., ETC.

THE analysis of waters for their potability is one of the most vexed questions which the chemist is required to answer. Until within the last few years many chemists were under the impression that some five or six analytical data were sufficient to allow of the acquittal or condemnation of a water; yet it has been too often made painfully evident that the opinions of analysts differ very widely as to the character of a given sample; even when the same analytical data have been obtained. Some lay great stress upon the presence or absence of nitrites and nitrates, while others have laid down a fixed boundary for the free and albuminoid ammonia. To add to the confusion resulting from so many conflicting ideas the bacteriologists now adduce very strong facts to throw discredit upon the merely chemical analysis of a drinking water.

Drs. Dewar and Ogle (Report of the Commissioners to inquire into the Metropolitan Water Supply, 1893, § 141 and § 142), with other scientists, acting in an official capacity, have recently declared that the water-question has in fact passed from the domain of chemistry into that of biology; and, in confirmation of this, it is shown that waters containing large amounts of organic matter can be drunk by a population with impunity, while waters with so small a proportion of organic constituents as almost to defy detection may actually be of the most poisonous character.

In order to ascertain whether this were really the case, I divided a sample of Bradford town's water into two equal portions, and to one of these I added a drop of a typhoid (enteric) fever stool. The chemical analysis gave:—

	Unpolluted Water.	Polluted Portion.
Total solid matter..... Grains per Gallon.	7·6	8·9
Organic and volatile matter...	1·1	1·3
Saline matter .....	6·5	6·7
Chlorine.....	0·50	0·54
Nitrites.....	None	None.
Nitrates .....	None	None.
Free ammonia .....	0·03	0·08
Albuminoid ammonia.....	0·03	0·04

From these data the contaminated water would have been pronounced as "potable"; yet the bacteriological analysis gave:—

	Unpolluted Water.	Polluted Portion.
Bacillus typhi abdominalis..... In 1 cc.	None	All present. Total 68,430.
" coli communis .....	None	
" viridis pallescens .....	None	
" putrificus coli.....	None	
" fluorescens liquefaciens .....	12	
" albus.....	36	
" aerophilus .....	104	
Micrococcus aquatilis.....	100	
	312	

One finds micrococci so small that 16 trillions of them would only weigh a single grain, and if we assume that an average bacillus measures  $2\mu \times 1\mu \times 1\mu$ , 300,000 millions would be required to equal to this weight. Microbes contain 80 per cent. of water and 2 per cent. of nitrogen, and as by the Wanklyn albuminoid-ammonia process only a portion of the total nitrogen is liberated as ammonia, we are not far from the truth in stating that 0·01 mgrm. of ammonia thus obtained would represent 10,000 millions of these bacilli! In the case of actual pollution by infiltration through soil, we are aware that for every microbe present there is an incomparably larger proportion of decomposition products—such as free ammonia, nitrites and nitrates; nevertheless it is certain that great numbers of dangerous microbes are repeatedly found in waters given as pure by chemical analysis. Chautemesse and Widal found numerous typhoid bacilli during the epidemic at Pierrefonds in well-water, which contained only very small proportions of nitrogenous matter; and Macé (Traité Pratique de Bactériologie, 1892, 694) asserts that pathogenic germs can vegetate and multiply enormously in distilled water, utilising such minute proportions of organic matter that the most refined chemical methods fail to discover them. One domestic sewage gave me as many as 36 millions of microbes in 1 cc., and many of these were bacilli of the colon (*B. coli communis*, &c.); yet one part of this diluted with 1,000 parts of a good drinking-water increased the organic constituents so slightly that the quality of the water appeared to be scarcely affected; yet to culture media this mixture yielded a superabundance of objectionable microbes.

In considering such startling facts as these it is well not to lose sight of the truth that the nature of the organic matter present in a water has much to do with its power of supporting germ-life. In striking illustration of this I record the following experiments:—I made dilute solutions of the substances named with sterilised water, and then thoroughly re-sterilised the mixtures, and, after cooling, infected each with an equal but minute amount of typhoid bacilli. After two days' continuance in the dark at 60° F. the proper dilutions were made and the usual bacteria-flask cultures were initiated.

	Microbes in 1 cc.	Ratios.
(1) 0·05 per cent. domestic sewage..	55,080	58·9
(2) 1·00 " " " ..	244,800	257·0
(3) 0·01 " peptone .....	2,550	2·7
(4) 0·05 " " .....	117,000	123·0
(5) 0·01 " cane sugar .....	950	1·0
(1) 1 grm. of the dry sewage organic matter gave .....	110,160,000,000	116,000
(4) 1 grm. dry peptone gave .....	2,340,000	26
(5) 1 grm. dry sugar gave .....	95,000	1

These results illustrate the necessity of a chemical as well as a bacteriological analysis. Experiments are in progress with reference to the "germ-development power" of waters, as this should prove a useful factor.

It is in this connection that one may find some value in the total microbial richness of samples, as a large number of microbes may indicate either some source of aerial pollution or the presence of decomposing organic matters capable of supporting the rapid development of pathogenic germs. Dr. G. Roux (Précis d'Analyse Microbiologique des Eaux, 1892, 262) not only emphasises, this fact, but affirms that the quantity of micro-organisms in a water is of the greatest importance in enabling us to judge of its quality. A large number of chemists adduce apparently strong reasons for ignoring these data of the water-bacteriologist; it is urged that a very large proportion of microbes are quite harmless, and though they might not care to repeat Dr. Emmerich's experiment of drinking sewage with, as he has stated, some slight benefit to himself! yet they consider mere numbers of germs not necessarily an



evil sign. Macé (Traité Pratique de Bacteriologie, 1891, 712) gives the following table to express the qualities of waters in terms of their microbial contents:—

Bacteria per 1 cc.	
Very good waters contain . . .	0— 50
Good waters contain . . . . .	50— 500
Mediocre waters contain . . . .	500— 3,000
Bad waters contain . . . . .	3,000— 10,000
Very bad waters contain . . . .	10,000—100,000 and above.

A sceptical attitude towards these figures is for the present advisable. Dr. Migula (Centralblatt für Bakteriologie, &c., 1890, No. 12), of Carlsruhe, shows the necessity of counting not merely the numbers but the species present in a water, as he has frequently found that waters with a comparatively small number of germs often contain more pernicious species than others with larger totals, thus proving the importance of the species-determination.

The only attempt at a recognition of this principle is seen in the statements of the "liquefying" and "non-liquefying" colonies which a water is capable of yielding to nutrient gelatine. I have abandoned this distinction as valueless. The assumption is that the liquefying microbes are more harmful than the non-liquefying; now in waters one finds some 22 species (Dr. Frankland's "Micro-organisms in Water," 1894, 398) of microbes pathogenic either to men or animals, and of these I find that 14 are liquefying and 8 are non-liquefying, while the germs pathogenic to man are divided equally between the two classes; but as cholera and anthrax germs can only occur very rarely in our water supplies, while the typhoid bacillus and its congener bacillus coli communis are frequently to be sought, and as both these are non-liquefying organisms, we may fairly rule the distinction out of court as a guide to the potability of a water. Roux (*lib. cit.* 192) shows that the ideas of Proust, who gave forth this distinction, are quite at variance with his own observations; inasmuch as the waters of the Seine, though much more impure than those of the Rhone, contain infinitely less liquefying microbes. In confirmation of Roux's assertion, Lacassagne notes that corpses decompose much more quickly in the Rhone than in the Seine; attesting to the larger proportion of liquefying organisms.

I find that a far better method for making a general distinction between harmless and more or less objectionable microbes is to submit a portion of the water to a temperature of blood heat (98° F.) for 12 hours, and then determine the proportion of micro-organisms existing. I was led to try this process after a consideration of the fact that all pathogenic microbes not only resist, but under suitable conditions largely multiply at the body temperature; while the majority of harmless microbes, such as the commonly occurring *Bacillus albus* and *Bacillus aquatilis*, speedily perish. As examples of the utility of this method I adduce a few results:—

	Pure Upland Surface Waters.		ATyphoidised Water.	A Polluted Well Water.
	A.	B.	C.	D.
Before exposure to 98° F.	112	28	85,500	12,204
After exposure to 98° F.	2	0	51,500	8,000
Microbes surviving 98° F., per cent.	1.78	0	60	66

As further evidence of the dangerous character of C and D, I may note that both contained a considerable number of facultative anaerobes. If the heating is continued for longer than 12 hours the surviving microbes begin to multiply enormously, particularly in waters rich in organic matters; after continuing the heating of D another 12 hours each cc. yielded 110,000 colonies to nutrient gelatine.

A sharp process, of value at all events as affording negative evidence of bacteriological impurity, is to take 1 cc. of the water in a sterilised pipette, giving about 40 drops for the 1 cc., and add a drop to each of 40 sterilised tubes of bouillon; exposing the tubes to 98° F. for 12 hours. One part of sewage in 1,000 parts of sterilised water will give an abundant growth in every tube, while really good waters will show at the most a very few developments; indeed, several waters thus examined by me have not given a single growth, thus affording a speedy indication of bacteriological purity. In every case it is advisable to count the total colonies a water is capable of yielding; to this end the majority of bacteriologists use plate cultivation. My experience of such cultivations is anything but satisfactory; although I have set apart a special laboratory for this work and have taken considerable precautions against the invasions of dust, reducing the average number of germs in the air to about two per litre, yet blank gelatine plates almost always show some colonies. Should penicillium spores find their way on to the plates, the spores will rapidly extend and contaminate all the cultures, as I have repeatedly found. Roux (*lib. cit.* 153) notes the same objection, and affirms that the plates are sufficiently long exposed to the air before and after the solidification of the gelatine to receive contamination with aerial germs. With Roux I agree that it is somewhat "eventual" for an observer to pretend that he can discover by the superficial character of the colonies the number of germs which have been aerially deposited.

I place my faith upon bacteria flask cultivations. I have had specially made a considerable number of large, flat-bottomed shallow flasks, and find that these can be readily charged with nutrient gelatine, and can be as easily sterilised, while blank experiments show that in introducing 1 cc. of a water there is practically no risk of aerial pollution. Although one cannot as readily apply the microscope to these flasks as to the plates, yet one can apply a very fair magnification and can also obtain prick-cultures from the colonies. A Wollfhuigel's counting-plate and a 1-inch objective enable one to make a very satisfactory enumeration of the colonies. The plate contains 144 sq. cm. divisions, and some of these are sub-divided into nine; if one of these smaller divisions contains ten colonies, then—

$$(\frac{1}{2} \times 3.14) \times 9 \times 10 = x, \text{ number of colonies.}$$

Dr. Griffiths (Chemical News, May 1893, 234) recommends a species of Hesse's tube, the sides of which are coated with nutrient gelatine; by means of a spray 1 cc. of the water can be distributed over the surface of the medium. I have tried this method, and discard it because the liquefying colonies speedily spread themselves over the sides long before the colonies are entirely developed; thus the counting becomes difficult and even impossible. Pétri's dishes are useful for certain kinds of work, but are not as convenient for general purposes as the flasks.

The full bacteriological analysis of a water requires much labour, and it is not within the scope of this paper to describe the methods by which it is accomplished; sufficient it must be to indicate the general principles of procedure. By making a number of dilutions of the sample with sterilised water and by inoculating with these a large series of nutrient gelatine and bouillon tubes one is able to arrive at that stage when some tubes are unaffected by germs, while others contain pure cultures. By utilising various media for the further study of the pure cultures so obtained, and by noting their microscopical features and measurements and reaction to dyes, they may be finally classified.

The table on page 1159 expresses some of the results thus obtained, and may be of interest as showing the relation of chemical composition to bacterian contents.

F is indeed a rare example, as the 1 cc. taken did not contain a single microbe! It is worthy of note that this water was quite free from nitrites, nitrates, and albuminoid ammonia, and that it was drawn from an artesian spring.

B was drawn from under the peat near the sea, and although containing a large excess of saline and organic matter, and doubtless possessed of enormous germ-development power, was none the less free from objectionable

		A.	B.	C.	D.	E.	F.
Total solid matter .....	Grains per gallon	6.5	222.0	7.5	6.8	7.6	34.2
Organic and volatile matter ...	"	0.7	51.0	1.5	1.0	1.5	1.5
Saline matter.....	"	4.8	171.0	6.0	5.8	6.1	29.7
Chlorine .....	"	0.50	74.5	0.50	0.65	0.50	2.80
Nitrites .....	"	None	None	None	None	None	None
Nitrogen as nitrates .....	"	"	0.70	"	"	"	"
Free ammonia .....	Parts per million	"	4.00	"	"	"	0.08
Albuminoid ammonia.....	"	0.08	2.00	0.04	0.03	0.04	None
Microbes in 1 cc. ....	"	"	"	"	"	"	"
<i>Bacillus albus</i> .....		20.0	220.0	96.0	100.0	22.0	None
" <i>terno</i> .....		10.0	"	"	"	12.0	
" <i>fluorescens liquefaciens</i> .....		12.0	"	"	"	"	
" <i>ubiquitus</i> .....		"	"	"	"	1600.0	
" <i>aërophilus</i> .....		"	"	104.0	"	"	
<i>Micrococcus aquatilis</i> .....		"	2000.0	100.0	"	"	
" <i>cereus albus</i> .....		"	1980.0	"	300.0	"	None
<i>Bacillus typhi abdominalis</i> .....		"	"	"	"	"	
" <i>coli communis</i> .....		"	"	"	"	"	
" <i>putrificus coli</i> .....		"	"	"	"	980.0	
" <i>fluorescens putidus</i> .....		"	"	"	"	"	
" <i>viridis pallescens</i> .....		"	"	"	"	"	None
<i>Penicillium</i> spores .....		1.0	"	280.0	"	"	
Totals .....		43.0	6180.0	580.0	400.0	2011.0	
Number of species .....		1.0	3.0	4.0	2.0	9.0	0

microbes; this is surely further proof of the absolute necessity of a chemical as well as a bacteriological analysis. E was a water contaminated by typhoid dejecta. A and C were pure upland surface waters.

Public analysts are occasionally called upon to analyse waters which have been suspected as the cause of an outbreak of enteric (typhoid) fever, and there can be but little doubt that in a large number of cases, for reasons already indicated, the chemical analysis must not merely often fail to show that the water is dangerous, but it will be always valueless in proving the absence or presence of the specific bacillus, the discovery of which can alone be sufficient to condemn the sample on the main charge. Even to professional bacteriologists the detection of typhoid germs in water is admittedly a very difficult task. Considerable progress has been made recently in this direction, and we may yet hope to discover more certain methods for the discovery of this microbe.

Before reporting the results of a number of experiments made with this object in view, it will be necessary to note certain facts, the apprehension of which lies at the very door of the inquiry. Dr. Arthur Gamgee (Text-book of the Physiology and Chemistry of the Animal Body, Part II., 437), in his recent classical work, gives a table of the chief micro-organisms which normally inhabit the human intestine, and to these we may add several more as regularly inhabiting the intestinal tract and urinary passages; in all some 16 species of microbes, some of which are always to be found in human sewage. The principal microbe which is sure to be found in any water contaminated with typhoid is not that of the typhoid itself, but the *bête noir* of the water-bacteriologist—*Bacillus coli communis*. The latter bacillus far exceeds in number that of the specific *B. typhi*, even in typhoid stools; this has been my experience at least. I have found that the secretions or excretions of

*B. coli* com. soon prove fatal to *B. typhi*, and that the latter soon disappears from a mixture containing the two microbes. This is the reason why the bacteriologist often fails to find the specific bacillus in waters certainly contaminated by typhoid; but he is sure to find *B. coli* com.—a microbe perhaps more resistant to conditions of temperature and antiseptics than any other. This germ is not difficult to discover, and from subsequent considerations it will be seen that its discovery in appreciable numbers in any water should be sufficient to condemn it as capable of communicating enteric fever.

Dr. E. Henke (Centralblatt für Bakteriologie, &c., 1894, 481) has recently proved the great ubiquity of *B. coli* com. in nature, and states that it plays a much greater rôle in the outer world than has hitherto been suspected; that after death it speedily penetrates to the spleen, liver, kidneys, and even to the heart's blood; and that it has been found in strumitis and several other affections. J. H. Kellog (Centralblatt für Bakteriologie, &c., 1894, 132) charges this bacillus with having produced a typhus epidemic in the case of a well-water polluted by infiltrations from closets, and shows that by passage through the human body this germ acquires all the virulence of *B. typhi*. It is only in this way that we can account for a number of the typhoid epidemics quite untraceable to any infection by the specific bacillus itself. The Lyons School of Bacteriologists and Macé (Traité Pratique de Bactériologie, 1891, 435) have long maintained the typhogenicity of *B. coli communis*. Quite recently, Chantemesse (La Semaine Médicale, 1894, 215) has shown that the waters of the Vanne, Avre, and Dhuis caused the spring epidemic of typhoid in Paris, although only virulent *coli* bacilli and no typhoid bacilli could be found in these waters. These facts are sufficient to show that the presence of *B. coli communis* in any water must be regarded with suspicion as certainly indicative of

pollution with faecal matters. In the next paper, I hope to show the best methods for the detection and enumeration of these microbes, with several analyses illustrating the relations which exist between the chemical composition and the bacteriological contents and biological capabilities of waters.

#### DISCUSSION.

Mr. T. A. GLENDINNING pointed out the importance of the qualitative examination for the bacteria in water, and also the difficulty of distinguishing between pathogenic and very similar non-pathogenic species—as, for example, in the case of *B. coli communis* and *B. typhus*,—only two distinguishing features being known. Even inoculation experiments on animals did not give satisfactory evidence that the organisms were harmful to man, because the same organism affected different animals differently.

Mr. H. R. PROCTER said that the difficulty of carrying out inoculation experiments in this country added materially to the difficulties of the subject.

The CHAIRMAN thought that it was doubtful whether it was possible or even desirable that analysts should, in the ordinary routine of work, take up the bacteriological side of water examination. This could only be really satisfactorily done by State or municipal laboratories with a specially trained staff.

Mr. F. W. RICHARDSON, in reply, said that he had already indicated some very useful qualitative modes of testing the bacteriological quality of a water; for instance, the determination of the total number of bacteria which could be grown from a sample of water before heating it to 98° F. for some hours and after such treatment, gave useful indications as to the nature of the microbes present; the non pathogenic organisms were very largely killed by this heating. In reply to Mr. Glendinning, there were some eight methods of distinguishing *B. coli communis* from *B. typhus*, and he also thought that the department of bacteriology relative to water-analysis could be satisfactorily cultivated by chemists; indeed, it was impossible for an analyst to give a definite dictum as to the real quality of certain waters unless he could say something as to their microbial contents.

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Meeting held on Monday, December 3rd, 1894, at the  
Yorkshire College, Leeds.

PROFESSOR A. SMITHells IN THE CHAIR.

#### ON THE ACTION OF CERTAIN ANTISEPTIC SUBSTANCES ON YEAST.

By HAROLD H. MANN, B.Sc., A.I.C.

SINCE the time at which Pasteur, by his brilliant researches on the micro-organisms of fermentation, first opened out the world of bacteria to science, the study of antiseptic action, and of antiseptic power has been approached in a variety of ways. Yet, although considered by a great number of observers as a hygienic, physiological, medical, or commercial question, the investigation of the chemical role of any antiseptic substance seems to have been almost entirely neglected. Even to this day researches on very similar if not identical questions often lead to entirely opposing results, and this cannot be in all cases due to errors of work, but must be regarded as a consequence of our almost complete lack of knowledge as to the rationale of the action of the antiseptic in any particular case.

Acting therefore on the advice of Professor Duclaux, of the University of Paris, I have during the past year turned my attention to the study of one problem involved in this hitherto almost unploughed part of the field mentioned above. Taking yeast as a typical micro-organism, I have attempted to consider the question of the relation between the antiseptic power of a substance, and the quantity of yeast on which it was made to act.

By most observers this influence has been entirely left out of consideration, and the strength of the antiseptic solution necessary to destroy a micro-organism has been regarded as independent of the quantity of organism there present. That this position is not in all cases true has, however, already been shown in several cases. Take chloroform as an example. Here Duclaux showed, in opposition to Liebig, that while this body (when an aqueous solution is completely saturated with it) stopped the commencement of fermentation, yet it was powerless to produce the same effect when fermentation had actively commenced, *i.e.*, when the number of cells had sufficiently increased to succeed in continuing their action in spite of the substance added. With borax a similar effect can easily be produced, and, in this case, if the amount of yeast used be sufficiently increased, the dose of borax can be augmented with impunity without hindering the completion of the fermentation.

The work which I am about to detail is, however, concerned with the action of certain metallic salts, and with that of phenol on yeast cells. These latter were sometimes used in the form of a pure culture of *saccharomyces cerevisiae*, and sometimes, when the quantity became too large to be conveniently prepared in the laboratory in that of ordinary commercial beer yeast.

The antiseptic power of a substance has been usually measured, in the past, by two methods. The one of these consists in measuring the exact amount of antiseptic which, added to a nutrient liquid containing the yeast cells, would absolutely prevent their growth, and the other in finding the strength of antiseptic solution necessary to destroy the reproductive power of the organism in question, even when removed to the most favourable conditions. However good these methods may be in theory, I found them very long and tedious to carry out in practice. The process was therefore modified so that only enough antiseptic was added to partially prevent the growth, or destroy the reproductive power of part only of the cells used, as the case might be.

In order to carry this out by the method first mentioned the following process was adopted:—Several samples of nutrient liquid, containing an equal amount of the antiseptic to be used, were prepared, sufficient to partially prevent the growth of yeast. These were sown with different determined numbers of yeast cells, and left to develop under the same conditions for an equal length of time. After this development had taken place, the numbers of cells present in a sample taken from each liquid were counted. At first, this counting was done under the microscope, by means of one of the pieces of apparatus in use for counting the corpuscles of the blood. It was soon found, however, that the amount of yeast necessarily present, in order that an accurate numeration of the cells should be possible by this method, was too large to allow for the free development of each cell in the liquid, although it was very far from being the maximum which could exist there.

Microscopic counting was therefore abandoned, and cultivation of the cells contained in a sample of the liquid on nutrient gelatin was substituted for it. This gelatin was composed of 100 grms. cane-sugar, dissolved in water, inverted with a little acid, and neutralised; 40 grms. barley sprits, boiled with 1 litre water and filtered; 200 grms. gelatin; the whole made up to 2 litres, filtered, and sterilised. By this means an extremely small number of cells could be counted, small enough to allow their free development while also permitting their accurate numeration. This is shown in the following experiments, where, as would be expected, the number of cells found after development is approximately proportional to the quantity originally introduced. In the column headed B the numbers of cells are given which would be found were the method absolutely exact (taking, of

course, one of the numbers actually found as basis), while the column A represents the relative numbers added:—

	Number of Cells Added.	A.	Number of Cells Found.	B.	Time of Action.
I.	(1) Not determined, but	1	14.0	12.0	Hours. 3½
	(2) proportionally, as in A.	2	24.0	24.0	3½
	(3)	4	48.0	48.0	3½
II.	(1) Not determined, but	1	19.0	19.0	6
	(2) proportionally, as in A.	2	44.0	38.0	6
	(3)	3	56.0	57.0	6
III.	(1) 13	1	23.5	21.5	17½
	(2) 26	2	46.5	43.0	17½
	(3) 104	8	171.5	172.0	17½

As previously stated, however, we can also investigate antiseptic action by determining the amount of antiseptic required to destroy the subsequent developing power of the organism. Looking at the question in this light I made some experiments in the following manner:—A small and a large number of cells were put into equal volumes of an antiseptic solution, *not sufficiently strong to kill all*, and after different lengths of time, say for instance, 10 minutes, 20 minutes, and half an hour, samples were taken. The number of cells in these which were still living, was determined (after washing the sample from antiseptic in sterile water) by culture on gelatin. By comparison of the numbers thus obtained it can easily be seen whether the antiseptic power of the substance in question is variable with the number of cells on which it has to act.

For the first series of experiments phenol was used as the antiseptic substance. Employing the method first named, the following table shows that upon the power of this antiseptic the quantity of yeast present exercises little, if any, influence.

	Number of Cells Added.	A.	Cells Found.	B.	Strength of Phenol.	Time of Action.
I.	(1) ..	1	32.0	32.0	0.013	Hours. 2
	(2) ..	2	62.0	64.0	0.013	2
	(3) ..	4	128.0	128.0	0.013	2
II.	(1) 2,315	1	15.0	17.0	0.053	3½
	(2) 4,630	2	36.0	35.0	0.053	3½
	(3) 9,260	4	69.0	60.0	0.053	3½
III.	(1) 92	1	22.0	19.5	0.05	16½
	(2) 384	2	39.0	39.0	0.05	16½
	(3) 532	1	18.5	23.5	0.04	17½
IV.	(1) 1,064	2	43.5	47.0	0.04	17½
	(2) 2,128	4	93.5	94.0	0.04	17½

Here A and B retain the meanings they possessed in the previous table.

These results are confirmed by the following experiments, carried out according to the second method. Here  $x$  is the number of cells used in the one case, while  $y$  represents the number (taken from the same culture) employed in the other. The ratio  $\frac{y}{x}$  will, therefore, show any variations which may occur in their relative values.

	Time of Action.	$x$	$y$	Ratio $\frac{y}{x}$
Minutes.				
I.	(1) 5	97	220	2.4
	(2) 10	86	220	2.56
	(3) 15	74	182	2.46
II.	(1) 6	208	450	2.16
	(2) 11	198	420	2.15

Here the phenol was employed of a strength corresponding to 0.55 per cent.  $C_6H_5OH$ .

Such being the results obtained with phenol, the same methods were applied to a representative of the salts of the heavy metals possessing antiseptic properties, namely, copper sulphate. The following table represents the numbers obtained by the first method above given. Here, however, B represents the relationship between the number of cells found after the development in each series. The result of a blank experiment is given in each case where the conditions were exactly the same as in that opposite to which it is placed, save that no antiseptic was introduced into the nutrient liquid.

	Number of Cells Added.	A.	Number of Cells Found.
I.	(1).....	1	87.0 (Blank Expt. = 45)
	(2).....	2	167.0
	(3).....	4	417.0
II.	(1).....	1	178.5 (Blank = 234)
	(2).....	2	438.5
	(3).....	4	801.5
III.	(1).....	1	260.0 (Blank = 405)
	(2).....	2	569.0
	(3).....	4	1,160.5
IV.	(1).....	1	355.0 (Blank = 583)
	(2).....	2	761.0
	(3).....	4	1,344.0
V.	(1).....	1	237.5 (Blank = 330)
	(2).....	2	565.5
	(3).....	4	1,265.5

	B.	Strength of $CuSO_4$ .	Time of Action.
Per Cent.			
I.	(1).....	0.01	Hours. 18½
	(2).....	0.01	18.
	(3).....	0.01	18.
II.	(1).....	0.005	18.
	(2).....	0.005	18.
	(3).....	0.005	18.
III.	(1).....	0.016	18.
	(2).....	0.016	18.
	(3).....	0.016	18½
IV.	(1).....	0.016	21½
	(2).....	0.016	21½
	(3).....	0.016	21½
V.	(1).....	0.013	21.
	(2).....	0.013	21.
	(3).....	0.013	21.

From the repeated excess of the number of cells found with the higher proportions of yeast over those which would be expected, calculating on the basis of those introduced, it may safely be concluded that the antiseptic effect of a copper sulphate solution becomes less on augmenting the amount of yeast on which it is made to act. This effect is also seen in the following experiment conducted according to the second method.

Time of Action.		a.		b.		Ratio $\frac{a}{b}$	
Minutes.							
I.	10 and 10½	425.0		935.5		2.2	
	20½ and 20	381.5		886.5		2.32	
	30½ and 31½	204.0		487.5		2.41	

(Strength of  $\text{CuSO}_4$  solution = approximately 0.2 per cent.)

It is then sufficiently evident that the power of copper sulphate solution is weakened when brought into contact with a large amount of yeast, and the question now arises. How is this weakened effect produced?—The simplest hypothesis is undoubtedly that which supposes a fixation of the metal by the yeast.

To test this hypothesis, a quantity of commercial yeast was treated with copper sulphate solution, filtered, and washed until the washings were free from copper. On calcining the yeast which remained, and testing the residue for copper, a distinct reaction was repeatedly and always obtained. We may then conclude that a fixation of copper does occur. This has already been pointed out by Will. (*Zeit. für das gesamte Brauwesen*, 1893) as the result he obtained on treating yeast with copper sulphate solution, but he did not further investigate the cause of the fixation. This now remains to be done, as well as to determine the conditions under which it takes place. Does it vary in amount with the time of action? with the dilution of the solution of antiseptic? or with the condition of the yeast? Further it is wholly due to the soluble contents and products of the organism, or is it partially fixed by the cell-wall?

In order to resolve these questions the solution of copper sulphate was allowed to act directly on the yeast. On filtering, a measured portion (50 cc.) was taken for analysis, and the copper therein determined. Knowing the quantity originally present in the same volume of the liquid, the amount fixed by the yeast was readily obtained.

On heating the yeast to 100° C. with water, not only is the organism killed, but coagulation of the albumen and forcing out of the contents of the cell ensue. The first experiments were relative to the amount fixed comparatively under these, and under the ordinary conditions, after different periods of time. The copper was determined by precipitation with zinc, and in these particular experiments the amount of yeast corresponding to each number given, contained about 1 gram. of dry matter.

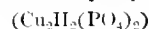
Time of Action.		A.		B.	
		Amount Fixed (Cu).		Amount Fixed (Cu).	
Hours.	Minutes.	188°0	...	183°0	...
1	179°0	9°0		..	
1	175°0	10°0		17°0	
4	177°5	10°5		17°5	

(The quantities of copper given under A and B represent the amounts corresponding to 1 gram. of, in A ordinary yeast, and in B, yeast which has been previously heated to 100° C.)

In answer to the question as to whether all is fixed by the soluble products of the yeast, experiment answers in the negative. On washing the yeast with boiling water until the solution obtained gave no longer the reaction for

phosphates (these forming probably the substances most adherent to the cell itself), treating the residue with copper sulphate solution, and again washing until free from this metal, copper was still repeatedly found to have been fixed in appreciable quantity. Evidently then the insoluble material of the cells take some part in the fixation of copper during these experiments.

On the other hand, the soluble products are responsible for by far the greater portion of the effect, and it was interesting to know in what state the copper was precipitated by them. A portion of the precipitate produced by copper sulphate in yeast water was therefore washed until free from soluble copper. On treating the residue with dilute nitric acid a part dissolved containing the whole of the copper, leaving a gelatinous mass on the filter paper free from this metal. This gelatinous mass had evidently been carried down with the copper containing part of the precipitate. On determining the copper and the phosphoric acid in two equal portions of the filtrate, they were found to be in approximately the proportions necessary to form the second phosphate of copper—



thus—

$$\text{Ratio } \frac{\text{P}_2\text{O}_5}{\text{Cu}} \begin{cases} \text{Found} = 0.8 \\ \text{Theoretical for } \text{Cu}_2\text{H}_2(\text{PO}_4)_2 = 0.89 \end{cases}$$

It very probably consists essentially of this substance.

The question now arose: Does this phosphate of copper act as an antiseptic when introduced into nutrient fluid? On sowing a trace of *saccharomyces cerevisiae* under these circumstances, it was found that no culture supervened. This may, however, be due, not to the action of the above insoluble phosphate of copper, but to that phosphate possibly produced by its partial solubilisation by the acid phosphate of potassium undoubtedly present in the nutrient liquid. The filtered liquid was, in fact, found after the experiment to contain a trace of copper.

Another series of quantitative experiments was now undertaken in which, instead of separating yeast and solution by filtration a centrifugal machine was employed, thus avoiding several objections which might possibly have been raised against those done under the previous conditions. In addition the copper was here determined by the potassium iodide and thiosulphate method. Here are the results of the experiments. All those in each series contained the same amount of yeast, but in B a stronger solution of copper was used than in A, while C, usually similar to either A or B, as far as quantity was concerned, acted upon yeast which had previously been heated to 100° C. Each number given corresponds to a volume of solution of 50 cc.

	Time of Action.	Amount Fixed (Cu).			Total Amount Cu originally in A, B, and C.	
		A.	B.	C.		
	Hours.	Mgms.	Mgms.	Mgms.	Gm.	Gm.
I.	(1)	$\frac{1}{2}$	1.7	2.0	1.2	A = 0.0212; B 0.053
	(2)	$7\frac{1}{4}$	2.7	3.0	1.2	C = 0.0206
II.	(1)	$\frac{1}{2}$	3.3	4.0	..	A and C = 0.0176
	(2)	$7\frac{1}{4}$	..	6.0	3.5	B = 0.088

As a result of these experiments with copper sulphate, I think it may be taken as proved, that a fixation of the metal by yeast does take place, that it is slightly increased in amount as the time of contact becomes greater, that the amount thus fixed is practically the same after heating to 100° C., as previous to the operation, and that the influence in this case of the concentration of the solution is only very slight. Further, that while the greater portion is fixed by the soluble contents and products of the yeast, some at least is taken up by the cell substance itself.

Having obtained such results with copper it became interesting to know whether other metallic salts behaved in a similar manner. Ferric sulphate, lead acetate, and

mercuric chloride were therefore successively used. Though in all cases a fixation was shown to take place, yet it differs either in quantity or conditions in each case.

Perhaps the most nearly allied to that of copper is the action of mercury. In this case the fixation is however very much greater in amount. The figures given below show the amount fixed under various conditions. A, B, and C have the same meanings as in the above given results, and separation of yeast and solution was again effected by means of the centrifugal machine. The mercury was determined by the method of Haunay (by titration with potassium cyanide), either in its original form or in the modification by Chapman Jones (*Journ. Chem. Soc.* 1892).

	Time of Action.	Amount Fixed (Hg).			Total Mercury originally in A, B, and C (50 cc.).
		A.	B.	C.	
I. { (1)	Hours. 1	Megms. 27.0	Megms. 44.0	Megms. 28.5	A and C = 0.135
(2)	24	31.5	..	37.0	B = 0.405
II. (1)	1	52.5	..	55.5	A and C = 0.1515
III. { (1)	1	12.5	..	..	A = 0.106
(2)	5	128.5	278.5	..	B = 0.588

It will be seen that the amount fixed is extremely high. Even in the last case where no less than 0.25 gram. are shown as fixed, the quantity of yeast corresponding to each sample only contained about 0.6 gram. of dry matter.

When we came to an iron salt, another important difference presents itself. Up to this point the effect produced by previously heating the yeast to 100° C. has not been very great. It rises to large proportions in the case now before us, namely, that of ferric sulphate, and this is doubtless due to the insolubility of the acid phosphate ( $\text{FeH}_2(\text{PO}_4)_2$ ) which will be formed at the expense of the acid potassium phosphate of the cell contents (this being probably the only phosphate of potassium occurring inside the cells).

The following are the results of experiments on this question:—

	Time of Action.	Amount Fixed (Fe).			Total Iron originally in A, B, and C.
		A.	B.	C.	
I. { (1)	Hours. 2	Megms. 6.9	..	26.0	A and C = 0.029
(2)	22-24	8.2	..	26.0	.. ..
II. { (1)	21	6.2	..	21.9	A and C = 0.215
(2)	9	8.9	..	22.5	.. ..

In these cases the separation was again effected by filtration, but by means of a reversed filter, thereby doing away with a great many objections to the process of filtration applied for this purpose in its ordinary form.

Nevertheless, centrifugal force was again adopted in the next series, that bearing on the effect of concentration of the solution on the amount of metal fixed. Here it was once again made evident what an important part this may play in the question we are considering.

	Time of Action.	Amount Fixed (Fe).			Total Iron originally in A, B, and C.
		A.	B.	C.	
I. (1)	Hours. 22-24	Megms. 9.2	Megms. 19.3	..	Grm. A = 0.0132; B 0.066
II. { (1)	1	9.6	21.5	..	A = 0.0109
(2)	18	9.9	27.1	..	B = 0.0433

We now arrive at the list of the metals which have been used, namely lead. This evidently acts as a precipitant of many of the organic matters in yeast. The results, especially as concerns the series I., II., and III. are only approximate. The lead was in all cases estimated as chromate, according to the process recommended by Fresenius, and the salt used for these experiments was in all cases the acetate.

The numbers are as follows, where, as in previous cases, the yeast was separated by the centrifugal machine.

	Time of Action.	Amount Fixed (Pb).			Total Lead originally in A, B, and C.
		A.	B.	C.	
I. { (1)	Hours. 1	Megms. 75.0	Megms. ..	Megms. ..	Grm. A = 0.128
(2)	2 1/2	..	85.0	..	B = 0.405
II. { (1)	51	15.0	44.0	91.5	A and C = 0.194
(2)	18	..	..	94.5	B = 0.405
III. { (1)	1	41.0	49.5	79.0	A and C = 0.069
(2)	20	..	75.5	..	B = 0.379
IV. { (1)	1	38.8	48.0	..	A = 0.076
(2)	19	..	70.0	82.5	B and C = 0.379

These results taken as a whole confirm one another and place this metal by the side of iron, differing from it, however, in the much larger total quantity fixed by about the same quantity of yeast.

Such, then, are the results obtained with the metallic salts, an examination of whose action has formed the principal portion of this paper. The conclusions to be drawn from a consideration of the results may be briefly stated as follows:—

(1.) That in the case of certain metallic salts possessing antiseptic properties, the amount of antiseptic required in order to kill the yeast cells, varies with the quantity of these last, in the sense that it becomes greater as the amount of the latter augments. With phenol this effect has not been shown with certainty to exist.

(2.) That with the aforementioned metallic salts, namely those of copper, lead, iron, and mercury, this effect is due to a fixation of the metal by the yeast. This fixation varies in amount with the different metals, and may vary with the time of action, with the dilution of the solution, and with the condition of the treated yeast. This seems, indirectly, to give grounds for objection to their use as antiseptics in practice, owing to the rapid exhaustion, say, of a solution of mercuric chloride at 1 in 1,000, which must ensue.

(3.) That this fixation takes place, partly at any rate, by precipitation of the metal as phosphate. At the same time, however, it is fixed in a more intimate sense in the cell-wall. It may cause, in addition, the precipitation of some part, at any rate, of the organic cell materials.

In conclusion, I can only express my sincerest thanks to Professor Duclaux, who first suggested the work to me, and who has, during its whole progress, been ready with help, advice, and suggestion. I would also here acknowledge my obligations to his chief assistant, Dr. Fernbach, who has ever been at hand to afford all the valuable aid of which I might stand in need.

#### DISCUSSION.

Mr. F. W. RICHARDSON asked whether the author had noticed any difference in the shape of the cells under the influence of different antiseptics, and whether he had observed that very small quantities of the antiseptic increased, instead of diminishing the rapidity of growth of the organisms. Minute quantities of mercuric chloride and also of boric acid had been observed to exert such an influence on the growth of *saccharomyces cerevisiae*. He thought that for the determination of small quantities of copper, titration with potassium ferrocyanide was to be preferred to precipitation with zinc.

Dr. CAMERON referred to the interest, from a medical point of view, of investigations such as that of Mr. Mann, and more especially of chemical work on the substances produced during the growth of micro-organisms.

Mr. H. R. PROCTER pointed out that the antiseptic power of the salts examined was to some extent comprehensible, as they were absorbed by the organisms and chemically changed, but the action of phenol which apparently underwent no change, and was neither absorbed nor destroyed, was very difficult to understand.

Dr. LEWKOWITSCH asked the author what proofs he was able to give that the fixation of the metallic salts by the yeast was due to the formation of metallic phosphates, and pointed out that physical processes, such as absorption, probably also played a part in the fixation. It is well known, for example, that charcoal fixes considerable quantities of many bodies which cannot be removed by washing.

Dr. EWAN agreed with the last speaker, and thought that the salt must also pass into the cell to some extent by diffusion through the cell wall.

The CHAIRMAN (Professor Smithells) commented on the interesting nature of the action of phenol, which appeared to be independent of the quantity of yeast acted upon, within fairly wide limits.

Mr. MANN, in reply, said that he had not noticed that  $\text{CuSO}_4$  had any influence on the shape of the yeast cells, but that very small quantities of phenol caused the yeast cells to stick together, so that they formed long chains like those of *S. pastorianus*. Very minute quantities of  $\text{CuSO}_4$  had been observed to stimulate the growth of yeast. Phenol appeared to be quite unchanged by its action on yeast, though it is not easy to determine accurately the quantity of phenol in the solutions. He thought that small quantities of copper could be most accurately determined by precipitation with zinc, though the method was tedious. Pure cultures of *S. cerevisiae* were always used in his culture experiments. The insoluble substance obtained from the yeast which had been treated with  $\text{CuSO}_4$  solution contained copper and phosphoric acid in the proportions in which they exist in copper phosphate.

In addition to the copper absorbed as phosphate, however, there was another portion the absorption of which was doubtless due to some physical action, perhaps absorption by the cellulose of the cell wall. He thought that osmosis was not a factor of importance, as this additional fixation took place also with cells which had been disintegrated by boiling.

of oils used was considerably altered. I need not enter into any description of the old system, as I cannot claim to be very familiar with it, but it seemed to lay great stress on the exclusion of mineral oil, under which term in practice all unsaponifiable matter was included.

I need not trouble you with the details of the present system further than to say that whatever the original intention of the framers may have been, in practice, in Yorkshire at least, when a mill is being rated, the chemist, if he is consulted, is asked to determine the flash-point of the oil (minimum  $171^\circ \text{C.}$ ), and the unsaponifiable matter, the risk being considered greater as the percentage rises, the classes being under 30 per cent., above 30 per cent., and below 50 per cent., and above 50 per cent. An undertaking is asked for from the oil merchant or manufacturer supplying the oils, which unless the very highest possible rate is to be charged, disclaims the presence in the oils in question of any linseed, rape, cotton seed oil, &c., &c., not forgetting mineral oil,\* which term dies hard in insurance schedules. As the oil manufacturer, especially if he recovers oil from the cloth manufacturers' waste soap liquor, cannot be supposed to know what oils were originally used, and as in most cases detection of the oils named is almost impossible in such complicated mixtures as black or brown cloth oils, he makes no scruples in signing this undertaking. Indeed, I understand that the unanimity displayed by the oil trade in disclaiming the use of all and sundry oils objected to, and the practical impotence of the insurance companies in the face of this, has led recently to the abandonment of this written undertaking scheme altogether, and the insurance companies rather rely on the price paid for the oil, perhaps rather a more rational proceeding, but still on an essentially wrong principle, as it presumes that an oil that is good from a cloth manufacturers' point of view—scouring properties, &c.—is safe from the insurance standpoint.

Now, instead of getting a supposed character with the oil from the oil manufacturer which is of little value, and a determination of the unsaponifiable which means nothing from the insurance point of view unless perhaps, that an oil, high in unsaponifiable constituents, is in all probability a very safe oil as far as liability to spontaneous combustion is concerned, I have always considered that retaining the flash-point as at present, oils should be tested by some simple and easily applied method to ascertain their liability to spontaneous combustion as compared with some standard oil or mixture of oils (probably a standard mixture of mineral and olive oil would be best) when spread on cotton waste.

I am sorry that I am unable to bring any such apparatus to your notice to night as the result of my work.† But in the course of my experiments I obtained some results which I think open a question that should be dealt with before any fresh regulations are issued.

I was working with cotton seed oil, and in comparing the behaviour of neutral cotton seed oil with that of free fatty acids derived from it, when heated to  $110^\circ$ – $150^\circ \text{C.}$  spread on cotton wool, I found that the cotton wool treated with the free fatty acid, invariably browned much more rapidly than the cotton wool containing the neutral oil.

Examining this further, both in the case of cotton seed and olive oils, I obtained the following results.

I should say that I selected cotton wool for my experiments as being a form in which pure cotton could be readily obtained, and as giving quicker results than cotton waste or cloth.

The experiments were carried on in an ordinary copper water jacketed oven, the water being at boiling point, the cotton wool, about 15 grms., containing 50–60 grms. of oil or fatty acid, being placed in a porcelain basin, a thermometer stuck in the heap, the stem of which protruding through the top opening of the bath allowed the temperature to be read off.

\* No account is taken of the difficulty or rather the impossibility of detecting mineral oil proper in presence of the hydrocarbons formed when an oil is distilled.

† There is an apparatus, "Ordways," in use in America, but it has not been adopted in this country.

## SPONTANEOUS COMBUSTION OF OILS SPREAD ON COTTON.

BY WILLIAM McD. MACKEY.

### PART I.

#### EFFECT OF FREE FATTY ACID.

I HAD hoped to give a paper on this subject later on in the session, but I found that it would be impossible for me to carry the experiments any further in the time at my disposal, and as the experiments done seem to have an important bearing on fire risks in cloth mills where cotton or mixtures containing cotton are used, I think it better at this juncture to give my results in the form of a preliminary note.

About two years ago the insurance companies, or those of them that are known as the tariff offices—being all bound to assess risks according to one code, including nearly all the British offices—is-ued new regulations bearing on the rating of mills, and in this the rating according to the kind



Parallel experiments with the oil and fatty acids derived from the same stock of oil were done in each case.

It should be mentioned that when the fatty acids were used wet, immediately after washing, the neutral oil was also washed before the experiment.

The following short tables give my results.

TABLE I.  
COTTON-SEED OIL ON COTTON WOOL.

Neutral Oil.	Free Fatty Acids.
Time 1½ hours; temp. below 100° C.	Time 1½ hours; temp. 151° C., maximum attained.
" 8½ " " 121° C., maximum attained.	" "

In this and the following experiments the heap attained a maximum temperature and then gradually declined; thus, in the fatty acids experiment the temperature rose to 151° C. in 4½ hours, and in 7 hours had fallen to 130° C. The neutral oil attaining its maximum, 121° C., in 8½ hours, and in 13 hours falling to 113° C.

In neither case did the cotton wool actually fire, though in that of the fatty acids experiment it was very strongly charred.

I cannot say that the oil, though called neutral oil, was really so, as I neglected to test it for acidity; in all probability it contained 1 or 2 per cent. of free fatty acid.

TABLE II.  
OLIVE OIL ON COTTON WOOL.

Neutral Oil.	Free Fatty Acids.
Time 2½ hours; temp. 96·5° C.	Time 2½ hours; temp. 165° C., max.
" 8½ " " 173·0° C., max.	" "

In this case I afterwards found that the oil called here neutral, really contained 8·7 per cent. of free fatty acids, stated as oleic acid.

TABLE III.  
OLIVE OIL (another Sample) ON COTTON WOOL.

Neutral Oil.	Oil containing 50 per Cent. Free Fatty Acids.	100 per Cent. Free Fatty Acids.
Time 3 hours; temp. 100° C.	169° C.	192° C., max.
Time 3½ hours; temp. 103° C.	187° C., max.	" "
Time 6½ hours; temp. 202° C., max.	" "	" "

In these three parallel experiments the neutral oil was absolutely neutral, the small amount of free fatty acid originally present (0·9 per cent.) having been dissolved out with alcohol, the oil being afterwards thoroughly washed with water. That containing the 50 per cent. free fatty acid was made up by adding the calculated amount of free fatty acid (derived from the same stock of oil) to some of the original oil.

TABLE IV.

The following results were obtained with olive oil on pure wool:—

Neutral Oil.	Free Fatty Acids.
Time 4½ hours; temp. 109° C., max.	130° C., max.

The neutral oil was from the same stock as that used in the experiments, results of which are stated in Table II.

The question now arises as to what really takes place in the case of cotton seed or olive oil fatty acids, as compared with the corresponding neutral oils.

From the iodine absorption of the fatty acids we should not expect them to take up oxygen more readily than the neutral oil, and it seems to me that the fatty acids have something akin to a charring action on the cotton wool, that chemical act on takes place between the fatty acid and the cotton with evolution of heat, and that the rise of temperature is not entirely due to the absorption of oxygen, as we may suppose it to be in the case of neutral oil.\*

To throw some light on this point the following parallel experiments were undertaken, slag wool being substituted for cotton wool, as a substance totally unacted on by any weak acid at 100° C.

TABLE V.  
COTTON-SEED OIL ON SLAG WOOL.

Neutral Oil.	Free Fatty Acids.
Time 7 hours; temp. 103° C., max.	105° C., max.

Several more experiments would be required to settle this point, but these two experiments undoubtedly go to show that in the case of an inert body, like slag wool, neutral oil and free fatty acids act much alike.

I tried some further experiments with cotton seed oil and cotton wool, giving both the neutral oil and fatty acids a previous treatment with manganese dioxide.

Thus, 50 grms. neutral cotton-seed oil and 50 grms. fatty acid were treated with 5 grms. manganese dioxide, heated at 105° C. for one hour, spread on cotton wool, and the cotton wool, as before, placed in the water-oven.

TABLE VI.

Neutral Oil.	Free Fatty Acids.
Time 1½ hours; temp. 98° C., .....	192° C., max.
" 2½ " " 192° C., max.	" "

Again, in this case the oil and fatty acids (50 grms.) boiled 10 minutes with 5 grms. manganese dioxide.

TABLE VII.

Neutral Oil.	Free Fatty Acids.
Time 2½ hours; temp. 208° C., .....	265° C. max.
" 3 " " 225° C., max.	(Cotton wool completely charred.)

These results are in the same direction as the others, treatment with manganese dioxide accelerating the process; and I think it or some other "drier" might be used in any standard process in which the behaviour of an oil might be inferred from, say, a 2 hours' test; at the same time, the regulations as to condition of experiment would have to be very strictly laid down and adhered to if comparable results were to be expected, as I found that slight differences in the manganese dioxide treatment have a serious influence on the result.

To return to the question whether the heat evolved is due to absorption of oxygen—a drying action—or partly to that and partly to a more vigorous action of the free fatty acids on the cotton. In connection with this Lavauche found (this Journal 1886, 494) that at the ordinary temperature neutral olive oil treated with finely-divided lead and

\* The oil (originally absolutely neutral) used in experiment I., Table III., afterwards squeezed out of the cotton wool at the end of the experiment, was found to contain 10·23 per cent. of free fatty acid as oleic acid. This might point to the oil becoming acid before the heating becomes pronounced.



spread on a watch glass, in 7 days absorbed 1.7 per cent. oxygen, whilst the free fatty acids similarly treated only absorbed 0.7 per cent. in 8 days.

Again, in the case of cotton-seed oil, the neutral oil absorbed 5.9 per cent. in 2 days, the free fatty acids only 0.8 per cent.

It may be that the higher temperature at which I wrought, and this would particularly apply to the free fatty acids from cotton-seed oil, the fluidity of the acids and the state of fine division on the cotton account for my results.

Yet the results given in Tables IV. and V., particularly the latter, in my opinion point to the cotton itself playing a part in the reaction resulting in the evolution of heat.

But before anything definite can be said much work will be required. Had I had time I should have liked to determine the iodine or bromine absorptions of the neutral oils and free fatty acids before and after the experiments, also the increased acidity, if any, of the product of the neutral oil experiments.

But putting the theory of the reaction aside, it appears to me that the results have some importance as bearing on insurance risks, particularly when we consider that under the present system of rating recovered oils, practically free fatty acids are allowed to be used without any extra rate if they contain below 30 per cent. unsaponifiable matter.

It is a matter of common knowledge that the presence of unsaponifiable matter, at least when consisting of hydrocarbons, has a distinct influence in correcting the tendency of such oils as olive- and cotton-seed oil to induce heating and eventually spontaneous combustion when cotton saturated with them is heaped in a heated atmosphere. Hence I question if the insurance companies act wisely in aiming at the introduction of pure free fatty acids.

There can be no doubt that if the unsaponifiable matter consists of mineral oil it should be of very high flash point, but that can be easily ascertained by a simple and well-understood test.

That the manufacturers of certain classes of goods should prefer to work with oils containing large percentages of unsaponifiable matter, may at first appear strange, but it may be depended on that they know their own business best; and I contend that a flash-point test and a test to determine the tendency of an oil to spontaneous combustion under the conditions obtaining in use, are the points of importance from an insurance point of view. And certainly until some standard test for the latter is adopted, when the chemist is consulted, he should be required to make tests on which to base a rational opinion, and not as at present, asked merely to determine the flash point, and further the unsaponifiable matter in order that the oil in question may be classed for rating but not its safety or the reverse demonstrated.

#### DISCUSSION.

Dr. LEWKOWITZ said that little was known about the chemistry of the oxidation of oils in air, though the matter was of importance from the point of view of insurance risks. The comparative ease with which spontaneous inflammation took place on cotton waste was due doubtless largely to the great surface of oil exposed to the air favouring rapid combination. The fatty acids in the oils oxidise much more readily than glycerin, which, indeed, is little acted on. He compared in this respect oxidised oils to "boiled" oil, which contained very nearly as much glycerin as raw linseed oil.

Mr. H. R. PROCTER referred to Mr. Mackey's observation that the oils containing fatty acid when exposed to air on cotton wool developed a much higher temperature than oils which are free from fatty acid, whereas no such difference was observable when slag wool was used. This seemed to show that the cotton wool took part in the action. He asked whether an unsaponifiable mineral oil of equally high flash-point is so dangerous as an oil which can be saponified.

Mr. G. W. SLATTER said that many manufacturers would not buy oils containing more than a certain percentage of fatty acids, though this was more on account of other

properties than because of their greater liability to spontaneous inflammation. He also suggested that the value of Mr. Mackey's experiments would be greater if by aspirating a slow current of air through the oil soaked wool he approximated more to actual conditions.

Mr. PEMBERTON quoted some observations which seem to show that pure vegetable oils were more liable to spontaneous inflammation than oils containing an admixture of mineral oils. He also said that some oils, partially saponified by treatment with steam, were more easily inflammable than the same oils before steaming.

Mr. HOLBROOK said that the insurance companies had based their scale of charges on the results of a series of experiments made for them on the liability of different oils to take fire spontaneously or otherwise. They believed that the use of cotton seed oil in mills was very dangerous, and would not accept a mill known to make use of this oil.

Mr. BURRELL thought the experiments would be more valuable if oils other than cotton-seed had been more extensively employed as manufacturers did not use that oil owing to the prohibitive nature of the tariff.

It was not correct to say that the oils submitted by the tariff offices were simply tested for their flash-point and percentage of mineral oil. The insurance companies required the oils to be classified and this rendered it necessary to test for the dangerous seed-oils whenever their presence was suspected.

He thought that the use of the apparatus devised by Ordway would be preferable to a water-bath for the conduction of these and similar experiments.

The CHAIRMAN said that from the scientific point of view the investigation of the subject of the spontaneous inflammability of oils did not appear to present serious difficulties, and the most satisfactory plan both for the insurance companies and for mill owners would be doubtless to work out separately the behaviour of each oil in order to have a firm basis of ascertained fact to build upon.

Mr. MACKEY, in reply, said that it was difficult to say whether it was more dangerous to use a mineral oil whose flash point was, say, 350° F., and which was certainly not spontaneously inflammable, but which carried a fire when started very rapidly, or to use vegetable oils with a much higher flash-point (550° F. say) which were liable to spontaneous ignition. Mr. Pemberton's observations on oils which had been steamed agreed with the results of his own experiments, as the steamed oils would contain fatty acids owing to their partial saponification.

### Scottish Section.

Chairman: Robt. Irvine.

Vice-Chairman: J. Clark.

#### Committee:

G. Beilby.	J. S. Macarthur.
R. Cox.	E. Ostlere.
C. J. Ellis.	T. L. Patterson.
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D. Harris.	J. B. Readman.
G. G. Henderson.	H. Rose.
R. A. Inglis.	E. C. C. Stanford.
W. I. Macadam.	D. R. Stewart.

Hon. Secretary and Treasurer:

J. Stanley Muir, Chemical Laboratory, University of Glasgow.

SESSION 1894-95.

Dates of Meetings for 1895.

Glasgow. — Philosophical Society's Room, 207, Bath Street.  
January 8th; March 5th; May 7th.  
Edinburgh. — Philosophical Institution, 4, Queen Street.  
February 5th; April 2nd.

*Meeting held in the Philosophical Society's Rooms, 207, Bath Street, Glasgow, on Tuesday, December 11th, 1891.*

MR. R. IRVINE IN THE CHAIR.

### NEW FORM OF LAMP FOR BURNING PARAFFIN OIL WITH A SMOKELESS FLAME, FOR LABORATORY USE.

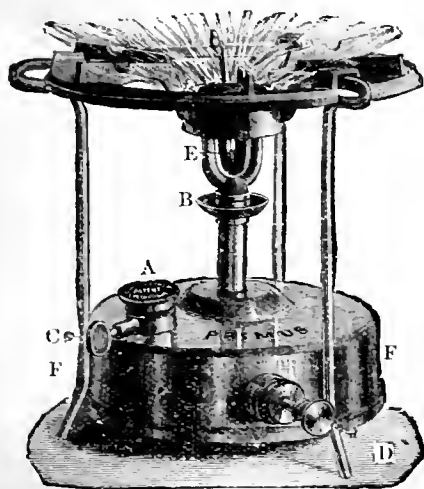
EXHIBITED BY MR. R. IRVINE.

MR. IRVINE explained that he had endeavoured to construct a lamp, producing a smokeless flame from paraffin oil, which would be adaptable as a "Bunsen" in laboratories where no supply of coal-gas was available. He had obtained some measure of success when he discovered that a lamp answering this purpose, the invention of B. A. Hjorth and Co., was in general use in Sweden and to some extent in this country for cooking purposes.

The patented apparatus which he exhibited has an air-tight reservoir F capable of containing about 2 pints of ordinary paraffin burning oil, and which can be filled at tubulure A. The burner is similar in construction to that of the well-known "showman's naphtha lamp." Directly below the burner is a small cup B, into which spirit of wine is poured. On igniting the spirit the coil of pipe E becomes red hot. By means of the small air-pump D, a few strokes of which is sufficient to compress the air in the upper portion of the reservoir, the oil is forced through the red-hot coil and becomes vaporised. The oil-vapour escapes through a small orifice in the coil and burns with a flame which closely resembles that of the Bunsen burner. The heat of this flame maintains the gasifying temperature in the "retorting" apparatus. Thus, so long as the pressure is maintained and the oil supply lasts, no further addition of spirits of wine is necessary, and the lamp continues to burn with an intense, smokeless, and odourless blue flame.

By turning the screw C, and thus relieving the air-pressure on the surface of the oil, the lamp is extinguished.

The amount of oil consumed during a period of from 20 to 30 hours costs about 1d.



MR. J. GRAY asked what pressure the reservoir of the lamp was guaranteed to stand.

THE CHAIRMAN believed that each lamp-reservoir was tested up to a pressure of 45 lbs. per square inch, but in ordinary circumstances it would be impossible for so small a pump to give a higher pressure than from 10 to 15 lbs. That pressure was amply sufficient to keep the lamp burning without again using the pump for from three to four hours.

MR. J. GRAY saw numerous applications for lamps of the kind. He asked if there was no probability of the gasifying tubes becoming choked with carbon.

THE CHAIRMAN replied that he considered the application of the principle of the lamp to be capable of great practical extension, especially for the purpose of steam-raising. In most oil-burners cold oil was forced into the furnaces by means of a great excess of steam or cold air. Thus the flame was robbed of much heat. With good oil the gasifying tube would not foul to any extent. If the tube choked it could readily be cleaned by means of a wire.

MR. BERRY inquired if there was any chance of "firing back."

THE CHAIRMAN thought not, so long as the pressure was maintained on the surface of the oil.

THE members present then took part in a discussion bearing on subjects referred to by the Chairman in his inaugural address. (This Journal, 1894, 1038—1047.)

## New York Section.

Chairman: Alfred H. Mason.

Vice-Chairman: Arthur McGeorge.

Committee:

G. T. Bruckmann.	John McKesson.
T. Lynton Briggs.	W. H. Nichols.
H. Comer.	Francis J. Schleicher.
H. Endemann.	Jas. H. Stebbins, jun.
Jos. D. Geisler.	T. B. Stillman.
Jas. Hartford.	J. H. Wainwright.
E. G. Love.	

Hon. Treasurer: R. C. Woodcock.

Hon. Local Secretary:

Dr. H. Schweitzer, 159, Front Street, New York, U.S.A.

The following gentlemen have promised papers:—

Dr. G. Archbold: "Technology of Starch."

Mr. H. Comer: "On Cresote."

Mr. E. N. Dickerson: "The relation of the U.S. Patent Laws, and Decisions thereunder, to American Chemical Industry."

Dr. H. Endemann: "Magnesia as a Waste Product, and its Uses."

Mr. E. E. Luzzwitz: "Commercial Analysis of Whale Oil."

Dr. R. C. Schupphaus:

(1.) "Evolution of Smokeless Powder."

(2.) "Chemical Nature of Celluloid."

*Meeting held at the College of Pharmacy,  
Monday, 5th November 1894.*

### CHAIRMAN'S ADDRESS.

BY ALFRED H. MASON.

GENTLEMEN,—When I yielded to the call to preside at the preliminary meeting held to consider the desirability of forming a New York Section of the Society of Chemical Industry, I did not anticipate its being more than a temporary situation, and was willing to do my utmost to accomplish the object we had in view; but owing more to the generosity of your committee than to any fitness of mine, I have been called upon to fill the position of first chairman of the Section, and I am convinced, from the interest already shown, that in the near future this Section will prove no mean part of the whole.

The formation of this Section creates a new phase in the history of the Society. There are 350 members in the United States and Canada, and we are heartily welcomed by the Council. The President in his annual address says:—

"We are pleased to add the 'Stars and Stripes' to our highly respectable old colours, and to shake hands across the ocean with the great English-speaking race, who 'shall brothers be for n' that.'"

May we not add to this, that we hope the day is not far distant when the shaking will be hand in hand, and the

Annual Meeting of the Society be held on this side of the Atlantic. In this connection the Annual Report says:—

"It is with great pleasure that the Council announces that it has acceded to the request of some of our fellow-workers in the United States to form a Section in New York. This is the first time that such a request has been preferred from abroad, and makes a new stage in the history of the Society, the importance of which cannot be too highly estimated."

We believe there is room for our Society in America. It was founded, not for the benefit of a class, but rather for the purpose of blending together the manufacturing branch on the one hand, and the abstract on the other, and that amongst English-speaking chemists. We have no wish to stand in the way of, or appear as rivals to, any other organisation, but rather as coadjutors with them. The American Chemical Society, with its sections in different parts of the country, has theoretical work to do, just the same as the Chemical Society in London has, and does it; and many members and fellows of both Societies are also members of this Society, recognising that the industrial applications of chemistry have become so numerous that the existence of a separate body to especially consider this branch is desirable. That such a want is filled by this Society, is proved by its remarkable growth.

### *Chemistry and Pharmacy.*

In the sixteenth century, that new race of alchemists, or pyrragists, as they were termed, arose, who, abandoning the search for the philosopher's stone, began to direct their energies to the discovery of chemical remedies for the various diseases of the body. "The true use of Chemistry," says Paracelsus (1493-1541), "is not to make gold, but to prepare medicines." It is not my purpose to call to your minds the various theories which followed this starting point, but it appears to me that I must have been impressed by the statement of Paracelsus, my life having been devoted to the study and practice of pharmaceutical chemistry in all its ramifications, though I have not yet discovered the way to make gold for myself. I make this, however, my apology, if such be needed, for asking your attention first of all to this branch of industrial chemistry.

We are the inheritors of the accidental success of these ancient chemists, and though we have outgrown their system, and have a truer knowledge of the workings of nature, we can get no wider field of aspiration than the health and wealth of mankind. Finding the human constitution much less refractory than that of the metals, the art of the chemist came to be regarded as almost exclusively applicable to the production of various powerful medicinal agents, and the value of his labours in dealing with inanimate nature was for a time almost forgotten. Chemical science, however, is able to provide for the wealth as well as for the health of mankind, and it is most encouraging to see in how many directions the work of this Society reflects the many-sidedness of the science.

There can be no doubt that pharmaceutical progress is chiefly to be won by the assistance of chemistry. The work done in this country is recorded in the several journals and transactions of colleges and societies devoted to the purpose, and we have a right to regard with considerable pride that work as part of the subject we are dealing with.

There are few subjects more interesting, either to the scientific or to the practical chemist, than the active principles of plants. The pharmacologist wants to know from the chemist the construction as well as the composition of the larger molecules; and until the chemist can tell him the exact manner in which such large molecules, as, for instance, morphine and strychnine, are built up, and until the pharmacologist has obtained a wider knowledge of the smaller molecules, he cannot understand the effects of the modification which can be produced in these larger molecules. At present he does not comprehend why substituting a molecule of methyl for one of hydrogen in morphine weakens its sleep-producing and pain-quelling power, whilst the subtraction of a molecule of  $\text{H}_2\text{O}$ , takes both away, and converts it into a powerful emetic; but it is to the chemist he looks to obtain

this knowledge, and with such assistance new and more powerful remedies may be constructed.

The study of the natural alkaloids has not only enriched science with a class of bodies of endless interest and opened out fields of researches of the highest promise, but has been of the greatest practical benefit to the human race. Owing to the great complexity and ready destructibility of these molecules, their synthesis, with few exceptions, appears so far to have eluded research.

Researches in this direction are of the highest theoretical interest, but whether they will ever bear fruit industrially in the production of natural alkaloids is another question. Indirectly such researches have already borne fruit in the discovery of a class of alkaloids not found in nature which possess therapeutic properties more advantageous than natural products. It is to Germany chiefly that we are indebted for these developments, because there so much skilled labour is bestowed upon, and so much encouragement given to, original research. In this country, with its abundant supply of medicinal plants, both native and acclimatised, we naturally look to the chemist for developments in this branch of our art.

The 1880 census gives 592 manufacturing druggists and chemists in this country, 112 of these in New York State, 72 in Pennsylvania. By the enterprise of these establishments pharmaceutical and chemical preparations are manufactured on an enormous scale under the most favourable circumstances, and with such excellent results that the United States takes a leading position amongst the manufacturing chemists of the world.

American pharmacists were the first to manufacture fluid extracts of standard strength, of which one minim represents one grain of the drug. These are now manufactured in many laboratories, and this system has been adopted officially in several other countries. It is not our province to enlarge upon the advantages of this class of preparation, but we may state further that America was the first country to introduce what is known as "elegant pharmacy," by which life is made more pleasant, and nauseous medicines even tempting.

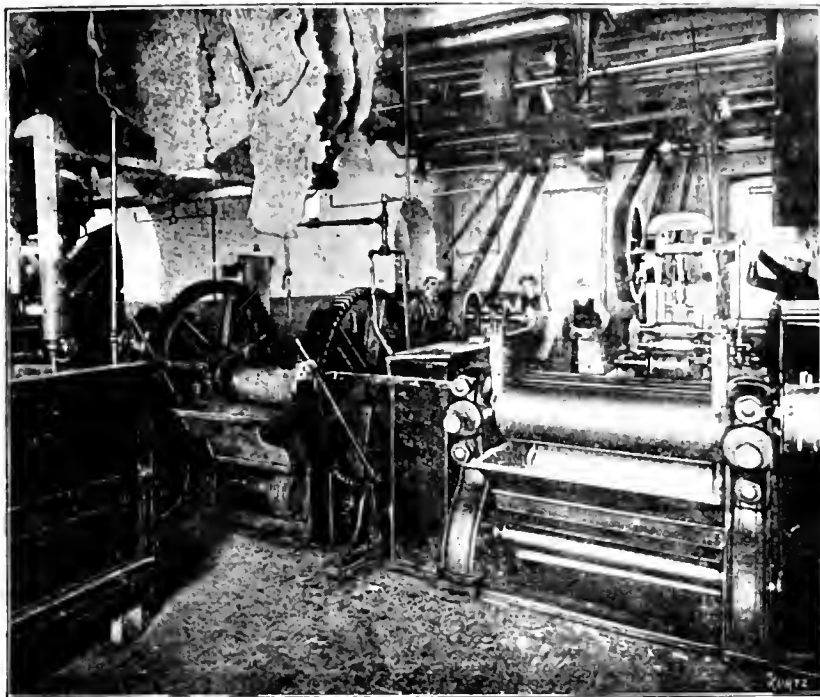
The manufacture of eclectic remedies, resinoids, &c., is also of importance. In a nation noted for its dyspeptics the study of digestive ferments would naturally be one of importance. In the manufacture of pepsin America takes the lead, and there are several manufacturers who have made their reputation in this industry. Another important domestic industry is the manufacture of spread plasters and surgical dressings on scientific principles. Some years ago Colonel Shecht, of the United States army, invented a process which, by the incorporation of india-rubber with inoffensive gums in a plaster mass, produced an article which, when spread upon cloth and perforated, would stick without warming. This was a mechanical plaster, but in 1876 Seabury conceived the idea of incorporating with a rubber combination such standard medicaments as belladonna, opium, aconite, &c., and perfected an industry purely American. Other manufacturers have followed in his track, and a large home and export trade is done. It is computed that at least 20 millions of porous plasters are manufactured annually in the United States and distributed all over the world, in addition to large quantities of spread plaster distributed in rolls. In one factory alone 33,000 pounds of belladonna root, yielding 8,000 pounds alcoholic extract, was employed for this purpose in one year.

In making these plasters Para india-rubber is first soaked and steamed and then passed through spirally corrugated iron rolls under a stream of water until well cleansed. The resulting crinkle sheets are hung up to dry, and then worked to a homogeneous mass on warm, smooth calender mills. The steaming tank, washing mill, grinding calender, and some sheets of worked rubber are shown in Plate 1.

Olibanum is prepared for use by stamping and sifting: Burgundy pitch and other resinous matter, by melting and straining.

The rubber, resins, and other ingredients are worked with the extract to a uniform mass on warm calender mills. No solvent is employed, the force of iron rolls doing the entire work. When thoroughly incorporated, and of just the right temperature, the mass is spread on cloth by means of heavy calenders (Plate 2).

Plate 1.

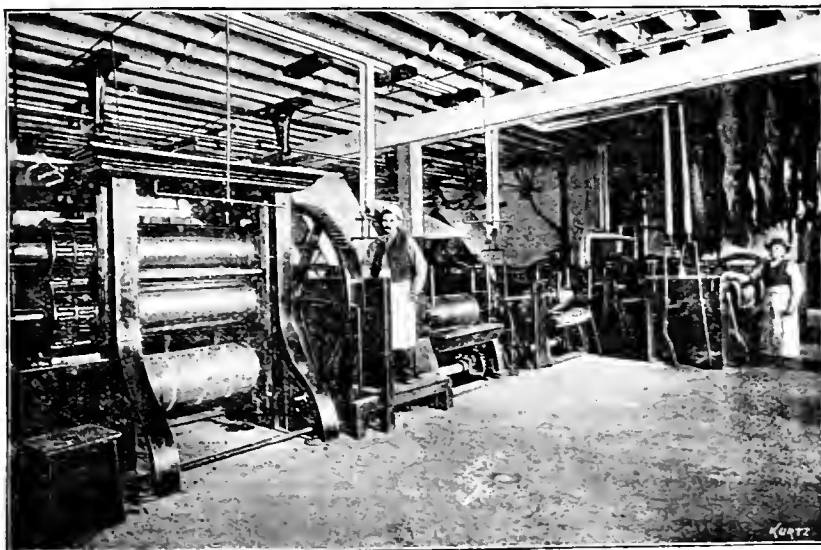


The cloth, usually about 120 yards long by 1 yard wide, is fed through the calenders, and passing under the lower rolls is connected with a large reel behind the machine. The calenders, being gauged for the right thickness, are started, and the mass fed upon the cloth between guides. After standing a time on the reels, the material is wound up on drums, cut, and finished. Punching machines for perforating the plasters are used.

The great advantages of the rubber combination base are its remarkable keeping qualities and permanent preservation of the incorporated medication as well as lasting flexibility, a great improvement for which mankind is indebted to America.

The exhibits at the Columbian Exposition at Chicago last year show how wide a field is claimed for chemistry and for the chemical industries of this country, and yet

Plate 2.



even the most careful classification cannot include all the directions in which chemistry has found, or will find application.

Even the 23 heads, under which the abstracts of our Journal are divided, do not always include all the subjects which really belong to us, and even in those industries which are now undoubtedly within our field we find not a few branches where as yet chemistry is but imperfectly applied, and where still the empiric stage has hardly yielded to strict science; and other branches where processes really chemical are carried on without even an attempt to bring science to bear upon them. We ought not to rest content till science shall have pervaded every operation where chemical action is involved; and though this may seem to open up an over-wide field, where can we stop short of such a claim?

The census of the United States in 1880 gave 1,349 chemical establishments in operation, that in 1890 gave 1,626 such establishments. I have tabulated statistics of the production of several articles for the sake of comparison, but the report on chemicals, which will form a part of the general report of manufactures, is still in the hands of the printer and will not be available until the completion of the whole report. I, however, sent a list of articles to the Commissioner in Washington, and he courteously complied with my request as best he could; but the classification adopted in the 1890 census is not the same as that of 1880, so it is impossible to compare all the items. However, such statistics as will be published then are also tabulated.

PRODUCTION OF CHEMICALS IN THE UNITED STATES  
ELEVENTH CENSUS, 1880.—1,349 ESTABLISHMENTS IN OPERATION.

Class of Products.	Quantity.	Value.
	Lb.	Dols.
Ammonia (sulphate of) .....	16,575,988	618,485
Alum .....	39,217,725	808,165
Aniline colours .....	80,518	107,292
Anturacene .....	344,114	99,242
Borax .....	3,692,143	277,233
Bromine .....	494,890	114,752
Phosphorus .....	56,292	29,271
Stearic acid candles .....	18,363,066	2,281,609
Oleic acid soap .....	33,058,411	1,707,969
Other hard soaps .....	378,743,627	18,290,350
Soft soaps .....	34,454,100	558,280
Glycerin .....	7,117,825	961,477
Nitro-glycerin .....	3,639,722	1,830,417
Manufactured manures .....	727,453	19,921,400
Dry colours .....	67,482,415	4,088,821
White lead .....	123,477,899	8,770,699
Other salts of lead .....	11,375,466	758,689
Ground barytes .....	19,165	371,829
Zinc oxide .....	29,121,761	766,337
Acetate of lime .....	6,593,069	156,892
Potash and pearlash .....	4,516,671	232,643
Soda .....	40,259,938	866,569
Sulphur .....	1,200,000	21,600
Sulphuric acid .....	308,755,432	3,061,876
Glucose .....	151,740,400	4,551,212
Other products .....	..	44,927,101
Total value of all products .....		117,377,324

The total value of all products in the 1880 census report was 117,007,976 dols., and in the 1890 census report 137,007,976 dols. The total value of chemicals, drugs, and dyes imported in 1890 was 26,817,265.69 dols. free of duty, and 14,607,523.94 dols. dutiable. The comparison can only be made in a few instances.

Some of these, however, were produced by only a few establishments and in limited quantities, the greater portion consumed within the United States being imported; for instance, only one establishment mined and produced sulphur, yielding 1,200,000 lb., while 176,283,000 lb. were imported; three establishments produced 80,518 lb. of aniline colours, while 176,238,600 lb. were imported; one establishment produced 56,292 lb. phosphorus, valued at 29,271 dols., while the importation was valued at 78,253 dols.; three establishments produced 40,259,938 lb. of soda salts, while 360,301,309 lb. were imported; and six establishments produced 39,217,725 lb. alum, while 2,112,570 lb. were imported.

ELEVENTH CENSUS OF THE UNITED STATES.

*Statistics of Manufactures.*

STATEMENT showing QUANTITIES and VALUES of SELECTED PRODUCTS reported by ESTABLISHMENTS engaged in the MANUFACTURE OF CHEMICALS and ALLIED PRODUCTS during the CENSUS YEAR ending May 31, 1890.

Class of Products.	Quantity.	Value.
Alum .....	Lb. 93,998,008	Dols. 1,616,710
Coal-tar products .....	..	687,591
Dyeing and tanning extracts:		
Extracts .....	Lb. 68,537,940	7,081,806
Chipped wood and other products under this head.	" 119,368,971	1,775,278
Gunpowder .....	" 95,013,174	6,740,099
High explosives .....	" 30,625,738	4,253,032
Fertilisers:		
From mineral phosphates .....	Tons 1,276,708	23,122,448
From raw bone .....	" 274,759	6,620,925
All other .....	" 347,339	5,776,468
Pigments:		
White lead .....	Lb. 143,620,471*	3,297,627
Oxide of lead .....	" 24,602,067*	1,238,577
Barytes (ground or floated) .....	" 43,143,900	377,939
Oxide of zinc .....	" 17,648,000	685,920
Lampblack and hydrocarbon blacks .....	" 11,617,396	608,800
Fine colours .....	" 27,215,687	3,481,349
Iron oxide and other earth colours .....	" 138,372,483	1,068,800
Pulp colours (so'd moist) .....	" 10,440,763	507,918
Paints in oil, in paste .....	" 211,545,504	13,448,569
Paints, ready mixed for use .....	Galls. 12,716,101	12,947,315
Varnishes and japans .....	" 14,535,202	13,987,268
Pharmaceutical preparations .....	..	16,744,643
Potash and pearlash .....	Lb. 5,106,939	197,507
Soda ash .....	" 94,801,200	1,179,720
Sal soda .....	" 144,641,705	1,581,766
Bicarbonate of soda .....	" 60,678,750	2,009,900
Caustic soda .....	" 33,002,720	661,114

\* Establishments engaged in the manufacture of white lead, oxide of lead, and sulphuric acid, report quantities of these articles consumed as intermediate products. In such cases the quantity is included in the total quantity reported; the value of the same is not shown. The quantity of white lead manufactured in this manner was 80,603,330 lb.; of oxide of lead, 312,252 lb.; and of sulphuric acid, 581,536,200 lb.

## Statistics of Manufactures—cont.

Class of Products.	Quantity.	Value.
Sulphuric acid:		Dols.
50° Beaumé .....	Lb. 1,009,683,407*	1,826,372
60° Beaumé .....	" 20,379,908	122,940
66° Beaumé .....	" 354,533,657	3,249,466
		137,007,976

In the eleventh census the quantity of alum produced had increased to 93,995,008 lb., whilst the total imports of alum and aluminous materials of all kinds was 6,832,025 lb. The production of potash and pearlash increased about 500,000 lb., but the value decreased materially. The production of soda and soda salts was 333,124,357 lb., against 40,257,938 lb. in 1880, whilst the imports were 364,723,196 lb., and the production of sulphuric acid was 956,449,765 lb., against 308,765,432 lb. in 1880.

TABLE OF CERTAIN CHEMICALS ENTERED FOR CONSUMPTION IN 1893.

## Duty free.

Article.	Quantity.	Value.
		Dols.
Alizarin .....	Lb. 5,729,221	1,125,506*00
Aniline arseniate .....	" 46,015	2,847*00
" salts .....	" ..	432,134*00
Argols, crude tartar .....	Lb. 28,740,279	2,341,575*00
Acid, salicylic .....	" 260,027	" ..
" carbolic .....	" 1,411,618	" ..
Bromine .....	" 780	" ..
Barytes carbonate .....	" ..	21,640*00
Quinine sulphate .....	Ozs. 3,027,819	" ..
Cobalt and cobalt ore .....	Lb. 5,314	" ..
Cryolite .....	Tons 8,549	111,569*00
Iodine, crude .....	Lb. 327,248	589,186*09
Bleaching powder (chloride of lime) .....	" 120,780,233	2,212,606*00
Citrate of lime .....	" 629,739	75,271*00
Magnesite .....	" 8,209,600	16,782*00
Manganese ore .....	" 192,279,674	" ..
" oxide of .....	" 874,340	" ..
Potash, black ashes .....	" 10,115,097	" ..
" caustic .....	" 2,338,868	" ..
" chlorate .....	" 4,064,176	" ..
" muriate .....	" 73,387,180	" ..
" nitrate .....	" 16,569,403	" ..
" sulphate .....	" 13,846,774	" ..
Total potash .....	" 120,321,418	" ..
Sulphur .....	Tons 119,025,010	" ..
Verdigris .....	Lb. 87,125	" ..
Total value of chemicals, drugs, and dyes free of duty, 1893.		36,601,283*14

\* See note on preceding page.

## Dutiable.

Article.	Quantity.	Value.
		Dols.
Acid, benzoic .....	Lb. 771,775	" ..
" citric .....	" 13,315	" ..
" tartaric .....	" 170	" ..
Ammonia carbonate .....	" 554,824	" ..
" nitrate .....	" 1,217,925	" ..
" sulphate .....	" 14,925,759	" ..
Cobalt oxide .....	" 35,729	" ..
Copper sulphate .....	" 8,941	" ..
Glycerin, crude .....	" 14,325,111	" ..
" refined .....	" 248,795	" ..
Copperas .....	" 1,010,069	" ..
Calomel, mercuric .....	" 21,416	" ..
Magnesia carbonate (medicinal) .....	" 66,316	" ..
" sulphate .....	" 61,337	" ..
Morphine .....	Ozs. 23,580	" ..
Phosphorus .....	Lb. 89,874	" ..
Potassium bicarbonate .....	" 74,983	" ..
" bichromate and chromate .....	" 979,067	" ..
" nitrate, refined .....	" 129,246	" ..
" prussiate, red .....	" 16,697	" ..
" " yellow .....	" 1,947,910	" ..
Soda bicarbonate .....	" 1,193,310	" ..
" bichromate .....	" 671,503	" ..
" caustic .....	" 55,531,899	" ..
" sal. cyst .....	" 27,777,488	" ..
" ash .....	" 388,409,970	" ..
" sulphate .....	Tons 218	" ..
Saltcake .....	Lb. 3,363,886	" ..
Sulphur, sublimed .....	Tons 128	" ..
Tartars and lees (crystals) .....	Lb. 20,145	" ..
Total value chemicals, drugs, dyes, &c., 1893 .....		15,768,332*40
Duties .....		4,769,213*84

All the crude materials on this list were used for the manufacture of aniline colours, cream of tartar, tartaric acid, citric acid, resublimed iodine, &c., whilst dutiable goods were required in many instances owing to the manufacture here not having met the demand. For instance, salicylic acid is manufactured in the United States, and probably demands could be met for this; but a certain foreign brand is called for. Although quinine is imported in large quantities, 3,000,000 ozs. are being made annually in this country (three factories being erected for its manufacture, but only two running). Carbonate and sulphate of ammonia are manufactured here. Sulphate of copper is manufactured and exported in large quantities. Glycerin, although imported largely, is produced here in large quantities. Phosphorus is manufactured and consumed in the manufacture of matches, &c., yet we require to import 90,000 lb. more to meet the demand. Morphine is manufactured, but, a special brand being called for, has to be imported.

### NON-METALLIC PRODUCTS OF THE UNITED STATES IN 1893.

Products.	Quantity.	Value.
		Dols.
Bituminous coal..... Long tons	114,629,671	122,651,618
Pennsylvania anthracite.... "	48,185,306	85,687,078
Lime..... Barrels	58,000,000	35,960,000
Building stone..... "	..	33,845,573
Petroleum..... Barrels	48,412,666	23,932,326
Natural gas..... "	..	14,346,250
Clay (all except potters' clay)..... "	..	9,009,000
Cement..... Barrels	8,002,467	6,262,841
Mineral waters..... Galls. sold	23,544,495	4,246,734
Phosphate rock..... Long tons	941,568	4,136,070
Salt..... Barrels	11,816,772	4,054,068
Limestone for iron flux..... Long tons	3,958,055	2,574,833
Zinc white..... Short tons	21,059	1,604,420
Potters' clay..... Long tons	490,000	900,600
Gypsum..... Short tons	253,615	696,615
Borax..... Lb.	8,699,000	652,425
Mineral paints..... Short tons	37,714	530,284
Fibrous tale..... "	35,861	403,436
Asphaltum..... "	47,779	372,232
Soapstone..... "	21,071	255,067
Precious stones..... "	..	264,011
Pyrites..... Long tons	83,277	275,302
Corundum..... Short tons	1,713	142,325
Novaculite..... Lb.	..	135,173
Mica..... "	66,971	88,929
Barytes..... Short tons	28,970	88,506
Bromine..... Lb.	348,399	104,520
Fluorspar..... Short tons	12,400	84,000
Feldspar..... Long tons	18,391	68,037
Manganese ore..... "	7,718	66,614
Flint..... "	23,671	63,792
Graphite..... Lb.	843,103	63,232
Sulphur..... Short tons	1,200	42,000
Marls..... "	75,900	40,000
Infusorial earth..... "	..	22,582
Millstones..... "	..	16,645
Chromic iron ore..... Long tons	1,450	21,750
Cobalt oxide..... Lb.	8,422	10,346
Magnesite..... Short tons	704	7,040
Asbestos..... "	50	2,500
Total value of non-metallie mineral products.....		358,830,804
Total value of metallie products.....		249,981,866
Estimated value of mineral products, unspecified*..		1,000,000
Grand total.....		609,812,670

### METALLIC PRODUCTS OF THE UNITED STATES IN 1893.

Products.	Quantity.	Value.
		Dols.
Pig iron..... Long tons	7,124,502	84,810,426
Silver..... Troy ozs.	60,000,000	77,575,755
Gold..... "	1,739,081	35,950,000
Copper..... Lb.	337,416,848	32,054,601
Lead..... Short tons	163,982	11,839,590
Zinc..... "	78,832	6,306,560
Quicksilver..... Flasks	30,164	11,108,527
Aluminum..... Lb.	339,629	266,903
Antimony..... Short tons	250	45,000
Nickel..... Lb.	49,339	22,197
Tin..... "	8,938	1,788
Platinum..... Troy ozs.	75	517
Total value of metallic products.....		249,981,866

#### Natural Advantages for Production.

**Coal.**—Among the materials used, fuel is an important item for chemical establishments, and, other things being equal, cheap coal will invariably draw the manufacturing chemist to its neighbourhood. The world's annual output being about 488 million tons, America stands second in the list as a contributor to this enormous output. Pennsylvania yields nearly all the anthracite, the bituminous coal being found in (1) the Triassic fields, (2) the Appalachian fields, (3) the Southern fields, (4) the Central field, (5) the Western field, (6) the Rocky Mountain field, (7) the Pacific fields.

**Coke.**—Pennsylvania is still the chief coke-producing State, contributing 65·8 per cent. of the total, and Alabama second, contributing about 12·2 per cent. In New York State during the past year, 12 hy-product ovens, on the Semet-Solvay principle, have been built at Syracuse, N.Y., and treated Pennsylvania coal. The operation of these has been highly successful. Coals, that have not been regarded as very high grade coking coals, have been used with most gratifying results. The yield of coal in coke was 84·18 per cent., including not only large coke, but "breeze" as well.

**Petroleum.**—While petroleum has been found in every State and territory, the localities in which it has been obtained in paying quantities are few. Practically, all the petroleum produced in the United States is from four districts: the Appalachian; the Limestone, or Ohio, Indiana; the Florence, of Colorado; and Southern California fields.

The world's production for 1893 was, approximately, 84,330,809 barrels (of 42 galls. each); of this, the United States produced 48,412,666 barrels, or 57 per cent.

During the year 1883 most of the illuminating oil used in the United States was produced from the limestone oil of Lima and Indiana, 1893 having marked the era of complete success in refining these oils.

**Natural Gas.**—Petroleum and natural gas have a common origin, and are both members of that wonderful series of chemical compounds known as paraffins, of which paraffin wax may be regarded as a representative of the solid portion of the series; petroleum, the liquid portion; and natural gas as the best known gaseous member. This gas is fuel of high value, and was largely utilised for industrial and domestic purposes at such great industrial centres as Pittsburg. One million cubic feet of gas are said to do the same amount of heating as about 60 tons of anthracite coal; but in Pittsburg to-day the price charged for natural gas to manufacturers is such that, ignoring the cost of plant, producer-gas can be supplied to these works

\* Including building sand, glass sand, limestone used as flux in lead-smelting, limestone in glass-making, iron ore used as flux in lead-smelting, tin ore, iridosmine, nitrate of soda, carbonate of soda, sulphate of soda, bauxite, and alum clays used by paper manufacturers.



as cheaply. In Pennsylvania natural gas first began to be used extensively as a domestic and industrial fuel.

Two hundred and eighteen companies for the production of natural gas reported to Washington, in 1893, the following numbers of producing wells owned by them. In Pennsylvania there were 841; in Indiana, 498; Ohio, 207; and these appear to be increasing. Natural gas for fuel purposes is used very largely in the western resident portion of Buffalo, and extensive additions are nearly completed for supplying a wide area on the east side. The sources of supply are the wells of Pennsylvania, Canada, and many small outlying districts east of Buffalo—a great factor in the reduction of consumption of coal in the district.

**Asphaltum.**—Deposits of asphalt are widely scattered over the United States, principally south of latitude 40°. The importations from the West Indies, North Coast of South America, and Europe, amounted to 74,774 short tons from Trinidad and 65,420 tons from Beyrout, value 21,207.44 dols. in 1893.

The commercial applications of asphalt in this country are for roadways, walks, roofs, and floors in the form of compressed asphalt and mastic, and for varnish and paints, waterproofing compositions, roofing felts, insulators, bituminous masonry, and concrete.

Professor Sadtler, of Philadelphia, has been studying the matter of asphalt paving composition, and the results of his experiments will be found in the journal "Paving and Municipal Engineering," Vol VII., September 1894. He concludes that better, stronger, and probably more durable paving composition, can be made from oil residuum.

**Ozokerite.**—In 1889, 75,000 lb. of crude ozokerite were produced, and the product of 1891 probably exceeded 300,000 lb. Petrolatum is an article largely exported.

**Sulphur.**—The total product of sulphur in 1893 was all mined by the Utah Sulphur Company at Black Rock, Utah, 105,529.00 long tons of crude sulphur, 41.00 of refined sulphur, and 721,699 tons of sulphur ore were entered for consumption during 1893.

**Pyrites.**—The quantity of pyrites mined in 1893 was 83,776 long tons, but the quantity used for acid-making was only 75,777, being less than any year since 1888. The product was entirely from Massachusetts and Virginia. An experimental enquiry by Mr. T. S. Gladding, into the reliability of the various methods of estimating sulphur in pyrites, is published in the "Scientific American," October 1894.

**Sulphuric Acid.**—The relative merits of sulphur and pyrites as raw materials for the manufacture of sulphuric acid have been considerably discussed. American writers have attributed the larger use of brimstone in this country principally to the outlay necessary to make the changes in furnaces, which manufacturers hesitated to incur.

Mr. Karl F. Stahl claims that, "besides the somewhat more complicated and more costly construction of furnaces, and the constant higher cost of labour in burning pyrites, there are other factors in favour of brimstone:—(1.) It requires less chamber space to produce the same amount of acid. (2.) Lead chambers are said to last longer with brimstone than with pyrites. (3.) The acid produced is of different quality."

The first proposition is doubtless true. In regard to the second, Mr. Stahl admits that it is difficult to get exact data, and states that while the higher temperature of the gases (and not the arsenic in the pyrites, as is claimed by some) is detrimental to the lead, this could be avoided by proper construction so as to cool the gases before entering the first chamber.

Mr. Stahl gives the following estimate for the relative cost of acids from brimstone and pyrites.

#### Cost of Chamber Acid from Brimstone.

	Dols.
2½ tons brimstone (5,600 lb.) at 21 dols. per ton.....	52.50
225 lb. nitre (= 4 per cent. of the brimstone) at 4.50 2 cents per lb.	4.50
220 lb. sulphuric acid, 60° B., at 40 cents per 100 lb. . .	0.88

Four men at 1.50 dols. (two firemen for boilers included).	6.00
Wear and tear on building, furnace, chambers, &c., 40 cents per ton chamber acid produced.	5.40
Repairs on buildings, furnace, chambers, &c., 20 cents per ton chamber acid produced.	2.70
Fuel, lights, &c. ....	2.00
Office expenses.....	5.00
Interest on capital invested.....	5.00
Product:—134 tons (2,904 lb. each) .....	83.98

One ton chamber acid, 50° B., cost 6.25 dols.

#### Cost of Chamber Acid from Pyrites.

	Dols.
5½ tons pyrites (= 12,324 lb., 37 available sulphur, at 4.00 dols.)	33.00
180 lb. nitre (= 4 per cent. of the available sulphur) at 2 cents per lb.	3.60
220 lb. of sulphuric acid, 60° B., at 40 cents per 100 lb.	1.00
Breaking of the pyrites, and removing the residue ..	1.00
Six men at 1.50 dols. (two firemen included for boilers).	9.00
Wear and tear on buildings, furnaces, chambers, &c., 50 cents per ton chamber acid produced.	5.50
Repairs on buildings, furnaces, chambers, &c., 25 cents per lb. chamber acid produced.	2.75
Fuel, light, &c. ....	2.00
Office expenses.....	5.00
Interest on capital invested.....	5.50
Product:—11 tons (2,000 lb. each) = 22,000 lb., cost ..	68.75

One ton chamber acid, 50° B., cost 6.25 dols.

From this table it will be seen that pyrites is taken as costing 6.00 dols. the long ton, and the cost of acid made from it is 3 cents more per ton than from brimstone. But the price of pyrites is too high, the average price for 1893 being 3.50 dols. per long ton, and a fair average for the past three years would not be more than 3.00 dols. per ton; and with the pyrites at 3.50 dols. per ton, the cost of acid would be 5.00 dols. instead of 6.25 dols. per ton.

With the United States almost entirely dependent upon Sicily for her supply of brimstone, there seems to be little reason why our pyrites deposits should not be made the source of supply for commercial acid, which can be made as well and cheaply in the United States as anywhere, the conditions being the same, though labour is 50 to 100 per cent. more than in foreign countries, and almost exactly 100 per cent. more than was paid 48 years ago.

Canadian competition is a matter of some importance; the Canadians manufacture more than they can use, and sell the balance in our market at an advantage which manufacturers here cannot compete with, owing to their protective rate of ½ cent per lb.

Sulphuric acid is largely manufactured in this country at different factories in the east, especially the petroleum refineries and the fertiliser factories, where they have plant for the purpose.

**Salt.**—The salt product of the United States in 1893 was as follows:—

Table salt .....	1,021,203
Pure .....	767,374
Common, fine.....	5,475,054
" coarse.....	443,498
Packets .....	95,557
Solar .....	2,110,287
Rock .....	1,884,145
Milliner .....	5,141
Agricultural.....	6,413
Total value.....	4,051,658 dols.

It is produced in Michigan, New York, Ohio, West Virginia, Louisiana, California, Utah, Nevada, Kansas, Illinois, Virginia, Pennsylvania, Texas, &c.

American salt has been so improved by new processes, which each producer holds secret, that importation of refined salt has almost ceased. Table and dairy salts are now prepared practically chemically pure, free from gypsum, calcium chloride, and magnesium salts. The elimination of these impurities has been attempted for years, but only recently accomplished at a paying cost. The value



of imported salt entered for consumption in 1893 was 705,141.07 dols., the amount of duty paid 301,972.60 dols., the quantity of salt withdrawn from warehouse for curing fish 93,937,304 lb.

**Natural Sodium Salts.**—The geographical occurrence of natural carbonate of soda in the the United States is principally confined to the arid regions of the Great Basin, especially to the Soda Lakes, at Ragtown, Nevada; Mounslake, Mono County; Owens Lake, Inyo County; California and Albert Lake, Oregon; and to the many dry deposits and incrustations in the same regions.

These lakes are for the most part residues left by the evaporation of larger bodies of water, the shore lines of which can be traced at considerable distances, sometimes several hundred feet above the present desert. This concentration has increased the proportion of their mineral salts, and sometimes this concentration reaches the crystallising point, when the sodium carbonate appears as a white incrustation on the shores of the lakes.

The production of natural carbonate of soda in 1893 amounted to about 3,100 tons, of which one-fifth was soda ash and crystal carbonate.

The Wyoming sulphate has received new attention in the last two years. In 1892, 1,670 tons of *salt cake* were shipped from the Laramie works. Improvements were being made in the works in 1893, and new plants for producing the carbonate were erected in other places in Wyoming during the year. These causes interfered with the production of both salts.

**Natural Borax.**—The waters of the soda lakes also contain small quantities of sodium borate, which the volcanic neighbourhood easily accounts for.

In various places in the arid regions of California, Nevada, and further north, even in Eastern Washington, which form the volcanic belt, there are deposits of borax and borate of lime, which have been worked more or less successfully. The origin of the soda and the lime is to be ascribed to the feldspars of the volcanic rocks, which furnished those bases for the boracic acid which was supplied by the volcanic emanations. Some springs in the volcanic belt still contain traces of boracic acid. The borates formed in this way were concentrated in lakes or ponds in the same way as the carbonates until crystallisation took place. The present bed is former lake or pond deposits of this kind.

In 1892, 12,538,196 lb. of borax was produced; in 1893, 8,699,000. The falling off in 1893 was due to the dulness of trade. The average value from 1889 to 1894 has been 7½ cents per lb. At the time of the discovery of borax in California the price was 32 cents per lb. 543,967 lb. crude borates, 11,230 lb. refined borax, and 771,775 boracic acid for potters' use, were entered for consumption from bond in 1893, paying 53,469.26 dols. duty. Over 21,000 tons of borax are used annually, about one-third of which is produced in the United States.

The Occidental Alkali Company's property consists of a dry deposit of soda, about 800 acres in extent, situated at the Douth Springs, about four miles from the Carson and Colorado R.R., at Troua Station.

The deposit varies in depth from 5 in. 15 in. 18 in., and consists of sesquicarbonate of soda with some sulphate and chloride; its average composition is as follows:—

Na <sub>2</sub> CO <sub>3</sub> .....	39
NaHCO <sub>3</sub> .....	30
Na <sub>2</sub> SO <sub>4</sub> .....	15
NaCl.....	10
Insoluble (sand, &c.).....	10
Moisture.....	5
	109

The proportions vary slightly in different portions of the deposit, some parts yielding as high as 95 per cent. of carbonate and bicarbonate. When the surface deposit is removed, it renews itself, coming up at the rate of 1 in. per annum, and is practically inexhaustible.

The process of manufacture is as follows:—

1st. The crude soda is shovelled up in win rows, being allowed to stand some time. The rains dissolve the sulphate, raising the content of carbonate and bicarbonate.

2nd. The crude soda is hauled to works, where it is thrown into shallow wooden vats. Hot water is run in, and the whole mass stirred up, making a solution of 22° to 24° Baumé as it leaves the dissolver. The major portion of sand remains in the dissolver while the solution is run into a settling vat, where it is allowed to stand until clear. It is then drawn into ground vats, 200 feet square (which are dug in a peculiar impervious volcanic mud), during spring and early summer, until a depth of 18 to 24 inches is reached. As the season advances evaporation takes place, bringing the density of the solution up to 29° Baumé, when soda is precipitated in form of sesquicarbonate, or urao. (A mixture of carbonate and bicarbonate.) The soda thus precipitated is in the form of a hard cake, from 1 in. to 6 in. thick. When the bulk is precipitated, the solution is drawn off, and the cake taken out and piled up to dry. In this form it is used in the manufacture of borax from borate of lime, the excess CO<sub>2</sub> present being valuable as a precipitant for the lime of the borate of lime.

By heating to 150° or above, the moisture and free carbonic acid are given off, leaving a normal carbonate of soda or soda ash, often as high as 97, 98, and 99 per cent. Na<sub>2</sub>CO<sub>3</sub>.

After the crop of summer soda or urao is taken up, the solution is run back into the ground vat, care being taken to keep the density below 34°. As the season advances a crop of soda, low in carbonate and bicarbonate and high in sulphate, is thrown down. The solution is then weakened down to 25° to 26° B., at which point it is kept until frost occurs, when the entire contents as carbonate and bicarbonate, together with whatever sulphate is left, is thrown down as cold-weather soda, or hydrated carbonate, leaving chloride in solution. This salt solution is then run to waste (or into a separate vat, to precipitate salt for the following season) and the crop of cold-weather soda is taken out and piled up in the open air until warm weather approaches. It is then broken down and spread upon flat forms to dry, giving an effloresced soda (or crystal-carbonate so-called) which is very much liked by soap manufacturers for crutching, and by makers of washing powders, and is quite saleable as a substitute for common sal soda.

**Columbus Borax Company, Calville, Nev.,** manufacture principally concentrated borax and also boracic acid. They produced from 1887 to July 1893 about 50 tons concentrated borax per month, and since January 1894, about 30 tons monthly. Without Chinese labour they would have to shut down, prices being unremunerative.

**Phosphate Rock** is found in Florida and South Carolina. Phosphate nodules are found in Tennessee. Phosphate rock, containing about 50 per cent. of bone phosphate, is found at Castle Hayne, North Carolina.

**Fluorspar** is produced only in one locality in the United States, near Roseclare, Illinois. The product in 1893 was 12,400 short tons, valued at 84,000 dols. In addition to its use for metallurgical purposes, fluorspar is consumed in the manufacture of glass and of hydrofluoric acid.

**Aluminium.**—Practically the aluminium in the world is now in the hands of two large concerns, one being the Pittsburgh Reduction Company, now manufacturing at New Kensington with the advantage of cheap coal in the Pittsburgh seam (costing only about one half dollar per ton delivered at the works), and using compound engines and best forms of dynamos, pumping water from the Alleghany River, with works situated on their own property in the country. They are thus in a position to generate power as cheaply as possible with steam. They, however, expect to manufacture the metal at Niagara Falls with cheaper power. They have their own works there already completed, which will be in operation as soon as the Niagara Falls Power Company have their plant completed. The other company is the Aluminium Industrie Actien Gesellschaft, manufacturing at the falls on the Rhine at Switzerland, and making several tons of aluminium per day.

**Bauxite** is an ore which furnishes more and more of the raw material from which aluminium may be manufactured, and this is procured in Georgia and Alabama. The Arkansas bauxite deposits will probably be developed in 1894 for making alum.

The purity of commercial aluminium sold by the Pittsburg Reduction Company varies from 91 to 99.75 per cent. Of the output, a small proportion was in the form of aluminium alloys, especially bronze, but a large proportion of the bronze found in commerce is made from metallic aluminium previously obtained. The greater part of the product was used as an addition to steel. Nearly all the steel makers use a small proportion of aluminium, with the result of less waste in castings. For example, the amount of waste in crop ends of steel rails is lessened profitably. Ingot aluminium also is used for the manufacture of aluminium cooking utensils and novelties, and this industry is extending satisfactorily. The remainder of the product goes out as sheet and wire for many purposes, including printing from aluminium plates instead of zinc or stone.

**Copper.**—In addition to the product of American ores in 1893, 7,723,387 lb. were produced from imported pyrites. The exports of ore and matte, 835 cwt. 40 lb.; of pigs, bars, sheet, and old metal, 138,984,128 lb. Imports of fine copper in ore, 7,256,015 lb.; regulus and black copper, 3,175,559 lb. Sulphate of copper is largely manufactured in the United States, and is used for all technical purposes, for sprinkling vineyards, &c. The production is greater than the demand for domestic use, therefore large quantities are exported.

**Lead.**—American mines produced 356,781,000 lbs. pig lead, and 3,392,502 lb. were imported.

#### Paints.—

	Tons.	Lb.
The production of White lead .....	72,172	Import 686,400
" Red lead.....	6,122	" 854,982
" Litharge.....	11,077	" 42,582
" Orange mineral ....	257	
" Zinc white.....	24,059	
" Ultramarine .....	113,647	

**Bromine** is manufactured from the mother liquors of salt works, as obtained in Western Pennsylvania, West Virginia, the Ohio, and Kanawaba Valleys. Bromide of potassium is largely manufactured for consumption in this country, and for export.

**Acetate of Lime.**—Acetate of lime and its associate product, methyl alcohol, is manufactured on a large scale. Two qualities of acetate are produced, known as "brown" and "grey," the production of the latter forming about one-fifth of the total. In 1880, 35 firms were engaged in this manufacture, producing 550 tons of acetate and 600 barrels spirit per month. The manufacture is now in the hands of a syndicate, with an invested capital of about 4,000,000 dolrs., and the output is about 2,000,000 galls. wood alcohol and 20,000 tons acetate lime per annum.

In this connection, the manufacture of acetic acid may be mentioned, also the manufacture of chloroform from acetone and ketones, a purely American industry. The probable output is 300,000 to 325,000 lb. per annum.

**Coal-Tar Products.**—In 1883 there were nine different plants employed in the manufacture of coal-tar dyes, when protection made the production remunerative, but it is not probable that so many exist now, protection having been modified and the crude material having to be imported. The principal cause for non-development is as with many other industries—

- 1st. High wages.
- 2nd. Great first cost of plant and larger annual cost of wear and tear.
- 3rd. High cost of coal-tar preparations and other chemicals and materials.
- 4th. High tax on alcohol for industrial purposes.

Aniline oil, crude and distilled liquid carbolic acid, all kinds of aniline colours, coal-tar, pitch, creosote, dead oil, benzol, toluol, solvent naphtha, nitrobenzol, naphthalene, Turkey-red oils, &c. are manufactured.

**Ammonia.**—Sulphate of ammonia is obtained in this country from the ammoniacal liquor of gasworks and the bone liquor of bone works. The production of gas-liquor has steadily decreased for the past 10 years, owing to less coal-gas being required, until the quantity now is very insignificant. The sulphate of ammonia made by bone coal manufacturers is of inferior quality, fit only for fertilising purposes, and the entire product is small. The domestic production of carbonate of ammonia is only about one half of the total consumption. Anhydrous ammonia and *Liquor ammonia*, employed in the manufacture of artificial ice and the production of cold storage, are also largely manufactured.

**Fertilisers.**—The exhaustion of the soil in the eastern, middle, and southern States, has created a large demand for manufactured manures, particularly in the cotton and tobacco-producing sections, and their manufacture has increased to such an enormous extent, that the production of fertilisers has become one of the greatest industries in the United States, particularly in those districts where mineral phosphates are produced. The raw material commonly employed in the manufacture of commercial fertilisers are bone phosphate, rock, cotton-seed meal, blood, fish, tankage, sulphate of ammonia, nitrate of soda, chloride of potassium, sulphate of potash, and kainit and such materials.

The quantity produced in 1890, from different sources, was 1,897,806 tons.

The agriculture of every State in the union is watched by bands of trained scientific men, who take special interest in the State in which they are located. By Federal Acts of 1862 and 1890 large grants of land and money are made for the support and maintenance of at least one agricultural college in every State. The Central Office of the Experiment Stations is at Washington, with Dr. H. M. Wiley at its head.

From this body is organised the Association of Official Agricultural Chemists, formed "to insure uniformity and accuracy of the methods, results, and modes of statement of analysis of the materials connected with the agricultural industry." They report upon the chemical composition and physical properties of cereals, the principles and methods of soil analyses, and the capabilities of such soils, methods of analyses of commercial fertilisers, cattle foods, and dairy products, sugar and fermented liquors, fruit juices, alcoholic beverages, &c., and also inquire into the extent and character of food adulterations, &c.

As an example of the work done in one of the States alone, Georgia, under the charge of Dr. Geo. F. Payne, State Chemist, 1,375 official analyses were made during the season 1893-4, comprising 723 brands of fertilisers sold in the territory, and representing an output of 315,612 00 tons, the increase being very considerable since the organisation of the department in 1874-5, when 110 samples were analysed, representing an output of 48,648 00 tons. The bulletins published in these departments are of great value and interest.

**Glycerin.**—In 1848 the output of glycerin manufactured in America was 15 lb., which was manufactured by Shoemaker from lead plaster; in 1850 the output had gone up to 150 lb.; in 1865 the value of nitro-glycerin as a bleaching agent became recognised, and in 1867 it was largely employed in the manufacture of dynamite.

The glycerin used for this purpose is obtained from the candle factories and soap lyes, and the estimated quantity thus obtained is 8,000,000 lb. per annum.

The demand for refined glycerin, however, increased so fast, that in 1874 there was not sufficient to meet the demand, so that supplies of crude material suitable for refining had to be imported from Europe, and in that year 576,574 lb. of crude glycerin was imported, and the demand has increased so rapidly that, in 1893, 16,440,213 lb. were imported, all used in the manufacture of refined glycerin.

**Potash.**—The manufacture of potash and pearlash has been carried on for a long time by the leaching of wood ashes, the only source of these salts in the United States. The production has decreased materially owing to the

scarcity of wood, and although there is still a demand for it, it has to a great extent been replaced by soda.

*Bichromate of Potash*, was one of the first chemical salts manufactured in this country owing to the discovery of extensive chrome ore deposits in Maryland, from which source the greater part was obtained until the discovery of ore in California. The production in 1880 was 4,000 lb.

*Prussiate of Potash* has been manufactured for 50 years. In 1843, 69,470 lb.; in 1893, 646,226 lb. of red prussiate was manufactured, the raw material being crude carbonate of potash, produced in this country from wood ashes and waste, horns, leather, scraps, &c.

*Soda*.—The production of soda according to the census report of 1880 was 40,259,938 pounds or about 10 per cent. of the quantity consumed. The total production, according to the eleventh census report, 1890, was 333,124,357 pounds. The Solvay Process Co., Syracuse, N.Y., manufacture soda ash by the Solvay or ammonia process. In addition to soda ash they produce caustic soda and bi-carbonate of soda, and recently a more concentrated form of soda crystals. They have also lately put upon the market a crown filler, for paper-makers' use, for which the demand is rapidly increasing. Their output of soda-ash represents 100,000 tons this year, of which they will probably consume one half in their own works. The total quantity of soda ash produced in this country would not exceed 67,000 tons. The quantity of bicarbonate of soda produced in 1890 is estimated at 40,000 tons; the present production is estimated at between 50,000 and 60,000 tons. The salt used by the Solvay Process Co., is obtained from the rock salt deposits just south of Syracuse, and their coal from the Pennsylvania coal-fields. The Pennsylvania Salt Manufacturing Co., Natrona, Pa., manufacture sal soda, bicarbonate of soda, caustic soda, refined sulphate of soda, sulphuric acid, sulphate of copper, caustic potash, alum, copperas, chloride of calcium, nitric and hydrochloric acids, common salt, potassium sulphate, sulphate of alumina, &c. They are also large importers of cryolite. Natrona is reported to be the only factory in the United States which utilises all its materials in the most rational manner.

*Electrolytic Alkali*.—Should success follow the working of the "Castner" process to electrolyse sodium chloride for the preparation of caustic soda and chlorine, it is probable that the manufacture will be carried on an extensive scale in this country. So far electrolytic processes have not proved a commercial success.

### *The Power of Niagara.*

As power is an important factor in manufactures, especially since the electrolytic methods are coming into use, the idea of the Cataract Construction Company, through one of its subordinate organisations, Niagara Falls Power Company, for generating cheap power will undoubtedly develop many lines of work in the future, and may be of interest.

The water surface of the Great Lakes (except Lake Ontario) is 84,000 square miles. Their water-shed covers 240,000 square miles, or more than twice the area of Great Britain and Ireland. Length of shore lines, 5,000 miles; volume of water, 6,000 cubic miles, of which Lake Superior contains a little less than one half; and rate of out-flow at Buffalo, 217,000 to 275,000 cubic feet per second. Niagara Falls are 23 miles below Lake Erie, and 14½ miles above Lake Ontario. The drop is 165 ft., and the depth of water just below the falls, 189 ft. The volume of water in the lakes is such that it has been estimated that even if no rain fell, the flow of the river would be continuous at its present rate for 100 years if the lakes could be gradually drained. Such is the never failing reservoir from which the Niagara Falls Power Company proposes to draw its supply. The company has acquired 1,600 acres of land stretching to a point five miles easterly and three miles northerly from the Falls. Of this 1,071 acres are intended to be devoted for mill sites for manufactories.

Total water power of the Falls is 7,000,000 horse-power, of which it is proposed to use 125,000 horse-power.

*The Canal*.—The main canal is protected against floating ice by having its entrance directed down stream, and by a floating boom fastened to the iron piers at its mouth. The canal is 1,300 ft. long, 100 to 130 ft. wide, and the depth of water will average 12 ft. The greatest velocity in the canal will be 4.17 ft. per second when a sufficient amount is being fed to the tributaries to produce 100,000 horse-power. From this inlet canal the water is led through steel penstocks to the turbines and is discharged into a channel at the bottom of the wheel pit, from which it flows into the main tunnel and is returned to the Niagara River at the portal just below the upper Suspension Bridge. The wheel-pit (cut into the solid rock) is 178 feet deep, 21 feet wide, and at present 140 feet long. When it is continued to its full length as planned it will provide for 10 inlets and be about 400 feet long. The tunnel is lined throughout with vitrified brick, a total of over 13,000,000 being used in the construction. Three turbines of 5,000 horse-power each are already in position, and the dynamos are nearly ready to be placed. The tunnel is 7,250 feet long, 14 to 18 feet wide, and 21 feet high. The grade is 36 feet to the mile, and the tunnel passes 200 feet under the city of Niagara Falls.

The work of construction was commenced by Rogers and Clement of New York, the contractors, October 4th, 1890.

The company has entered into contracts with the Pittsburg Reduction Co., to secure 6,000 electrical horse-power for the manufacture of aluminium, and more recently a contract for 10,000 electrical horse-power has been entered into with Carborundum Co. for the manufacture of the new abrasive material—carborundum. The hydraulic power is already applied to the works of the Niagara Falls Paper Co., who grind between 40 and 50 tons of wood pulp per day, but the mechanical and electrical equipments of the plant are not yet in readiness for distribution of power. The works of the Pittsburg Reduction Co. are, however, nearly completed, and the foundation is now being laid for the Carborundum works.

*Other manufactures*.—There are 13 establishments in the United States, manufacturing bone black, animal charcoal, and ivory black, with an invested capital of 6,000,000 dol.

It is not to be expected that I could complete the list of chemical industries in this address, but I simply give some idea of what is being done in certain directions. Such manufactures as starch, sugar, glucose, dextrine, alcohol, &c., are produced in enormous quantities in several factories. Soaps of all kinds are manufactured largely for various uses, domestic and technical, and also exported in large quantities. Some of the factories are equipped with more modern appliances than any in the world. Other industries are the distillation of essential oils and the manufacture of synthetical odours, dyes, paints, colours, varnishes, cotton seed oil, linseed oil, glue, gelatin, and even the rare earths, lanthanum, neodymium, praseodymium, zirconium, cerium, and thorium (by the Welsbach Chemical Co., Gloucester, New York), the manufacture of ceramics, building bricks, pottery, terra cotta, &c., for which we have a high reputation.

### GLASS-MAKING INDUSTRY.

In the "Engineering Magazine," March, 1893, there is a most interesting and historical article by Mr. R. M. Atwater, on the glass-making industry in America. The manufacture of glass bottles, chemical ware, window glass, polished plate glass, pressed glass and coloured glass, is described. He writes:—"The flint glass business now centres in the United States Glass Co. of Pittsburg for table ware, and the works of Whithal Tatum and Co. of New Jersey for druggists, and chemists' ware. To the same department belongs pressed glass; and although a very old process, to American glass makers is due its practical and enormous development during the last 20 years. The glass press is a finely designed machine, and by it the cost of table ware is greatly reduced and the variety of shapes infinitely increased. The moulds that produce these shapes are of great cost and variety. The art has been copied by the foreigners and the best forms of the American moulds and presses are to be found in England and Germany, and their products are a close imitation of our best lines."

The making of chemical glassware in America was a direct result of the Philadelphia Exhibition of 1876. This glass is a triple silicate of potash, soda and lime, and also contains alumina. This last gives it a dull appearance, but adds greatly to its refractory character. Up to this time, the making of chemical glassware in America has not attained the high Bohemian or German standard, though America is probably the best market in the world for chemical glassware. In the development of low cost, in making metallic aluminium, it is possible that a glass may be produced that will have the flexible character of mica with the refractory nature of the best Bohemian glass. The making of polished plate glass in America was first successfully established by Captain John B. Ford, of the Pittsburgh Plate Glass Co. His early work resulted in failure and a considerable loss of fortune, but not daunted in 1883, he put new works in operation at Creighton, Pa., which were successful from the start. The fortunate discovery of natural gas in the vicinity of the works led to a profitable and growing business, which now forms the largest plate glass works in America and produces nearly half of the total American product. Their capacity is 340 pots, producing 200 sq. ft. of polished plate glass daily. The quality is fully equal to the French standard, and foreign plate glass is now practically out of the American market.

Mr. Thomas Gaffield, of Boston, discovered the first effect of sunlight on the colour of window glass. It is, perhaps, the only American contribution to the physics of glass that has taken rank with the best European work. Mr. Gaffield based his conclusions on the results of experiments carried over a period of five years, the practical result of which was to demonstrate that the only way to obtain permanent colourless window glass was to make it from the purest materials.

Before closing, I wish to call your attention to the subject of:—

**Alcohol.**—Alcohol is the chief raw material used in the manufacture of expensive chemicals, and is taxed at about ten times its cost of production. If that part of it which is actually decomposed in the manufacturing processes were exempted from taxation, this nation would make from it chloroform, ether, &c., at as low a cost as other nations, and their use as solvents would be as much increased as that of alcohol itself. To the manufacturing chemist alcohol is as essential as iron is to other branches of industry. Untaxed alcohol would sell at from 26 to 30 cents per gallon. At present it sells at about \$2.00 per gallon. The quantity of alcohol consumed for medical and manufacturing purposes and in the arts is very large, and, as a consequence, the tax paid is enormous.

This condition of affairs may appear strange in a country where there is a law which prescribes that:

"Any manufacturer finding it necessary to use alcohol in the arts, or in any medical or other like compound, may use the same under regulations to be presented by the Secretary of the Treasury, &c., and shall be entitled to receive from the Secretary of the Treasury of the United States a rebate of tax so paid."

The fact that the Secretary of the Treasury has declined to formulate the necessary regulations before Congress meets, and that there is a general feeling that when Congress does meet the law will be annulled, is my reason for bringing this subject before your notice. I suggest, for your consideration, whether we as manufacturers can, by any united action, take means to enforce the fulfilment of the law or any modification of it such as the duty of 50 per cent. per gallon, and by such a compromise satisfy the Secretary of Treasury.

Professor H. L. Wilson, member of Congress for West Virginia, in addressing the London Chamber of Commerce in August last, said: "The manufacturing supremacy of the world must eventually pass to that people and that country which has the largest basic materials of industry and the readiest and cheapest access to them, and which brings to their development the highest intelligence, the freshest results of art, science, and invention, and the best business methods to their production and distribution. It is because we are more and more confident as each year passes in the exhaustless supply of those materials, in our

cheapness of access to them and our ease of transporting them to the markets, and in our growing control of those labour-saving appliances which the world to-day is inventing and seeking to use, that we believe that the supremacy must some day or other pass to the United States of America."

As far as chemical industries are concerned, before this can be accomplished something has to be done. Quite recently a serious depression has been hanging over the country and commerce has been almost at a standstill, caused by the want of confidence in the stability of the laws, newspaper exaggeration of the instabilities, and above all the influence of partisan politics in congress. We want cheaper real estate, cheaper cost of plant, free coal, and free alcohol, as we stand to-day; or we want absolute protection for every existing industry, every man on equal footing, no trusts, and such protection guaranteed for a series of years (say ten years) sure, so that ample provision can be made for the requirements of the people by home manufactures and industries. If this should fail, then free trade and fair trade.

America in competition with the world could hold her own and more, first providing for domestic consumption at reasonable prices and then for foreign requirements; for as the population of the world increases, the increased demand for manufactured products will follow. Nature herself provides advantageous resources in every country for the development of natural industries, and by the free exchanges of commodities all should be benefited and the common brotherhood of mankind cemented.

I am indebted to Drs. F. P. Van Dunberg, Geo. F. Payne, W. P. Sadtler, Hugo Schweitzer, and to Messrs. S. A. Knapp, Seward W. Williams, W. M. J. Gordon, F. Schoellkopf, Chas. Pfizer, and others. Also to the census officials at Washington, United States Geological Survey, statistics compiled from the mineral resources of the United States, 1893, revenue returns, 1893, and to the Journal of the Society of Chemical Industry, the Chemist and Druggist, the Oil, Paint, and Drug Reporter, Scientific American, Engineer's Magazine, &c., and I desire to acknowledge the extreme courtesy with which my enquiries have been met.

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*Meeting held Monday, December 6th, 1894.*

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MR. A. H. MASON IN THE CHAIR.

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Mr. Ungerer read a paper on "Modern Perfumery," in which he mentioned the various organic substances used in this branch of chemical industry.

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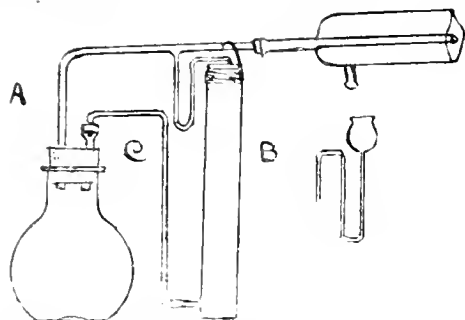
## AN APPARATUS FOR EXTRACTION.

BY CHAS. E. PARKER.

THE method of the immiscible solvents for the separation of alkaloids and other substances having proved itself a useful expedient for the purposes of proximate organic analysis, particularly pharmaceutical assaying, the frequent attention required in using the separatory funnel has created a demand for automatic apparatus for accomplishing otherwise the same result.

The contrivances of Von Ledden, Heulseboese, and Smetham, are recent inventions for the extraction of aqueous solutions with ether.

Analogous constructions to facilitate extraction with chloroform seem to have attracted less attention, though they are mentioned in Prescott's organic analysis and probably elsewhere.



The writer lays claim therefore to merely a convenient construction for applying well-known principles.

The (aqueous) solution to be extracted is floated in the extraction tube B, upon a sufficient quantity of chloroform to rise well above the side tube C at the bottom.

The flask, containing a small quantity of chloroform, being heated on the water or steam bath, the vapour rises through the bent tube A to the condenser and is returned in the liquid state through the trap tube, the solution in B, and outlet tube C, which is connected with the flask by a larger tube and a corked joint. The siphoning out of the contents of B is thus prevented. By shutting off the heat and opening this joint, a sample can readily be drawn for ascertaining the progress of the extraction.

In case an open joint at this point is such a desideratum as to counterbalance the objection to loss of solvent by evaporation, a funnel tube bent so as to form a trap may be substituted for the straight tube into which C discharges. The escape of hot vapour from the flask being thus prevented, the end of C is merely strung in position over the enlarged end of the funnel tube.

The tube B, being made from a screw-capped tubular bottle, the writer utilised the cap for its suspension by cutting away the end and attaching a copper wire hook to the threaded collar remaining.

The ascending limb of A should be jacketed with felt to prevent condensation.

## A NEW REACTION FOR THE DETECTION OF SOAP IN LUBRICANTS.

BY H. SCHWEITZER AND E. LUNGWITZ.

THE analytical chemist is very often called upon to determine the presence of soap in fats, oils, &c. Very frequently lubricants, such as mineral oils and tallow, are met in commerce to which soap has been added for the purpose of adulteration. The specifications accordingly of all the leading railroads of the United States distinctly point out that the lubricants must be free from soap. Mineral oils very often contain small quantities of soap, especially aluminium soap, which are added to make the mineral oil appear thicker and more consistent. Since the use of such adulterated products for lubrication has decided disadvantages, it is essential to have a sharp reaction by which the presence of soap, even in the minutest quantities, can be qualitatively established beyond any doubt. The methods which are at present used for this purpose do not give satisfactory results.

Our reaction is based on the fact that metaphosphoric acid is soluble in absolute alcohol or in sulphuric ether, whereas the metaphosphoric acid salts of the alkalis and alkaline earths are insoluble in these liquids. We

must add, however, that the metaphosphoric acid salt of ammonia is soluble in alcohol or sulphuric ether. But since the products to be examined are mainly lubricants which contain potash-, soda-, magnesia-, or alumina soaps, but never ammonia soap, this exception is of no importance for our new reaction. In doubtful cases, however, it will be required to test for ammonia after the well-known methods.

After these introductory remarks it is obvious that if we treat a solution of any fat, oil, or wax in sulphuric ether, benzene, or absolute alcohol with a saturated solution of metaphosphoric acid in absolute alcohol or sulphuric ether, we decompose any kind of soap present and precipitate the metaphosphoric salts from this solution.

The reaction is exceedingly sharp and absolutely reliable. But it must be remembered that some waxes and ozokerite are precipitated from their solutions in benzene by means of alcohol. In such cases we must use both the reagent and the fat in ethereal solution.

We have found that no substances which are present in coal-liver oil, fish oil, sperm oil, lanoline, except the fatty matter, give precipitates with metaphosphoric acid, and thus do not impair the value of our reaction.

To determine the accuracy of our test, we dissolved 5 drops of the substances enumerated below in 5 cc. of benzene (boiling point below 75° C.), and treated this solution with 1 cc. of a saturated solution of metaphosphoric acid in absolute alcohol. We prepare the latter reagent by shaking pulverised metaphosphoric acid with alcohol. After allowing to stand for some time, the clear liquid, representing the new reagent, is drawn off.

Simultaneously we carried out comparative tests under the same conditions with the same substances, to which we, however, added 1 cc. of a solution containing 1 gm. gelatin in 100 cc. benzene. As is well known, gelatin is the commercial article consisting of mineral oil in which 10 per cent. of alumina soap are dissolved.

The following fats and oils gave, even after 24 hours' standing, no trace of a precipitate:—

(1.) *Oils of Mineral Origin.*—Petroleum, paraffin oil, vaseline, ozokerite.

(2.) *Oils of Vegetable Origin.*—Cotton-seed oil, rapeseed oil, linseed oil, olive oil, sesame oil, peanut oil, castor oil, palm oil, Chinese tallow.

(3.) *Oils of Animal Origin.*—Lard, lard oil, tallow, stearin, oleic acid, stearic acid, neatsfoot oil, lanoline, herring's oil, fish oil, cod-liver oil, sperm oil, whale oil.

(4.) *Essential Oils, Wax.*—Rosin oil, camphor oil, spirits of turpentine, beeswax, spermaceti.

The mixtures of oil and soap, prepared as above, gave all of them characteristic precipitates.

At the same time, our reaction permits an approximate estimation of the nature of the soap present. We add to the alcoholic ethereal solution containing the precipitate of the phosphate, a solution of chloride of platinum in alcohol. In case the flocculent precipitate becomes crystalline, the presence of potassium is indicated. If the original precipitate is dissolved, then sodium, calcium, or magnesium, the platinum double salts of which are soluble in alcohol, may be present. If the original precipitate remains unchanged, aluminium or iron salts are present.

## DISCUSSION.

Dr. J. H. WAINWRIGHT asked Dr. Schweitzer what were the disadvantages of lubricants containing soap.

Mr. R. C. WOODCOCK said that if they contained a little potash or soda soap, probably that soap would contain a little free alkali. Might not that rather be an advantage than otherwise? The free fatty acids frequently met with in lubricants attacked copper bearings and zinc.

Dr. H. ENDEMANN said that a lubricant, which the vendor was anxious to introduce on the railroads, had some time ago come under his notice, which proved to be an alkaline soap in admixture with other lubricants, the soap therein being about 20 per cent. The lubricant was tried indoors, but in order to be useful had to be weighted down so as to

get it in contact with the shaft and its bearings. It was found that while the lubricant was quite good under conditions of a dry and not too low a temperature, in cold weather the soap would make the lubricant unmanageable, inasmuch as it could not be forced into the bearings. Moisture also gave rise to the formation of an emulsion. The railroads were now alive to the disadvantages of lubricants the efficiency of which was affected by moisture and low temperatures.

Mr. H. COMER asked what lubricants were in the market or used which contained soap.

Mr. VICTOR BLOEDE thought that Dr. Schweitzer did not quite do the soap question justice, in that he spoke of it purely as an adulterant. The presence of soap was a very great advantage to certain classes of lubricant oil. It increased the viscosity and enabled a higher degree of lubrication without any detriment resulting. Dr. Endemann had said that it would wash away. That applied more particularly to the true alkaline soaps, but not to alumina soap. While an excess of soap, and especially of alkaline soap, was undoubtedly added purely as an adulterant in many cases, and added no advantageous property to the lubricant at all, except to enable it to absorb water, yet that did not apply to soaps which were soluble in mineral oil. Through practical experience and many tests that he had made, he believed the viscosity and power of adherence to metal surfaces to be much increased.

Dr. H. ENDEMANN said that he had intended his remarks particularly to apply to oil to which an alkaline soap had been added.

Dr. H. SCHWEITZER said, in reply, that what Dr. Endemann had said against lubricants containing alkaline soap was confirmed by his own experience. That lubricant which consisted of paraffin oil and contained 10 per cent of alumina soap, to which Mr. Bloede had referred in his remarks, was certainly an adulterated product and was treated as such by R. Benedikt and others. Experience had shown that its use for lubrication was very disadvantageous. Firstly, the alumina soap was precipitated when it came in contact with water or steam, and the thus formed precipitate clogged the machinery. Another disadvantage was the fact that the friction was increased. Grossmann reported an instance in which the owner of a mill using water-power decided to use a consistent fat as lubricant instead of pure oil. In consequence thereof the water-power became insufficient for driving his machinery, and the mill-owner was forced to readopt his old method of lubrication.

J. E. WOODBURY, in Boston, mentioned that he used for one of the two rows of looms in a cotton-mill, under the same conditions, oil, and for the other a consistent fat. He found that that the temperature of the bearings lubricated with oil was only 4° higher than the temperature of the room, whereas the temperature on the other side was 22° higher.

An increase in friction meant an increase in the amount of fuel used, and greater wear and tear of the machinery. Granted that the viscosity of oils containing soap was much larger, and that the use of such lubricants was convenient and economical, nevertheless experience and the published literature showed that these advantages were not considered great enough to induce people to use the soap lubricants instead of pure oils.

In answer to Mr. Comer's question, there were only three kinds of lubricants with soap in the market: gelatin (paraffin and alumina soap), hot-box greases, and axle greases. The latter two products were mixtures of mineral oils with lime-soaps from vegetable or animal oils or resin soaps, at the same time frequently containing so-called fillers, such as talc, chalk, and sulphate of barytes.

## Journal and Patent\* Literature.

Class.	Page
I.—General Plant, Apparatus, and Machinery .....	1179
II.—Fuel, Gas, and Light .....	1182
III.—Destructive Distillation, Tar Products, &c. ....	1182
IV.—Colouring Matters and Dyes .....	1188
V.—Textiles: Cotton, Wool, Silk, &c. ....	1192
VI.—Dyeing, Calico Printing, Paper Staining, and Bleaching .....	1193
VII.—Acids, Alkalis, and Salts .....	1195
VIII.—Glass, Pottery, and Enamels .....	1197
IX.—Building Materials, Clays, Mortars and Cements, &c. ....	1198
X.—Metallurgy .....	1198
XI.—Electro-Chemistry and Electro-Metallurgy .....	1203
XII.—Fats, Oils, and Soap Manufacture .....	1207
XIII.—Pigments and Paints; Resins, Varnishes, &c.; India-Rubber, &c. ....	1209
XIV.—Tanning, Leather, Glue, and Size .....	1209
XV.—Manures, &c. ....	1210
XVI.—Sugar, Starch, Gum, &c. ....	1210
XVII.—Brewing, Wines, Spirits, &c. ....	1211
XVIII.—Chemistry of Foods; Sanitary Chemistry and Water Purification; Disinfectants .....	1215
XIX.—Paper, Pasteboard, &c. ....	1216
XX.—Fine Chemicals, Alkaloids, Essences, and Extracts .....	1216
XXI.—Photographic Materials and Processes .....	1219
XXII.—Explosives, Matches, &c. ....	1219
XXIII.—Analytical Chemistry .....	1220

## I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

### PATENTS.

*Improvements in Apparatus for Automatically Regulating the Temperature of Fluids Heated by Steam.* J. J. Royle, Manchester. Eng. Pat. 19,116, October 16, 1893.

THE invention relates to improved means for regulating the steam supply to boiling pans, storage tanks, and other steam-heated apparatus, and consists of a flexible bent metal bar having its ends fixed rigidly to convenient parts of the steam-heated apparatus, and with the centre bearing upon the stem of the balanced steam-supply valve. As the temperature rises the casing of the apparatus expands and pulls the bowed bar straight, and thereby partially closes the valve and diminishes the supply; in this way an automatic regulation of the steam supply is kept up.

—E. G. C.

*Improvements in and relating to Evaporating Apparatus.* Paul Donat de la Grée, Paris. Eng. Pat. 21,000, November 4, 1893.

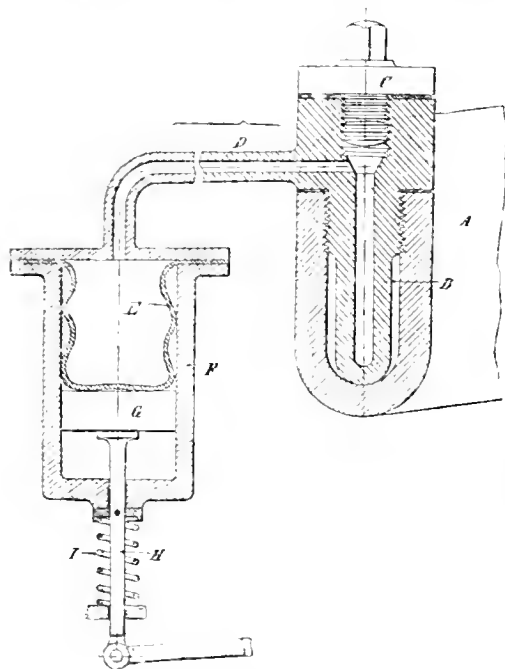
THE improved apparatus is for instantaneously evaporating water, and is designed to obviate the inconvenience caused by the evaporating coil becoming filled with water when the heating is not sufficient to completely evaporate the water as fast as it enters. The invention consists of a mechanical device by means of which the variations of temperature of the evaporating coil may be caused to regulate the movements of the valve controlling the feed. The device is shown in section in the figure.

It is a tubular chamber of small internal capacity, which is fixed within the evaporating pipe or coil at some point not directly in contact with the flame of the furnace. This chamber communicates, by a side tube D, with a flexible chamber E, contained within a cylinder F, which actuates,

\* Any of these specifications may be obtained by post by remitting 8d.—the price now fixed for all specifications, postage included—to Sir Henry Reade Lack, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.



in opposition to the spring 1, the piston G and rod H. The chambers B and E, and the tube D, are first filled with water, and then closed by screwing in the plug C. When the coil is heated, the steam within it heats the chamber B, and converts the water in the chamber partly into steam, which expands the chamber E and forces down the piston G; but if the temperature of the coil should fall too low, the steam in B will condense, and the piston G will be returned by the tension of the spring 1. By suitable connections,



ATTACHMENT FOR INSTANTANEOUS EVAPORATING APPARATUS.

the upward and downward movements of the piston-rod H may be made to close or open the valve which controls the admission of water to the coil. As, in practice, the apparatus will be adjusted so as not to completely close the valve until actual water has begun to accumulate in the coil, a relief valve is provided, in order that the pressure of the steam produced by the evaporation of this water in the closed coil, when the temperature again rises, may not become great enough to be dangerous.—L. A.

*Improvements in Mechanism for Controlling and Regulating the Discharge of Liquids from Tanks.* W. G. Strype, Dublin. Eng. Pat. 22,529, November 24, 1893.

THE inventor claims improvements in the mechanism for regulating the discharge of liquids from sewagetanks. The bottom of the tank has angular gutters, at the ends of which are sluices opening into a transverse gutter provided with a penstock valve. The shaft opening the sluices and valves, bears cams so arranged as to open every third or fourth sluice only, in addition to the penstock valve, three or four times every revolution, thus concentrating the scouring effect of the outflow upon some of the gutters. A second transverse gutter connects the further ends of the angular gutters so that liquid passes into those of the latter, which are open, from the others. A horizontal cam-shaft, turned by hand or otherwise, driving two worms and worm wheels in connection with both valve and sluices, supplies the motive power.—L. de K.

*A Means or Device for Protecting Steel Cylinders for Containing Compressed Gases from Shocks or Injury during Transit or otherwise.* W. E. Lea, Runcorn, Cheshire. Eng. Pat. 23,616, December 8, 1893.

THE patentee claims in this invention the "combination of steel cylinders or bottles used for containing compressed or liquefied or similar substance with an external protective covering of rope or other suitable material," the strands of which are wrapped round and encase the cylinder or bottle. Other suitable soft and twisted, or plaited material may be used, the object being to minimise risks of injury following a fall, blow, or shock, received by such cylinder or bottle.

—W. S.

*Improvements in Means for Utilising the Pressure in Cylinders or Reservoirs containing Compressed Gases, in the Application and Use of such Gases.* The Manchester Oxygen (Brin's Patent) Co. Lim., and W. M. Jackson, Manchester. Eng. Pat. 24,761, December 23, 1893.

FIG. 1 is a sectional elevation, and Fig. 2 a sectional plan of the apparatus, consisting of a casing *a* having an oxygen

Fig. 1.

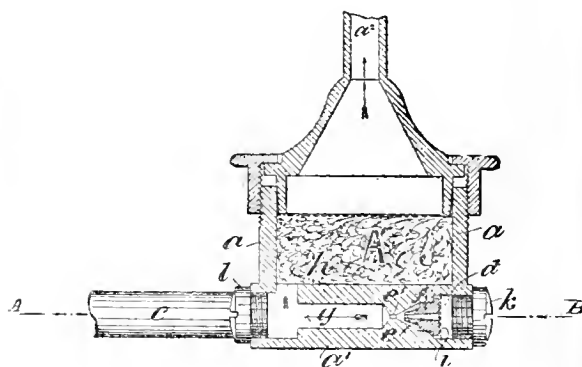
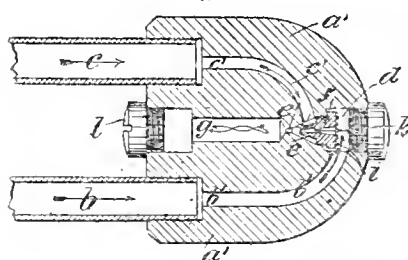


Fig. 2.



MIXER FOR OXYHYDROGEN BURNER.

gas-inlet tube *b*, and a similar one *c* for coal gas. The oxygen gas under pressure enters the passage *b* and passes to the back of the mixing cone *f* of an injector, whereby a current of coal-gas is drawn into the chamber *e* and mixes with the oxygen as it passes through the orifice *e* into the chamber *g*. The mixed gases afterwards pass by the orifice *h* into a chamber *A* containing a sound-deadening composition, and finally escapes by the outlet *a*. Suitable plugs, *k* and *l*, are fixed in the casing *a* to allow of access for repairs and cleaning, and wire-gauze discs are placed at the back of the mixing cone and in the sound-deadening chamber *A* in order to prevent the passages being blocked by foreign matter.—E. G. C.

*Improvements in the Construction of Filtering Beds.*

T. Cosham, Newark-on-Trent. Eng. Pat. 9519, May 15, 1894.

THE combination is claimed, in filtering beds, of two or more tanks enclosed in suitable walls, and having perforated floors over "flocculent chambers" so arranged that the sewage or other matters to be operated upon and filtered, is caused to pass alternately downwards and upwards, through filtering material.

The chief claim of the inventor is, however, that in the reversal of the order of the flow by means of pumping appliances, the whole of the arrested matters may be entirely removed from the tanks without interfering with the filtering materials.—L. de K.

*Improved Refrigerating Liquid.* Martin Wanner, Yorktown, Delaware, U.S.A. Eng. Pat. 13,194, July 7, 1894.

THE inventor's object is to prevent or retard the thickening or freezing of volatile liquids, such as carbon bisulphide, used in refrigerating machines, and to regulate their rapidity of evaporation. The inventor points out that by the rapid evaporation of carbon bisulphide, for example, the liquid falls in temperature more rapidly than the walls of the containing vessel, until it at first thickens and then freezes. Thus the efficiency of the apparatus is impaired. To prevent this thickening or freezing, he mixes with the volatile liquid "oils or other liquids or fluids of as nearly as may be the same specific gravity as itself, being perfectly miscible therewith."—L. A.

*Improvements in and relating to Apparatus for Heating Liquids, Air, and other Fluids.* E. Fudicker, Elberfeld, Germany. Eng. Pat. 15,413, August 13, 1894.

IN order to more efficiently heat liquids and gases, and more particularly water for baths, a form of geyser is employed in which the liquid or gas is introduced into a central vertical pipe, provided with a rose at the top, for distributing the fluid in finely-divided streams over a corrugated and perforated spiral encircling the central tube. As the liquid flows down the spiral it meets with the hot gases from a gas burner, placed at the base of the apparatus, and thereby becomes heated: the heated fluid escapes by a suitable outlet in the base of the apparatus.—E. G. C.

*Improvements in Apparatus for Treating Powdered Materials.* J. S. Detwiler, Philadelphia, U.S.A. Eng. Pat. 17,314, September 11, 1894.

THIS invention describes an apparatus for breaking up the lumps and for drying the powdered material. It consists of a cylindrical vessel, divided into a number of superposed compartments having bottoms formed of wire gauze of a degree of fineness increasing from the top compartment downwards. In each chamber there are a number of brushes fixed to frames carried by a central vertical shaft, by the rotation of which the brushes are made to revolve and sweep the gauze bottoms of the chambers. A current of air is, at the same time, driven by a fan into the lowest compartment, and passes upwards, through the gauze bottoms, from chamber to chamber, leaving the uppermost compartment through screened openings. The material, which is fed continuously, or in small quantities at intervals, into the uppermost compartment, being dried by the current of air which rises through it, and swept downwards through the gauze partitions by the revolving brushes, gradually falls by gravity into the conical bottom of the apparatus, whence it is drawn off from time to time.—L. A.

*Improvements in Apparatus for Blending Powdered Materials.* J. S. Detwiler, Philadelphia, U.S.A. Eng. Pat. 17,315, September 11, 1894.

IN this specification three forms of apparatus are described. The first consists of a vertical cylindrical chamber, having a contracted top and outlet, and a series of inlets for air arranged around the bottom. In the centre of the cylinder

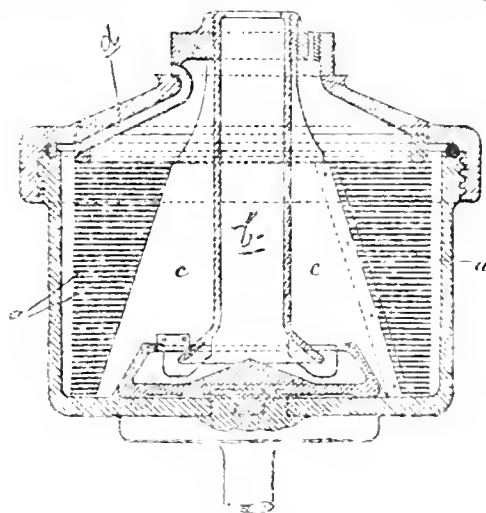
there is a vertical shaft, to which are fixed, at different heights, a number of fan blades and fingers capable of being revolved by the rotation of the shaft. The powdered material or materials which it is desired to segregate, intimately mix, and dry, are introduced into the apparatus at the bottom by means of a pipe containing a screw conveyor; the powder is then lifted by the current of air passing in at the air holes, and carried upwards among the revolving beaters, which break up the lumps and mix the material, and is finally forced out through the contracted top, and through a pipe into the settling chamber. The latter has the shape of a horizontal cylinder, and contains a screw conveyor, fixed along the bottom, which continually collects the settled powder and scrapes it towards a hole, through which it falls into casks placed to receive it; the air escapes through screened openings in the upper side of the cylinder.

The second form of apparatus is a revolving horizontal drum, having fixed blades on the inside and hollow end journals. The materials to be blended are fed into the hollow journal at one end, and are forced—by a blast of air which passes through the journal, into the drum, and out through the other end journal—into a settling chamber of the construction already described.

The third form of apparatus consists of a vertical fixed cylindrical box or chamber with flat top and bottom. The upper part of this chamber contains a vertical shaft, carrying a number of arms, which revolve between arms fixed to the walls. About half-way up, and below the revolving arms a blast of air is admitted into the chamber, and escapes through screened openings in the top. The materials, which are fed through the top of this apparatus, fall among the beaters which break up lumps and mix "and blend" the particles, by the aid of the rising current of air which also carries away moisture. The powdered mixed materials which have reached the bottom of the chamber, are drawn out thence by means of a pipe, and screw conveyor.—L. A.

*Improvements in Centrifugal Separators.* O. J. R. Ström, Stockholm, Sweden. Eng. Pat. 17,552, September 18, 1894.

IN order to effect a flow of the heavier liquid outwards in a centrifugal separating apparatus for milk and the like, a number of discs *a*, arranged in a pile within the casing of the separator in the manner shown, are corrugated in spiral



CENTRIFUGAL SEPARATOR.

lines radiating from the centre and provided with annular holes so as to form an annular chamber *c* round the central inlet pipe *b*. The liquid is poured into the central pipe *b*, and the heavier portions thereof, after penetrating through the passages formed by the plates *a*, are collected by the pipe *d*.—E. G. C.



*An Improved Method of and Apparatus for Drying Substances which are Viscous or Semi-fluid in the Hot State.* H. H. Lake, London. From R. Schicht, Bohemia. Eng. Pat. 18,214, September 25, 1894.

This invention is for a method of drying viscous or semi-fluid substances, which are liable to burn on the heating surfaces, when dried in the ordinary way. The improved method consists in heating the material, whilst under such great pressure that it cannot boil, to such a temperature that when it is allowed to flow into a vacuum vessel, the contained heat is sufficient to evaporate all the water in it. The apparatus consists of a high-pressure vessel, a heating vessel, and a low-pressure vessel, connected by pipes and valves. The material is introduced first into the high-pressure vessel, and hot air is forced in until the requisite pressure is attained; connection with the heating vessel is then opened, and also with the low-pressure vessel: the fluid then passes through a number of tubes, in the heating vessel, which are heated by steam on the outside, and is thereby raised to the necessary temperature, and it afterwards flows into the low-pressure vessel, where evaporation takes place. The latter vessel is furnished with baffle plates for distributing the fluid as it falls into the vessel, and also with steam pipes for supplying additional heat if required.—L. A.

*An Improved Method of Exhausting, and Apparatus Employed therefor.* S. H. Wright, Ganarew Manor House, County of Hereford. Eng. Pat. 19,654, October 19, 1894.

In order to create a vacuum or cause a current of air to flow through any vessel, it is connected by a side channel to a vertical downfall pipe through which water is flowing in a number of divided streams, thereby inducing a current of air. The water is divided up into streams by means of perforated plates inserted at suitable intervals in the downfall pipe.—E. G. C.

## II.—FUEL, GAS, AND LIGHT.

*The Explosive Nature of Acetylene.* L. Meyer. Ber. 1894, 27, 2764—2765.

As a general rule, mixtures of combustible gases, such as hydrogen, carbon monoxide, methane, &c., with just sufficient or a slight excess of oxygen, may be exploded without risk in an open glass cylinder, providing the latter is of uniform diameter and not constricted at the neck. Acetylene, however, forms an exception. In an experiment with this gas, the containing cylinder was shattered, even the thick foot of the jar being blown into a number of pieces. The author therefore points out the necessity for caution in dealing with explosive mixtures containing acetylene.—H. T. P.

*Experiments on Coals immersed in Water.* G. Arth. Bull. Soc. Chim. 1894, 11, 619—622.

VARIOUS kinds of coal were (a) Exposed freely to the air, (b) Immersed in water, both running and stagnant, for twelve months, and the changes produced in their composition and heat of combustion determined. Three kinds of coal were used: (1) From the Frankenholz mine in Rhenish Bavaria; (2) From Brocourt (Pas de Calais), and (3) From the mines of Aiseau-Prêle (Charleroi). The coals were broken up and sieved, the pieces being used which passed through meshes 10 mm. square, but not through meshes of 3 mm. square. Care was taken to obtain even samples. The measurements show that exposure to air or immersion in water for the length of time indicated, produces changes in composition and heat of combustion which are so small that for practical purposes they may be neglected.—T. E.

*Examination of the Products of Combustion of Coal-Gas, when burned by Argand and by Auer Burners respectively.* N. Gréhaud. Comptes rend. 1894, 119, 146.

See under XVIII. B., page 1215.

## PATENTS.

*Method of Manufacturing Artificial Fuel.* E. Kirbis, Berlin. Eng. Pat. 17,484, September 14, 1894.

THE object of this patent is to produce a fuel (for use in stoves, without stacks, &c.) which may be very easily kindled or caused to glow, and that continues glowing slowly and uniformly without evolving any smoke or smell. A scentless resin, such as tragacanth, is dissolved in water and the solution heated until it boils and is turned into a slimy mass. Into this is introduced pulverised charcoal, which has previously been twice glowd, in such proportion that the mixture of the charcoal and the resin solution contains 3 per cent. of the latter. The mass is worked through a suitable mixing machine till it looks flaky, and is then pressed into blocks and dried in a kiln. Very little air is said to be required for maintaining the combustion, and neither smoke nor smell is said to be perceptible.—L. T. T.

*Improvements in and relating to Mantles for Incandescent Gas Lights.* H. Blücher, Berlin. Eng. Pat. 18,309, September 27, 1894.

THE mantles, which have already been impregnated or treated by any of the known methods to enable them to become incandescent, are placed in a concentrated alcoholic solution of shellac (or other suitable resinous body) to which may be added either magnesium silicate, magnesium chloride, hydrated silicic acid, aluminium silicate, magnesium aluminium silicate, or potassium silicate, or any of them. The mantles are dried (ignited) in a Bunsen flame, which frees them from all organic matter. The mantles are then again preferably subjected to treatment with the alcoholic solution, and once more ignited. The mantles are thus made stronger, more durable, and more elastic. The degree of firmness and elasticity imparted may be varied by variation of the substances added to the solution.—L. T. T.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*The Transport of Petroleum in Bulk.* B. Redwood. Proc. Inst. C.E., Paper 2766, 116, 1893-94. Part II.

THE author principally treats, in this paper, of the subject of the transport of petroleum, from the point of view of minimising the risks of fire and of explosion. He has therefore (1) compiled a critical and historical account of the principal accidents which have occurred with petroleum and other inflammable liquids; (2) conducted a lengthy series of experiments on the evaporation of petroleum, the formation of inflammable and explosive mixtures of vapour and air and the ignition of such mixtures, involving a comparison with the experiments of others in the same field; (3) passed in review the principles of construction of petroleum tank-steamships; (4) enumerated the precautions to be observed in the management of tank-steamships, the use of naked lights, smoking, cleaning tanks, and ventilation; and (5) exhaustively treated the whole subject of the testing of the atmosphere of tanks and other spaces for inflammable gas or vapour. This latter part of his paper contains an account of the hydrogen lamp invented by Professor Clowes and himself for the purpose.

1. *Explosions.*—Perhaps the most interesting general feature brought out by the comparison of these explosions, which has been instituted by the author, is their great

variety, both as to the circumstances leading to the explosions and as to the constituents of the explosive mixtures implicated.

Apart from the disastrous results which have attended the introduction of a naked flame into confined spaces in which ether, carbon bisulphide, and the volatile products of petroleum, or crude petroleum itself, have been allowed to evaporate, it has been found that mineral oil of a not very volatile description is liable to be present in the air in a state of extremely fine sub-division, forming what may be described as a petroleum mist or fog of a highly inflammable character. Thus refined Russian kerosene, with a flashing point of 79° F. (Abel test), brought about the first serious accident connected with the transport of petroleum in bulk, viz., that on the S.S. "Petriana," in December 1886. Here, leakage kerosene lay on the surface of water in a ballast tank and in contact with the lower surface of the partition separating the ballast-tank from the kerosene tank above. The kerosene tank was empty, and the water in the ballast tank being under some pressure, very fine spraying of kerosene took place at certain defective rivet heads. The inflammable mixture of petroleum-fog and air thus gradually formed in the kerosene tank, was unfortunately ignited by workmen carrying open lights. In an experimental imitation of these conditions a similar atmosphere was found by the author to fire at a temperature as low as 50° F. Generally, however, the mixtures exploded have been those of hydrocarbon vapour with air, those hydrocarbons which occur dissolved in crude petroleum, and which are so readily given off, being

responsible for very many of the accidents. As instances of such accidents may be specified those on board the S.S. "Solway," "Petroleum," "Noteros," &c. The "spontaneous" ignition of benzoline vapour, which occasionally occurs during the "dry cleaning" of leather, wool, silk, &c., is due, as originally pointed out by the author (the matter has since been investigated fully by Richter—this Journal, 1893, 598), to electrical discharge, the result of friction between the benzoline and the goods operated upon; wool becoming negatively, and benzoline positively electrified.

2. *Experimental Matter.*—For assistance in this part of the work, the author states that he is indebted to E. R. Blundstone. The author's experiments were principally of two kinds. In the first set of experiments he endeavoured to collect some more accurate information as to the manner in which petroleum and its products evaporate. The apparatus employed consisted of a range of six graduated tubes connected together and immersed in water contained in a large glass tank. Through the petroleum in the tubes a current of dry air was passed, while the exit tube of the system was connected with a condenser cooled by ice water. Materials operated upon were crude petroleum, benzoline (sp. gr. 0.700 at 60° F.), petroleum spirit (0.680 at 60°), gasoline (0.639 at 60°), and pentane, while the water in the tank was maintained at 40°, 60°, 80°, and 100° F. for each material. The residuary volume of liquid in the tubes, after passing air, was periodically read. The author tabulates the results in five tables, the following being that for gasoline:—

TABLE II.  
GASOLINE (Specific Gravity 0.639).

Temperature of Liquid.	Volume of Liquid.	Volume of Air passed through the Tubes.	Volumes of Air to 1 Volume of Liquid.	Volume of Liquid evaporated by 100 Volumes of Air.						
				Tube 1.	Tube 2.	Tube 3.	Tube 4.	Tube 5.	Tube 6.	Total.
° F.	Ce.	Litres.								
40	46.53	1.6	34.3	0.181	0.067	0.018	0.012	0.006	Nil	0.285
40	46.53	2.75	59.1	0.163	0.049	0.020	0.016	0.012	0.007	0.27
40	46.53	4.0	85.9	0.142	0.05	0.03	0.017	0.016	0.01	0.26
60	44.7	0.9	20.1	0.40	0.12	0.06	0.05	0.03	Nil	0.66
60	44.7	2.15	48.0	0.28	0.11	0.07	0.05	0.05	0.03	0.59
60	44.7	3.55	79.4	0.21	0.11	0.07	0.05	0.04	0.03	0.51
80	43.9	1.0	21.1	0.59	0.32	0.30	0.20	0.18	0.15	1.74
80	46.9	2.05	43.7	0.37	0.26	0.24	0.17	0.14	0.12	1.30
80	46.9	3.9	83.1	0.20*	0.20*	0.17	0.14	0.12	0.10	2.03
100	36.0	1.15	31.9	0.49*	0.50*	0.33	0.30	0.16	0.32	2.69

\* Tube empty before reading was taken.

NOTE.—Rapid evaporation and distillation (with condensation of liquid in exit tube) occurred in experiment at 100° F.

Besides the information as to the amount of petroleum and its products which can be carried away at various temperatures in an air current, the tables show the gradual saturation of the air current with hydrocarbon vapour and the much greater amount so vaporised at higher temperatures.

They also show the great readiness with which air will take up the major part of the hydrocarbon it is capable of carrying, complete saturation only being effected by passage through a considerable bulk of liquid.

It is considered that the conditions of these experiments, as a whole, closely imitate the prevailing conditions existing when petroleum products are pumped from one vessel to another, or otherwise agitated with air. The relative quantities evaporated are shown in the table of curves on next page.

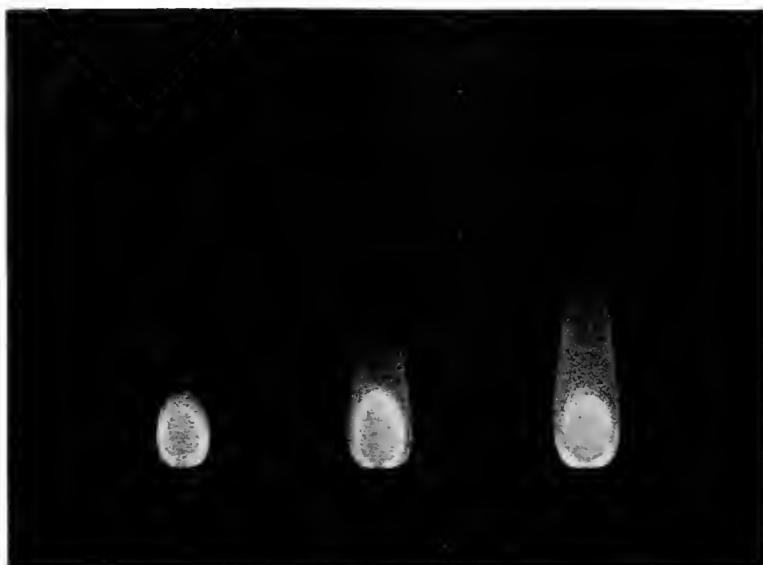
In the author's second set of experiments he endeavoured to determine the respective volumes of a given description of petroleum required for the production, with a given volume of air, of gaseous mixtures, which, under given conditions, are (a) just inflammable, (b) violently explosive, and (c) again only inflammable.

In the case of a liquid (such as pentane) of uniform composition and of ready volatility, it is convenient to introduce gradually increasing quantities of the liquid into a known volume of air and to explode the gaseous mixtures so obtained. The author's results are embodied in a table.

When, however, the liquids under examination were partially or unequally volatile, or with diffidently volatile, it was found impossible to employ this method, and the author therefore manufactured "air-gases" from the various petroleum derivatives and from crude petroleum itself.



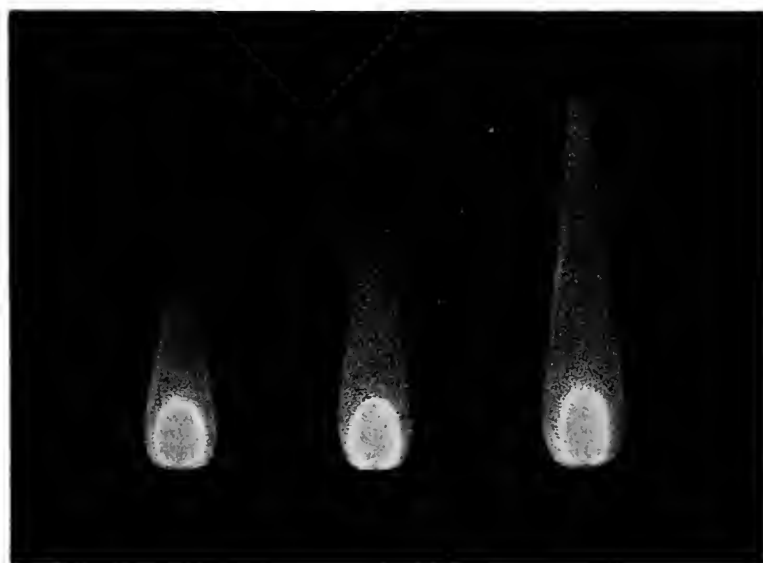




*a*

*b*

*c*



*d*

*e*

*f*

PETROLEUM VAPOR FLAME CAPS  
from Photographs by Messrs B Redwood and T.Horne Redwood

hydrocarbons of petroleum would, under these conditions, be less prone to escape when subjected to the action of the pump.

It is during loading and discharging that the chief risks of explosion occur with tank-ships. The tank covers should, in the case of the transport of crude oil, on no account be removed, for all necessary ingress or egress of air is otherwise provided for.

If it be necessary (as when repairs are to be carried out) to enter the tanks, they must, after the oil has been pumped out as far as possible, be filled with water, thus causing most of the residual oil to overflow. The water is then pumped out and steam injected. Next, "if the vessel is fitted with steam jet or fan ventilating appliances, these should be put in action, and soon afterwards men may be sent into the tanks to clean them down by hand with a stream of water from the pump, the operation being usually completed by drying the surfaces with sawdust." The ventilation must be continued until no vapour remains. "This may necessitate the testing of the atmosphere more than once, but whether this preliminary examination is carried out or not, the atmosphere of the oil-tanks and adjacent spaces should finally be efficiently tested by a competent expert" and a certificate stating the condition of the tank atmospheres, duly issued.

The absolute necessity of the above procedure was abundantly proved by the accident to the S.S. "Tancerville" in 1891, and it was after this accident that the inspectors for the Board of Trade made the following suggestion:—"When repairs require to be carried out in a vessel which has been carrying petroleum, there should, in our judgment, be a formal handing over of the vessel to those by whom the repairs are to be undertaken, and a certificate that the tanks and all dangerous spaces have, so far as practicable, been adequately cleansed and ventilated, and rendered free from risk of explosion or fire. If from any cause a complete certificate of this sort cannot be given, then it should be clearly notified which tanks or spaces have not been cleansed and are still dangerous." This recommendation has been now generally adopted.

**5. Testing the Atmosphere of Tanks and other Spaces.**—It was the accident to the "Tancerville," to which allusion has been made, which directed the author's special attention to this subject; for, although the cause of this accident has never been elucidated, it was at least abundantly evident that, had the tank atmosphere been properly tested for petroleum vapour, the accident would never have occurred. Prior to this accident the author had been accustomed to use the alcohol flame for testing the atmospheres, but he has since experimented with all the different instruments proposed, including the Liveing indicator (which depends upon the increased luminosity—when fire-damp, for example, is present—of a platinum wire heated to incandescence), the Ansell fire-damp indicator (depending on relative diffusion), the Pitkin and Niblett fire-damp detector (essentially two thermometers, the bulb of one of which, being coated with platinum-black, quickly heats in the presence of coal-gas), and with Heck's test (which consists in gently expelling a small syringeful of the suspected atmosphere against a minute flame, a blue flame being produced near the nozzle of the syringe when inflammable vapour is present). None of the foregoing appliances furnished sufficiently satisfactory results.

When the flame of a miner's safety lamp is much reduced in size, if fire-damp be present in the atmosphere, there appears above the flame the well-known "flame cap," caused by a conical region of the gas above the lamp flame becoming feebly luminous. This luminous region is always wider than the lamp flame, and increases in height as the percentage of hydrocarbon vapour in the atmosphere rises. The indications afforded by the miner's safety lamp in this way, are unsatisfactory for petroleum. Those similarly afforded by the large alcohol flame of the Pieler lamp are better, but infinitely more satisfactory and more sensitive are the "flame caps" of a small and almost colourless hydrogen flame. This advantage had been recognised by Pieler in 1883, and still earlier, in 1881, by Mallard and Le Chatelier, but no convenient method of practically using the hydrogen flame had been shown until Clowes

suggested the employment, for the purpose, of hydrogen stored under pressure. This investigator has, moreover, measured the "flame caps" yielded by various percentages of methane (fire-damp) in the air, and has thus placed at the disposal of the miner a portable lamp for not only detecting, but also estimating the amount of the dangerous gas he has to encounter in the atmosphere in which he works.

Meanwhile, the author has invented a lamp on somewhat similar principles, has performed similar experiments with tank atmospheres, and has, moreover, devised a set of apparatus for the collection of samples. The lamp and its accessories have now been in practical use for some time past, and so satisfactory is their use that the author is enabled without hesitation to give the "certificates," previously mentioned as having been suggested by the Board of Trade to be necessary after an examination of tank atmospheres; a result which is quite comprehensible, seeing that 0.75 vol. of pentane vapour may be readily detected in 100,000 vols. of air by the author's apparatus.

In the adjoining illustration the standard hydrogen flame burning in air, free from petroleum vapour, is shown, whilst the five other figures show the "flame caps," when 0.75, 1.5, 3, 5, and 6 vols. of pentane, respectively, are present in 100,000 vols. of air, equal to 0.144, 0.288, 0.576, 0.96, and 1.15 per cent. respectively of vapour in the air. In each case the hydrogen flame was 10 mm. high.

It has been shown by the author's experiments that the vapour of 6.65 vols. of pentane in 100,000 of air is the smallest proportion giving an inflammable atmosphere, and that this proportion must be nearly doubled to give an explosive mixture. The test is therefore a very delicate one, and it is obvious that if a tank or other space be ventilated until, with the author's apparatus, no "flame cap" is obtained, an ample margin of safety will have been provided.

The entire apparatus is shown in Fig. 1, while Fig. 2 gives sections of the lamp, and Fig. 3 sections of the sample collector. In Fig. 1, A is the lamp, B the cylinder containing hydrogen under 120 atmospheres pressure, C the collecting vessel. In Fig. 2, A is the hydrogen inlet tube with regulating valve B, and C is the hydrogen jet. D is the inlet for the atmosphere to be tested. The bore of this tube is greatly contracted, and immediately above its entrance into the chimney of the lamp there are baffles and three wire gauze discs (with not less than 781 openings to the sq. in.). By these means the rush of gas is controlled and back-ignition prevented. The chimney E fits air-tight at the base, but is capable of vertical movement on an inner tube. The chimney is partly metal (darkened inside) and partly glass, and on the window so formed, are marked horizontal lines corresponding to the various values (heights) of the "flame caps."

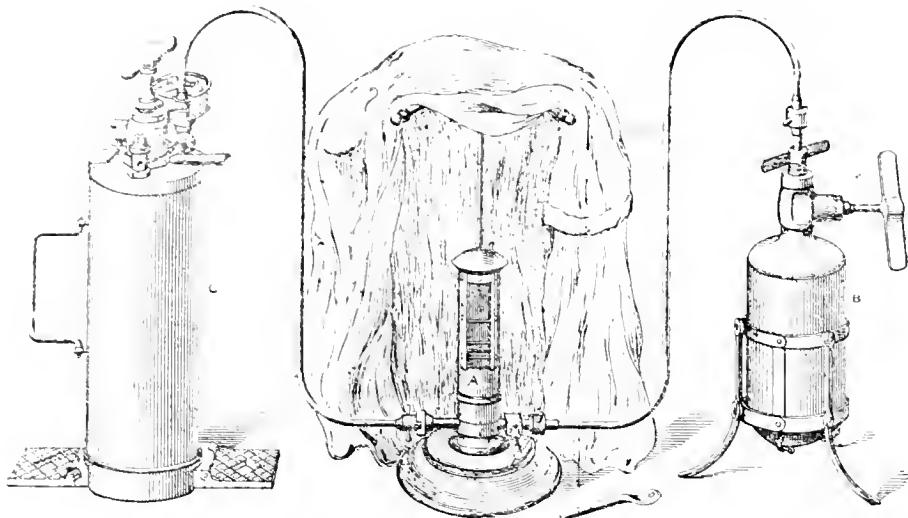
During observations the operator's head is enveloped in the cloth shown in Fig. 1, hydrogen is admitted to the jet at such a rate that the apex of the flame only just reaches the bottom of the window (10 mm.), and then the contents of the collecting cylinder are allowed to escape into the chimney, which they do during two minutes—a space of time quite long enough for observing the height of flame cap produced by the suspected mixture.

The collecting vessel is shown in Fig. 3, where A is the compression pump, with metallic spring piston, fitting the cylinder without the use of leather, and lubricated by plumbago. The surrounding annular space is that in which the sample (the volume of which = cu. ft. under a pressure of 30 lb. per sq. in.) is collected. E is a relief-valve, lifting at 30 lb. pressure. C is the cock connected with the pump, D a pressure-gauge, and F F brackets by which the apparatus is held down by the operator's feet.

The author states that he has recently experimented with an apparatus devised by Professor Dewar, consisting

\* This plate, which has been reproduced from a series of photographs of the actual caps, taken by the author and T. Horne Redwood, gives the true size and general appearance of the caps when viewed in the dark.

Fig. 1.



essentially of a closed cylindrical vessel (diameter, say,  $1\frac{1}{2}$  in.) with a vertical tube (length, say, 10 ft.) attached to the top, and a horizontal one (calibre, say,  $\frac{1}{10}$  in.) near the bottom of the side. A small quantity of a mobile liquid in the cylinder is forced to varying distances along the horizontal tube (which has attached to it a scale) according as the density of the gas in the vertical tube is greater or less than that of air. By increasing the length of the vertical tube or by increasing the ratio between the area of the surface of liquid in the cylinder and that exposed in the horizontal tube, Professor Dewar and the author found the sensitiveness of the instrument to be not inferior to that of the hydrogen flame for detecting small quantities of petroleum vapour in air. Unfortunately,

Fig. 2.

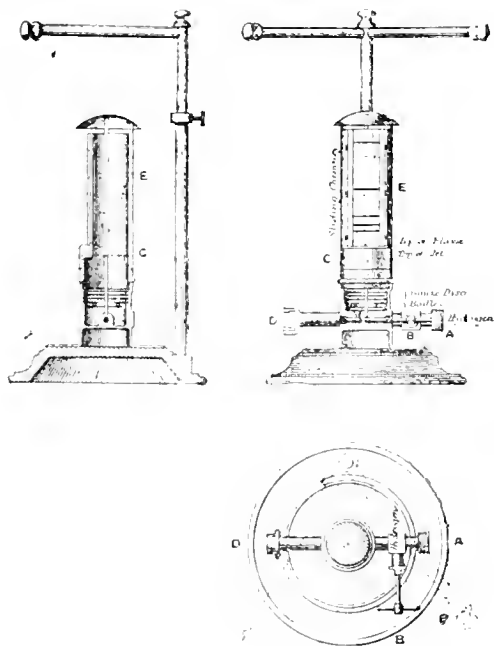
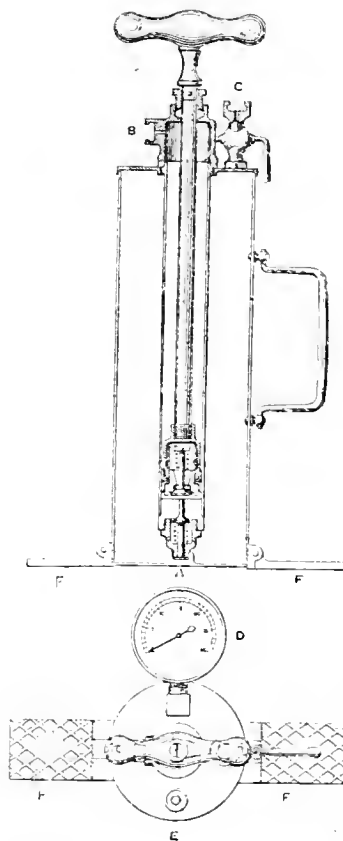


Fig. 3.



however, the instrument, as at present constructed, could obviously only be used on a ship in dry dock or in perfectly still water.—E. R. B.

*Terpenes in Petroleum.* R. Zoloziecki. Dingl. polyt. Jour. 293, 114—119.

CONTINUING his previous work (this Journal, 1891, 628), the author has examined the substances removed by the alkaline lye used for purifying a crude light petroleum obtained by distillation from a mixture of one-third crude Potok, and two-thirds crude Wietrno oil. The specific gravity of the crude light petroleum was 0.732, of the purified 0.736; 75 per cent. of the light petroleum distilled over below 120° C., and a further 16 per cent. below 150° C.

The lye (of which about 50 kilos. were worked up) was clear and of a yellow colour. When this lye was acidified, an oil separated out. This oil was amber yellow and was miscible in all proportions with water, forming an opalescent solution. As it probably contained alkyl-sulphuric, sulphonic, and petroleum acids and their ethereal salts, it was first distilled with a current of steam, the residue then extracted with ether, and the aqueous residue again distilled with a current of superheated steam. The final thick and tarry residue has not yet been examined. The two distillates were combined. Of the ethereal extract the non-saponifiable portion was added to the combined distillates. The small saponifiable portion of the ethereal extract yielded a mixture of petroleum acids boiling above 300° C. These high-boiling petroleum acids obtained from the low-boiling light petroleum may either be due to polymerisation or oxidation of the original petroleum acids present.

The combined distillate formed a mobile oil of pleasant ethereal odour. It absorbed bromine with a partial evolution of hydrobromic acid, showing the formation of both additive and substitution products. Fuming nitric acid formed nitro-derivatives and also oxidation products. Strong sulphuric acid dissolved a considerable part of the oil, even in the cold, and of the dissolved portion a part was precipitated on dilution of the acid with water, whilst a portion remained dissolved in the form of true sulphonic acids. Sodium caused a slow evolution of hydrogen and the formation of gelatinous compounds. Neither sodium hydrogen sulphite (bisulphite), nor phenyl-hydrazine had any action. It was thus clear that the distillate was a mixture of various hydrocarbons and oxygenated compounds. The distillate was therefore carefully fractionated. The portion boiling below 130° C. was lost. Of the remainder the four principal fractions 130°—140° C., 160°—170° C., 170°—180° C., and 210°—220° C. were treated with strong sulphuric acid. The undissolved oil (I.) was in each case then fractionated. The acid was diluted with water and the oil which then separated (II.) washed, dried, and fractionated. The aqueous acid was then neutralised with barium carbonate, the barium salt recrystallised and decomposed by heating with hydrochloric acid in closed tubes, and the oil thus formed (III.) washed, dried, and distilled.

In each case, I. (which formed about half of the fraction) was found to consist of hydrocarbons, of which a part boiled at about the boiling point of the original fraction, a part about 100° higher, and a part at a very high temperature. The lower boiling portions, from their analyses and properties, appeared to be mixtures of hydrocarbons of the  $C_nH_{2n-4}$  and  $C_nH_{2n-2}$  series, the former largely preponderating, and the higher boiling portions, polymerides of the same. Some of these hydrocarbons are almost certainly terpenes or isomerides of the terpenes.

The second products (II.) proved to be secondary or tertiary alcohols derived from the alkyl sulphates formed during sulphonation. The third products (III.) were aromatic hydrocarbons, amongst which para-ethyl toluene, and eymene were recognised. It is probable that other more condensed hydrocarbons were also present.

The lower boiling portion of I. absorbed bromine readily, and the compounds  $C_9H_{14}Br_4$  and  $C_{10}H_{16}Br_4$  were isolated. The portions of higher boiling point (polymerised) only absorbed bromine very slowly, and very soon evolved hydrobromic acid and formed resinous products. With much glacial acetic acid and little sulphuric acid these polymerides gave a green coloration. None of the constituents of I. caused any rotation of polarised light.

If these compounds really are terpenes the purification lyes from some petroleum may probably form a valuable

source for their preparation. Experiments seem to show, however, that all crude petroleum do not contain them, and apparently their absence is accompanied by absence of the petroleum acids. The presence of terpenes in petroleum would account for the tendency of the latter to resinify and to become discoloured, and for the ozonising action of some petroleum in the presence of sunlight. Their presence is no argument against the theory that petroleum are derived from animal remains by bacterial and brominising action, as terpenes have been proved to be formed during alcoholic fermentation.

The removal of the terpenes, &c. from the crude petroleum by the lye is probably due to their previous conversion into sulphonic and alkyl sulphuric acids by the treatment with strong sulphuric acid.—L. T. T.

*Quantitative Composition of the Creosotes obtained from the Wood of the Beech and from that of the Oak.* A. Béhal and E. Choay. Comptes rend. 119, 166.

THE two materials employed by the authors were the heavy oils, collected during the distillation of the tar obtained from the woods of the two trees in question, and, to secure uniformity of result, they were treated in identical apparatus and by the following method:—

The heavy oil was acidulated with hydrochloric acid, agitated with water, the liquids separated (the aqueous layer thus removing certain basic bodies occurring in the oil), the oily layer dissolved in soda-lye, a very considerable quantity of water added (necess. as concentrated solution of the alkali phenates dissolves a certain quantity of neutral substances), layers of liquid separated, alkaline solution treated with benzene, benzene separated, aqueous solution treated with a current of steam as long as distillate smells strongly. Hydrochloric acid is now added, the liberated phenols decanted, the aqueous layer agitated with benzene, the latter separated and distilled, and its phenol residue thus obtained added to the bulk.

The phenols thus separated were twice rectified, a Le Bel-Henninger fractional distillation apparatus with five bulbs being employed, and two distillates, passing over between 200° and 210° and between 210° and 220° respectively, collected.

In the case of beechwood-creosote the weights of the distillates were as 1,000 : 367 and the sp. gr. 1.085 at 17° and 1.085 at 15° respectively. The 206°—210° oak creosote distillate had sp. gr. 1.068 at 15°.

The authors have analysed these various distillates by the method they have previously described (this Journal, 1893, 618), and find as follows:—

Pure beechwood-creosote, distilling 200°—220° (obtained by mixing 1,000 parts of the 200°—210° fractions with 367 parts of 210°—220° fraction).

Monophenols .....	39.00
Guaiacol .....	19.72
Creosol and homologues .....	39.98
Loss .....	1.30
	100.00

The 200°—210° fractions yielded as follows:—

	From Beech-wood Creosote.	From Oak-Creosote.
Monophenols .....	39.00	55
Guaiacol .....	26.48	14
Creosol and homologues .....	32.14	31
Loss .....	2.38	..
	100.00	100

By comparing the two analyses of beech-creosote fractions, it will be seen that the 210°—220° fraction can contain no guaiacol.



By contrasting the oak- and beech-creosotes it will be seen that the former has the lesser density, and contains less guaiacol and more monophenols; it is the larger presence of the latter which makes it the more "caustic" of the two creosotes.

The authors have gone further, though they admit that their results now lose that precision which they declare is characteristic of the preceding work. In fractionating the monophenols the authors weighed all fractions coming over at a fixed temperature and divided up the intermediate fractions by calculation, half of each intermediate fraction being added to the phenol of highest boiling point. They consider this to be the best practical method of dealing with these fractions which are irreducible by distillation.

As an example of the difficulties of such "carrying over" phenomena, as are met with in the distillation of these creosotes, the authors mention that their 200°—210° fraction contains 5 per cent. of common phenol of boiling point about 179°, that is to say, 2.5° lower than that of the creosote containing it; while, on the other hand, it carries 35 per cent. of creosol and its homologues, of boiling point about 220°, that is to say, 10° higher than the creosote containing it. Working, however, according to their method they find, for the 200°—210° beech-creosote, as follows:—

Phenol .....	5.20
Orthocresol .....	10.40
Meta and paracresol .....	11.60
Ortho-ethylphenol .....	3.60
Metaxylenol, 1, 3, 4 .....	2.00
Metaxylenol, 1, 3, 5 .....	1.00
Phenols various .....	6.20
Guaiacol .....	25.00
Creosol and its homologues .....	35.00
	<hr/> 100.00

In conclusion the authors point out that creosote is popularly supposed to contain 60—90 per cent. of guaiacol, the remainder being creosol, with a small amount of monophenols; the above figures indicate for it a very different composition.—E. R. B.

#### *Tar of Pinus Maritima.* A. Renard. Comptes rend. 1894, 119, 165.

This tar, which is obtained, in the *landes* of South-western France, during the carbonisation of stacks of the wood of *Pinus maritima*, is met with as a thick, dark brown liquid of sp. gr. 1.054. Upon distillation it yields—

1. Water .....	3.5
2. Hydrocarbons (distilling under 300° C.) .....	12.0
3. " (between 300° and 360°) .....	45.0
4. Phenols .....	18.0
5. Pitch (containing much retene) .....	21.5
	<hr/> 100.0

The phenols are separated from their distillate by lixivation with soda—which must be used boiling to avoid emulsifying the product—and subsequent treatment with acid.

The author has isolated, by fractional distillation, operating upon 50 kilos. of the crude tar, three hydrocarbons; one of these he now describes.

It is a colourless liquid, with b.p. 171°—174°, sp. gr. 0.866 at 0°, rotation  $\alpha_D = -19.1$ , index of refraction = 1.4785, formula apparently  $C_{10}H_{16}$ , and with vapour density 4.6 (against theory, 4.7).

It absorbs oxygen more actively than turpentine does. A di-bromo additive compound ( $C_{10}H_{14}Br_2$ ), a tetrabromo derivative ( $C_{10}H_{12}Br_4$ ), and a crystalline dihydrochloride have been obtained.

With sulphuric acid much heat is evolved, and from the supernatant liquid, upon distillation, are obtained a distillate boiling about 170°, which is mostly cymene, and a very thick residue, which is a polymer of the hydrocarbon, boiling about 300°. Upon treating the 170° distillate with fuming

sulphuric acid, cymene sulphonie acid and another hydrocarbon were obtained. This latter, which is not acted upon by fuming nitric acid, nor by bromine in the cold, and has for its formula  $C_{10}H_{20}$ , is cymene hexahydride.—E. R. B.

#### *Pine Tar.* A. Renard. Comptes rend. 1894, 119, 652—654.

THE fraction of pine tar boiling at about 250°—280° C. contains a new hydrocarbon; it may be purified by treating the crude fraction with sodium, and is then obtained as a colourless liquid of sp. gr. 0.9419 at 0° C., and having a boiling point of 254°—257°. Its refractive index is 1.507. Analyses and vapour density determinations indicate that the new hydrocarbon has the formula  $C_{14}H_{22}$ . It furnishes a crystalline tetrabromo-derivative,  $C_{14}H_{18}Br_4$ , and a nitro-derivative  $C_{14}H_{21}(NO_2)$ , and gives a blue coloration when heated with sulphuric acid and alcohol, a reaction which according to Maquenne is characteristic of hydrogenised aromatic hydrocarbons. The hydrocarbon when treated with ordinary concentrated sulphuric acid, develops heat; if the solution be poured into water a sulphonic acid passes into solution, which yields an insoluble barium salt ( $C_{14}H_{21}SO_3)_2Ba$ , whilst the portion of the product insoluble in water, when steam-distilled yields a hydrocarbon,  $C_{14}H_{26}$ , boiling at 250°—253° C., which behaves as a saturated compound, and is perhaps dodecahydro-ditolyl, ( $C_7H_7$ ,  $H_6$ )<sub>2</sub>. The hydrocarbon  $C_{14}H_{22}$  may then be octahydro-ditolyl, ( $C_7H_7$ ,  $H_4$ )<sub>2</sub>. (See previous extract.)—A. R. L.

#### *New Constituents of Wood Oil.* E. Looft. Ber. 27, 1894, 1542.

THE present paper is in continuation of one published in Liebig's *Annalen*, 275, 366 (this Journal, 1893, 919). In addition to the compounds there described the author has isolated:—*Methyl pyridine* and *dimethyl pyridine* in the fractions boiling above 115° C., *butyl alcohol* (probably iso-) boiling about 115°, *pinelic ketone*,  $C_8H_{10}O$ , in the fraction boiling from 150°—160°, and an alcohol possessing the formula  $C_8H_{14}O$  in this same fraction.—T. E.

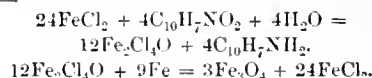
#### *Ey-products obtained in the Preparation of Butyltoluene.* A. Baur. Ber. 27, 1894, 1606.

See under XX., page 1218.

## IV.—COLOURING MATTERS AND DYES.

#### *The Technical Method of Preparing Aniline.* A. Wohl. Ber. 27, 1815—1818.

THE author makes reference to O. N. Witt's explanation of the small consumption of hydrochloric acid in the technical process of reducing nitro-compounds by means of metallic iron. According to this, the ferrous chloride formed is the real reducing agent, its action being due to its conversion into basic ferric chloride, whilst by the metallic iron it is constantly being regenerated from the latter. Witt's equations for the reduction of nitronaphthalene are as follows:—



While admitting the clearness of this explanation, and confirming the correctness of the conversion of basic ferric chloride into ferrous chloride, and the oxide  $Fe_3O_4$ , as assumed in the second equation, the author, in view of the fact of ferrous chloride being replaceable by salts ( $CaCl_2$ ,

Se.) not capable of exerting a reducing action by conversion into higher oxidation compounds, still thinks his view of the direct reducing action of metallic iron, as recently expressed (this Journal, 1894, 876), the one best expressing the reaction actually taking place.—F. M.

*Sulphur Compounds accompanying Aniline Bases, and their Colour Reactions.* A. Hantzsch and H. Freese. Ber. 27, 2329—2334.

CHEMICALLY pure aniline does not give the blue colour reaction with chloride of lime hitherto considered characteristic. The coloration obtained with aniline from coal-tar benzene is due to the presence in it of traces of a sulphur compound, probably amido-thiophene. The same rule applies to a number of other bases, and more especially to commercial para-amidophenol. Attempts to isolate the sulphur compound have as yet been fruitless, but a reliable method for its removal from aniline seems to consist in heating the latter for some time with acetone. This evidently forms a condensation product with the amidothiophene, which, on distillation of the aniline, remains behind as a resinous mass rich in sulphur. Para-amidophenol may be purified in like manner. The product crystallising from the acetone solution is distinguished from the ordinary para-amidophenol by its remaining colourless on exposure to air.—F. M.

*Tannin Orange.* C. Schoen. Bull. Soc. Ind. Mulhouse, August and September 1894, 350.

*Tannin Orange R*, a new colour manufactured by Cassella, of Frankfurt, is an azo colour, and possesses the property, rarely shown by this class of dyestuffs, of being fixed by tannin. It is obtained by the mutual reaction of para-amidobenzylidimethylamine and  $\beta$ -naphthol and has a composition expressed by the formula—



The colour is sold as a 50 per cent. paste, which gives a clear solution with distilled water; but when dissolved in calcareous water the solution rapidly becomes turbid. Its solution is precipitated by ammonia and the fixed alkalis, the precipitate being easily redissolved by acids.

As a printing colour it is fixed by tannin in presence of acetic acid. The addition of tartaric acid presents no advantage. It is not affected by sulphurous acid in steaming, and its resistance to chlorine is good. It has a medium fastness against light. It is less useful for dyeing on a tannin mordant, as the white grounds are strongly coloured. Its principal use will be in combination with Methylene blue or green, with which it gives pretty mode and grey tones.—W. E. K.

## PATENTS.

*Improvements in the Manufacture of Colouring Matters.* J. Y. Johnson, London. From "The Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 18,759, October 6, 1893.

THE soluble salts (compounds with acids) of safranine azo-naphthol bodies described in Eng. Pat. 4543 of 1891 (this Journal, 1892, 236) have been introduced into commerce under the name of Indoin blue, and afford a perfect substitute for indigo in some of its applications. It is now found that the insoluble safranine-azo-naphthol bodies can be converted into soluble basic dyes, practically equivalent to Indoin-blue, by prolonged washing with water, whereby the excess of alkali and salts is removed. For example, a 1 per cent. solution of safranine is made, taking one molecular proportion of the safranine, i.e., about 7 kilos, of safranine T (obtained by oxidation of tolylenediamine, o-toluidine, and aniline), or the equivalent quantity of another easily diazotisable safranine, and diazotised by adding a solution of 14 kilos. of sodium nitrite and 23 kilos. of 30 per cent. hydrochloric acid, the mixture being kept cool by ice. It is then run into an ice-cold solution of about 3 kilos. (one molecular proportion) of  $\alpha$ - or

$\beta$ -naphthol in 160 litres of water and 25 kilos. of caustic soda solution containing 35 per cent. NaOH. The products are washed with water until the wash-water becomes deeply coloured, and the washed paste, made up to a standard strength, is then ready for use.—T. A. L.

*The Manufacture and Production of New Compounds suited for Use in the Preparation of Diazo Compounds and their Application in the Production of Dyes.* J. A. Johnson, London. From "The Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 20,605, October 31, 1893.

THE diazo compound of  $p$ -nitraniline, when treated with an alkali at ordinary temperatures, is converted into the alkali salt of the isomeric nitrosamine. This is a tolerably stable substance, and readily crystallisable. When dissolved and mixed with sodium  $\beta$ -naphtholate and an excess of alkali it gives no colour, but if the nitrosamine salt be treated with rather more than one molecular proportion of acid, preferably in presence of a little nitrous acid, the nitrosamine is reconverted into the diazo compound, which will then combine with an alkaline solution of  $\beta$ -naphthol. Other aromatic amido compounds react in the same way, providing they contain substituting acid groups, such as one or more nitro groups, two or more halogen groups (in the one benzene ring), and with or without sulphonic acid groups. The following substituted amines have been employed: o-nitraniline, nitro- $p$ -toluidine ( $CH_3:NO_2:NH_2$  1:3:4); dichloro-aniline ( $Cl_2:NH_2$  1:4:3); 1,4-nitro-naphthylamine, dinitrobenzidine, the sulphonic and carboxylic acids of nitraniline and pteramic acid. For preparing the nitrosamine salt from  $p$ -nitraniline a 10 per cent. solution of the diazo compound from 135 kilos. of  $p$ -nitraniline is run into 8,000 kilos. of caustic soda solution containing 18 per cent. NaOH, at about 60—70° C. with vigorous stirring. On cooling, the nitrosamine salt separates in crystals, and may be purified by recrystallisation from 700 litres of hot water. In practice, it is only necessary to run the diazo solution into 18 per cent. cold caustic soda lye, taking care that the solution always remains strongly alkaline. The nitrosamine salt separates out and is filter-pressed and dried or kept as a paste. To convert it into free  $p$ -nitrophenyl nitrosamine, about 10 kilos. of the salt are dissolved in 250 litres of cold water, and decomposed with 30 kilos. of 6 per cent. hydrochloric acid. The free nitrosamine, which separates at once, is filtered off and must be used in the form of paste. The further conversion into the diazo compound is affected by adding more acid, as in the following example. About 10 kilos. of the nitrosamine salt are dissolved in 250 litres of water and this solution is run into 18 kilos. of 30 per cent. hydrochloric acid. The precipitate of the free nitrosamine disappears as it is converted into the diazo compound, and the addition of about 1 per cent. of sodium nitrite considerably accelerates the conversion. This conversion can be effected on the fibre by printing a mixture of the nitrosamine salt with certain phenols and amines, together with an excess of alkali, and gradually neutralising the alkali, either by exposure to the atmosphere or treatment with acetic acid, when the diazo compound produced immediately combines with the component present to form the colour.—T. A. L.

*The Manufacture of Colouring Matters Dyeing Blue with Mordants.* C. D. Abel, London. From "The Actien Gesellschaft für Anilin Fabrikation," Berlin, Germany. Eng. Pat. 23,311, December 4, 1893.

THE invention is based on the observation that ortho-quinones or their sulphonic acids form condensation products with alkylated  $p$ -phenylene-diamines which, when afterwards heated with sulphur and fuming sulphuric acid, yield blue colouring matters capable of forming lakes with metallic mordants, which dye fast blue shades and are especially suitable for dyeing and printing chrome mordanted wool and cotton. For example, 13.6 kilos. of dimethyl- $p$ -phenylenediamine hydrochloride are dissolved in 100 litres of water, and, while agitating, 27.6 kilos. of

the potassium salt of 1,2,3'-naphthoquinone sulphonic acid are added. The solution is kept slightly alkaline by sodium carbonate, salt is added, and the mixture slightly heated while a current of air is blown through it. The product, filtered off and dried, forms a blue powder. 25 kilos. of this intermediate product and 12 kilos. of sulphur are dissolved in 250 kilos. of 30 per cent. sulphuric anhydride. The mixture is well stirred for about 12 hours and finally gently heated until a sample dissolves in boiling water with a bright blue colour. The mixture is then poured into iced water and heated to boiling. The precipitated colouring matter is filtered off, dissolved in dilute soda, filtered again to remove sulphur, and finally precipitated with hydrochloric acid or salt. When instead of quinonemonosulphonic acids the quinones themselves are used, the same result is obtained, because sulphonation takes place in the process, and when such quinonesulphonic acids are employed as contain a sulphonic acid group in the position 4, this group is split off during the condensation.—T. A. L.

*The Manufacture or Production of Colouring Matters.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 23,392, December 5, 1893.

In Eng. Pat. 19,062A of 1891 (this Journal, 1892, 809) a process for producing dyestuffs was described, which consists in condensing tetra-alkylated diamidobenzhydrols with secondary amines, subsequently treating the so-formed leuco-products with nitrous acid, oxidising the resulting leuco-compounds containing a nitrosamine group, and finally splitting off the nitroso group. Some of these colouring matters can also be produced by the process described in Eng. Pat. 5038 of 1884 (this Journal, 1885, 279), namely, by allowing tetra-alkyl diamidobenzophenone to act, in the presence of phosphorus oxyhaloid or the like, on certain of the secondary amines. The number of secondary amines suitable for treatment by this last process is by the present patent extended, so as to include mono-methyl and mono-ethyl- $\alpha$ -naphthylamine. Benzyl- $\alpha$ -naphthylamine has already been included among them in Ger. Pat. 69,863. Analogous colouring matters are formed from *p*-alkyl-oxypheyl- $\alpha$ -naphthylamine or analogous alkyl-oxy compounds by either of the two processes above mentioned. The said *p*-alkyl-oxypheyl- $\alpha$ -naphthylamines are prepared by heating  $\alpha$ -naphthylamine with *p*-amido-alkyl-oxybenzene hydrochloride. The following is an example of the production of one of the dyestuffs. Eight kilos. of tetramethyldiamidobenzophenone are mixed with 8.1 kilos. of *p*-ethoxyphenyl- $\alpha$ -naphthylamine, and then 5 kilos. of phosphorus oxychloride added gradually, with continual stirring. After standing some time at the ordinary temperature the mixture assumes a metallic lustre and becomes crystalline. It is then heated for some time to 107°–110° C., powdered, washed with cold water, and dissolved in dilute hydrochloric acid (15 per cent. HCl). The dyestuff is precipitated from this solution by adding water. The colouring matter is easily soluble in hot water and gives greenish-blue shades on wool and on cotton mordanted with tannic acid.—T. A. L.

*The Manufacture or Production of Colouring Matters derived from Anthraquinone.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 23,585, December 7, 1893.

In Eng. Pat. 4871 of 1891 (this Journal 1892, 513) a process was described for the production of dyes by acting on certain polyhydroxyanthraquinones with atmospheric oxygen in presence of ammonia. A simpler method has now been discovered yielding similar results and of more general application. This consists in treating hydroxyanthraquinones, in presence of ammonia, with ammonium persulphate or other salt of persulphuric acid (J. Prakt. Chem. 48, 185, and this Journal 1891, 1004), using two molecules of persulphate to one of hydroxyanthraquinone. The action takes place readily in dilute aqueous solutions at a low temperature.

For example, 50 kilos. of 20 per cent. Alizarin bordeaux paste are dissolved in 500 litres of water and 100 litres of

20 per cent. ammonia. A solution of 8.5 kilos. of ammonium persulphate in 50–100 litres of water is then gradually stirred in, the mixture being kept cool by ice. The action is finished when a sample treated with acid gives a precipitate which dissolves in sulphuric acid with a cherry-red colour. The melt is then mixed with hydrochloric or sulphuric acid and the dark purple-red dyestuff filtered off and washed with water. This dyestuff differs materially from that formed by heating Alizarin bordeaux merely with ammonia (Eng. Pat. 8702, of 1891 this Journal, 1892, 514), the latter giving a blueish-red solution in sulphuric acid, whilst the pure new product dissolves in sulphuric acid with a yellowish-red colour. The new dyestuff produces on chromed wool intense greenish-blue shades retaining their colour in artificial light. As another example of the same process, the manufacture of a similar colouring matter from Alizarin blue paste is described.

The product combines with alkaline bisulphites forming compounds soluble in water. It yields beautiful greenish-blue shades on chromed wool. A table is given, showing the principal properties of nine colouring matters of the series, namely those formed from Alizarin blue, Alizarin bordeaux, Alizarin-pentaeryanine, Alizarin-hexaeryanine, hexahydroxyanthraquinone, and from the sulphonic acids of each of the last four. If the above process be carried out in the presence of sulphurous acid or a sulphite, sulphonated dyestuffs are obtained, the introduction of nitrogen ("amidation") and sulphonation thus taking place at the same time.

Sulphonated dyestuffs similar to the above may also be obtained by sulphonating the products of the action of ammonia on polyhydroxyanthraquinones with fuming sulphuric acid at 90°–100° C. The sulphonic acids yield clearer and greener shades than the unsulphonated dyes, and owing to their solubility are especially suitable for printing on wool.

The last described sulphonating process may be inverted, that is, polyhydroxyanthraquinones may first be sulphonated by the methods described in Eng. Pat. 12,715 of 1890 (this Journal, 1891, 759); Eng. Pat. 17,712 of 1890 (this Journal, 1891, 917); Eng. Pat. 12,580 of 1892 (this Journal, 1893, 596); and Eng. Pat. 8702 of 1891 (this Journal, 1892, 514), and the sulphonic acids so formed then treated with ammonia either by the process described in the last mentioned patent or by merely heating with a dilute ammonia solution at about 80° C. The isolation of the sulphonic acids of "amidated" polyhydroxyanthraquinones is best effected by acidulating the ammoniacal solution at a low temperature (to avoid decomposition and elimination of nitrogen) when the dyestuff either separates at once or on addition of salt. An example is given. The processes described in Eng. Pat. 15,246 of 1892 (this Journal, 1893, 598), and in the fifth claim of Eng. Pat. 15,325 of 1892 are hereby disclaimed.—T. A. L.

*Improved Process of Making Solid and Friable Colouring Matters from Logwood Extract and other Dye-wood Extracts.* P. R. J. Willis, London.—From W. J. Matheson, New York, U.S.A. Eng. Pat. 24,365, December 18, 1893.

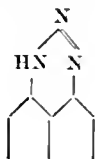
The product obtained from logwood by the process claimed in Eng. Pat. 2316 of 1893 (this Journal, 1893, 598) requires the addition of a small amount of acid to the dye-bath which in certain cases is objectionable. According to the present patent, a more valuable product is obtained having a higher tinctorial value by the addition of ammonium carbonate to a hot extract of logwood of high gravity. The following is the method employed. Logwood extract of about 70° Tw. is heated to about 65° C., and thoroughly mixed with 7 per cent. of ammonium carbonate previously dissolved in as small a quantity of water as possible. The extract is poured into shallow trays and dried at about 20° C. until the mass has become spongy and a hard crust has formed. When this is the case the trays are dried at 65°–82° C., and when thoroughly dry the mass is ground. It is not affected by atmospheric moisture, but is soluble in cold or hot water, and dyes chromed wool in the same way as cured chip logwood.—T. A. L.

*The Manufacture of Colouring Matters and of Intermediate Products related to the Rhodamine Series.* J. Y. Johnson, London. From "The Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 24,543, December 20, 1893.

By treating the rhodamines derived from phthalic or succinic acids with ammonia under pressure a replacement of oxygen by the imido group (NH) takes place. This body is not a dyestuff, but on treatment with condensation agents, such as phosphorus oxychloride or pentachloride, zinc chloride, or fuming sulphuric acid, water is split off and a new rhodamine dye yielding bluer shades is produced. If the ammonia be replaced by aniline or its homologues, analogous phenyl imides are obtained, which on sulphonation give valuable acid colouring matters dyeing bright shades on animal fibres and capable of dyeing darker shades than the basic rhodamines. About 1 kilo. of tetra-ethyl phthalic rhodamine (Rhodamine B extra) is dissolved in 5 kilos. of alcohol, and the solution is saturated in the cold with ammonia. The solution thus obtained is heated in an autoclave to 180° C. for 10 hours, and, after cooling, the crystals produced are filtered off and if necessary recrystallised from alcohol. They melt at 229° C. About 5 kilos. of the rhodamine-imide so obtained are mixed with 2 kilos. of phosphorus oxychloride and heated for two hours on the water-bath. The product is then dissolved in water, filtered, and the new dyestuff salted out. The new colouring matter gives violet-red shades on wool and silk and bluish-violet shades on cotton mordanted with tannin. The following method is given for the preparation of a dyestuff by the action of aniline on rhodamine:—About 1 kilo. of tetra-ethyl phthalic rhodamine is treated with 3 kilos. of aniline under an inverted condenser for four hours. After the excess of aniline has been removed the product is purified by washing with alcohol or recrystallising, and 1 kilo. of the rhodamine-phenyl-imide so formed, is then dissolved in 5 kilos. of 100 per cent. sulphuric acid and sulphonated on the water-bath by gradually adding small quantities of fuming sulphuric acid containing 23 per cent. SO<sub>3</sub>. The sulphonation is complete when a sample is completely soluble in dilute soda solution. The melt is then poured into water, the sulphonic acid filtered off, and converted into the sodium salt. This salt is sparingly soluble in cold water, but by further sulphonation a salt more readily soluble can be obtained. This forms a red powder easily soluble in water, giving a bluish-red solution with a yellowish-red fluorescence. The dyestuff gives red fluorescent shades on wool, the dark shades being especially brilliant, and the colouring matter is said to be in a high degree fast to light.—T. A. L.

*The Production of Azo Dyestuffs Derived from  $\alpha_1, \alpha_2$  Azimido-naphthalene.* S. Pitt, Sutton, Surrey. From L. Cassella and Co., Frankfurt-on-the-Maine, Germany. Eng. Pat. 24,714, December 22, 1893.

According to Ger. Pat. 63,507, 1,1'-naphthylene diamine disulphonic acid combines with diazo compounds, and these products, according to Ger. Pat. 70,031, are converted into the corresponding azo derivatives of 1,1'-amidonaphthol disulphonic acid by treating them with mineral acids or caustic alkalis. The same effect cannot be produced by nitrous acid since this reagent produces an azimido body the substance formed having the characteristic group—



It is stated that all the azo derivatives of the mono- and di-sulphonic acids of 1,1'-naphthylene diamine show this reaction, and the colouring matters produced show a certain similarity to the corresponding azo derivatives of the 1,1'-dihydroxy-naphthalene sulphonic acids. They are

said to be easily soluble and to be very fast to light. The method employed is as follows:—27.6 kilos. of *p*-nitraniline are diazotised and combined in an acid solution with 75 kilos. of 1,1',3,3'-naphthylene diamine disulphonic acid. When the combination is complete, a solution of 23 kilos. of sodium nitrite is run in and the dye stuff which had separated goes into solution. After a short time yellowish red needles of the azimido dyestuff begin to separate and can be filtered off after a few hours. The colouring matter dyes wool magenta red from an acid bath. Similar products are obtained if the *p*-nitraniline be replaced by *o*- or *m*-nitraniline, nitrotoluidine, amidonaphthalene or nitro-naphthylamine, the shades varying from bluish red to reddish violet. Colouring matters having similar properties but rather less soluble are obtained by replacing the above naphthylene diamine disulphonic acid by the 1,1' naphthylene diamine sulphonic acid of Eng. Pat. 1613 of 1893 (this Journal, 1891, 244).—T. A. L.

*The Production of Dyestuffs from Diazo-safranines and Amidonaphthols.* S. Pitt, Sutton, Surrey. From L. Cassella and Co., Frankfurt-on-the-Maine, Germany. Eng. Pat. 24,715, December 22, 1893.

THE dyestuffs referred to, obtained by the action of diazotised safranines on certain of the amido-naphthols, are basic and dye cotton mordanted with tannin, blue-black shades. As an example of the method employed, the following details are given:—35 kilos. of safranine, C<sub>20</sub>H<sub>14</sub>N<sub>4</sub>O<sub>2</sub>, are dissolved in water and diazotised with hydrochloric acid and 7 kilos. of sodium nitrite, and the diazo compound thus produced is added to a solution of 16 kilos. of 1,2'-amidonaphthol (Ger. Pat. 69,458) in 12 kilos. of hydrochloric acid. The formation of the dyestuff is complete in a short time, and after salting out, it is filter-pressed and dried. It dissolves in water with a bluish-black colour and dyes cotton mordanted with tannin, an intense blue-black. The combination may also take place in an alkaline solution, the dyestuff so obtained only differing slightly from that obtained in acid solution. Similar dyestuffs are also produced by substituting the 1,4', 1,1', 2,2', or 1,3'-amidonaphthol for that in the example described above.—T. A. L.

*The Manufacture and Production of New Colouring Matters.* C. Dreyfus, Clayton, Manchester. Eng. Pat. 24,870, December 27, 1893.

By combining the diazo compounds of *m*- or *p*-nitraniline, of nitro-toluidines, or of nitro-xylidines with the sulphonic acids of dehydro-biotoluidine, primuline base, dehydrothio-*m* xylidine or dehydrothio- $\psi$ -cumidine, the patentee obtains colouring matters ranging from greenish yellow to orange-shades, which dye unmordanted cotton. The combinations may take place in an alkaline or acetic acid solution, and the following details are given:—13.8 kilos. of *p*-nitraniline are dissolved in 500 litres of boiling water and 100 kilos. of 34 per cent. of hydrochloric acid, and after cooling diazotised with 7 kilos. of sodium nitrite. The diazo solution is run into a solution of 34 kilos. of dehydrobiotoluidine sulphonic acid and 80 kilos. of sodium carbonate in 1,000 litres of water, and after standing 24 hours the whole is boiled up and the colouring matter salted out and filtered off.—T. A. L.

*The Manufacture or Production of Sulpho Acids of the Naphthalene Series derived from Naphthalene  $\beta_1 \beta_3$  Disulpho Acid or Naphthalene  $\beta_2 \beta_3$  Disulpho Acid.* H. E. Newton, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 25,074, December 30, 1893.

THE naphthalene tetrasulphonic acid of Senhofer (Ber. 8, 1486) is a mixture of at least two isomeric acids and a mixture is also obtained, according to the method described in Ger. Pat. 40,893, in which 1 kilo. of naphthalene is heated with

5 kilos. of 10 per cent. anhydride at 160° C. In the present patent, methods are given for producing pure naphthalene tetrasulphonic acids by sulphonating 2,3'- or 3,3'-naphthalene disulphonic acid. The process consists in heating the calcium salt of the acid, previously dried at 200° C., with three times its weight of fuming sulphuric acid, containing 25 per cent. of anhydride, first of all for four hours to 90° C. At this stage a naphthalene trisulphonic acid is produced. The vessel is then closed and the temperature is raised for six hours to 260° C. The melt is dissolved in water, the excess of sulphuric acid removed by lime and after converting into the sodium salt, the solution is evaporated. Salt is then added which precipitates the sodium salt of the naphthalene tetrasulphonic acid. The tetrasulphonic acid derived from the 2,3'-naphthalene disulphonic acid when heated with caustic soda yields two new naphthol trisulphonic acids, the sodium salts of which are separated by their difference in solubility in water. If in the fusion, which in both cases is conducted under pressure, a larger quantity of soda be employed and the temperature is also higher, the tetrasulphonic acid is converted into two dihydroxy-naphthalene disulphonic acids, which are termed R and Y acids, the proportion of the two being about 3 : 7. The former acid separates in long hair-like needles and aqueous solutions of its neutral salts show a violet fluorescence. The Y acid is much more soluble and is separated by means of its potassium salt. Aqueous solutions of the neutral salts of this acid are deep yellow and show a green fluorescence.—T. A. L.

*The Manufacture or Production of Beta, Beta, Dihydroxy-naphthalene Beta, Beta, Disulphonic Acid.* H. E. Newton, London. From "The Farbentabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 25,074, December 30, 1893.

This patent describes another method for the production of 2,2',3,3'-dihydroxynaphthalenedisulphonic acid which has already been prepared according to Eng. Pat. 16,199 of 1893 (this Journal, 1894, 876), by sulphonating 2,2'-dihydroxynaphthalene. According to the present method the same product is obtained from 2,3,3',2'-naphthol trisulphonic acid by heating with caustic soda under pressure. 50 kilos. of the sodium salt of  $\beta$ -naphtholtrisulphonic acid are heated with 100 kilos. of caustic soda and 50 litres of water in an autoclave for 6—10 hours to 220°—300° C. until a sample no longer contains naphtholtrisulphonic acid which separates in needles. The melt is then mixed with acid when the acid sodium salt of the dihydroxynaphthalenedisulphonic acid separates out as a crystalline powder. Its alkaline solutions show a bluish-green fluorescence. When heated with 20 per cent. sulphuric acid to 200° C. it yields 2,2'-dihydroxynaphthalene, melting at 185° C., by means of which its constitution was determined.

—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Artificial Production of Woollen Crape.* F. Binder. Bull. Soc. Ind. Mulhouse, June 1894, 267—268.

THE raw woven woollen crape is crabbed to remove stiffening, first, for 10 minutes in water at 25° C., and then in boiling water for 15—20 minutes. It is then dried in the drying-room (not on drums), and is given a slight stiffening with gum tragacanth. This process is applicable to rough and smooth woollen, and also to cotton crapes.—H. I.

## PATENTS.

*An Improved Method of Preventing Spontaneous Ignition of Liquid Hydrocarbons used for Cleansing and other Purposes.* M. M. F. Richter, Hamburg, Germany. Eng. Pat. 20,818, November 2, 1893.

THE object of this invention is to prevent spontaneous ignition of liquid hydrocarbons, such as benzene, toluene, naphtha, gasoline, petroleum, &c., which is caused by frictional electricity produced on rinsing woollen or silk goods in the hydrocarbon. About 2 per cent. of anhydrous magnesium oleate, which is termed "antibenzene-pyrene," is dissolved in the hydrocarbon, and this solution is said to act antielectrically.—J. J. K.

*Improvements in the Production of and in Apparatus for Producing Artificial Silk.* F. Lehner, Zurich, Switzerland. Eng. Pat. 24,003, December 13, 1893.

IMPROVEMENTS are described in the production of artificial silk according to Eng. Pat. 11,831 of 1891 (this Journal, 1892, 680) and 22,736 of 1892 (this Journal, 1893, 1024). The solutions used frequently attack the metal of which the conducting tubes are composed, and this tends to discolor the product. This defect is obviated by the use of aluminium tubes which are not attacked by the solutions.

When, in drawing the filaments, the supply is interrupted by simply closing the tap, the orifice of the outlet tube often becomes choked and cannot be cleared. To avoid this a tight-fitting rubber cap is drawn over the aperture before closing the tap; the tube does not then become choked and the supply can be restarted immediately on removing the cap, even after many days' interruption.

The drying chambers for recovery of the solvents are omitted, and the solvents are recovered by heating the filaments on spools or in hanks, in water or other liquid to distil off the alcohol and ether. It is stated that this last improvement increases the tensile strength of the fibre.

—R. B. B.

*Improvements relating to Drying and Carbonizing Apparatus for Textile and other Matters.* G. Dasse and C. Fettweis, Brussels. Eng. Pat. 23,124, December 1, 1893.

A DRYING or carbonizing machine of any kind is enclosed in a chamber provided with a special aperture close to the entrance for the material. Through this aperture by means of a ventilator, the saturated hot air is drawn, passed through a heating apparatus, and thence again into the chamber. The air at the higher temperature is capable of taking up a further quantity of moisture, and the operation may be repeated by raising the temperature of the heater. As drying proceeds, the gaseous mixture in the machine contains less and less air, and the drying is performed by superheated vapour.

For wool the process may be advantageously carried out with a drying apparatus in which the wool passes between a number of superposed endless aprons. The wool is entered at the top and passes downwards, the outlet for the saturated gas being situated above the series of aprons.

—R. B. B.

*Improvements in Means for the Production of Waterproof Ventilating Cloth.* V. Hendrichs, Hückeswagen and Berlin, Germany. Eng. Pat. 12,954, July 4, 1894.

A SOLUTION is prepared containing 2,372.5 grms. of pure alum, 550 grms. lead acetate, and 400 grms. tannin, in 20 litres of distilled water. Lead sulphate is precipitated and the supernatant liquid, containing aluminium and potassium acetates and tannin, is carefully drawn off. Cloth steeped in this solution and allowed to dry is practically waterproof, but permits the free passage of air.—R. B. B.

## VI.—DYEING, CALICO PRINTING, PAPER-STAINING, AND BLEACHING.

*The Glossing of Woollen Crapes.* Edward Kopp. Bull. Soc. Ind. Mulhouse, June 1894, 266.

The unbleached woollen fabric is stirred by hand for five minutes in a wooden trough containing a solution of zinc sulphate heated to the boil. The zinc sulphate solution (200 litres) contains 500 grms. of the sulphate per litre of water and indicates 23° B. at the boiling point. The piece after this treatment is washed in another trough for five minutes with boiling water, and afterwards in cold, until the washings no longer show an acid reaction. It is then scoured and bleached in the usual way. Each piece is then found to have taken up some 7 kilos. of zinc sulphate. The crape thus obtained resists the various operations of bleaching very well, and only slightly "yellows" on steaming.—H. I.

*Zinc Ferrocyanide as a Fixing Agent for Aniline Colours upon Aniline Black.* Ferd. Oswald. Bull. Soc. Ind. Mulhouse, June 1894, 261—265.

The author obtains by double decomposition a precipitate of an insoluble ferro- or ferri-cyanide on the fibre and finds that aniline colours on fabrics thus prepared will stand treatment in a Mather and Platt machine very well, whereas if potassium ferrocyanide be used alone, the colours obtained are not fast to soaping.

The best results were obtained with zinc ferrocyanide and on aniline black as a ground.

The fabric is padded with aniline hydrochloride, potassium chlorate, and potassium ferrocyanide, and then printed with a thickened solution consisting of the aniline colour, 5 per cent. of zinc sulphate, and 20 per cent. of magnesium acetate (which acts as a resist for the aniline black).

With Methylene blue the quantities used were as follows:—

- 500 grms. of magnesium acetate of 20° B.
- 400 grms. of dry powdered starch.

heated together and then mixed in the cold with—

- 15 grms. of Methylene blue dissolved in
- 100 grms. of acetic acid of 6° B., and
- 60 grms. of zinc sulphate.

Good results were also obtained with Magenta and Safranine, Auramine, Thioflavine, Malachite green, the different violets, Rhodamine, &c. By admixture of the above colours all shades may be produced.—H. I.

*Coloured Discharges on Aniline Black Grounds.* W. E. Kay. Bull. Soc. Ind. Mulhouse, August and September 1894, Part II., 85.

This was a communication from the author, of the firm of Walter Crum and Co. (The Thornliebank Co., Glasgow), respecting the statement in the bulletin of May 7, 1894, that M. Camille Schoen had succeeded in effecting the fixation of basic coal-tar colours as reserves under aniline black in the presence of hydroferrocyanic acid alone, without the aid of tannin. Mr. Kay claimed priority of discovery of the process (this Journal, 1893, 1027) and forwarded patterns illustrating the process. The principal constituents of Kay's reserve are acetate of soda and acetate of zinc, the latter constituent being employed for the purpose of producing insoluble ferrocyanide of zinc, which, it is said, more effectually serves to fix the basic coal-tar colours (Methylene blue, Rhodamine, &c.) used. In certain cases the reserve also contains bisulphite of soda or acetate of chrome. Thiosulphates and sulphocyanides are less suitable as reserving agents. The procedure is the ordinary one, namely, padding of the black, drying, printing the reserve, and steaming. (See also this Journal, 1893, 41, 1027; 1894, 485).—W. E. K.

*Azo Colour Resists on Steam-printed Indigo (Schlieper Method).* L. Bloch and Ch. Schwartz. Bull. Soc. Ind. Mulhouse, June 1894, 260—263.

The authors recommend the use of sulphur as a resist in steam indigo printing.

*Direct Colours.*—The best results were obtained with the following colours:—Chrysamine R (Bayer) gives a yellow, Alkaline orange G (Dahl) a lemon yellow, Eriochrome (Berlin) a rose, Benzopurpurin 1 B (Bayer) a strawberry colour, all undergoing a change in shade.

The general method of application may be given as follows:—110 grms. of colour, 330 grms. of sodium phosphate, 115 grms. of soap, 3,300 grms. of powdered starch, and 8 litres of hot distilled water, made into a paste with 2,200 grms. of precipitated sulphur. This is printed on the fabric, which is afterwards dyed as usual. The colours obtained in this way are not moderately fixed; they resist a slight soaping sufficiently well, but are attacked by the sulphur in presence of soda, giving weaker shades. Chrysamine suffers the least.

*Azo Colours produced on the Fabric.*—The procedure consists in printing the thickened diazo compound mixed with precipitated sulphur on a fabric previously padded with sodium naphtholate and glucose (which are without action on the indigo), then dipping in the indigo vat, oxidising, and washing. Resist reds, yellows, and oranges of great brilliancy and fastness may be thus produced on indigo grounds. Most of the colours are nevertheless more or less changed in shade.

Xyldine red gives an orange; Para nitramine red is destroyed. The reds obtained from cumidine and  $\beta$ -naphthylamine, and the claret from  $\alpha$ -naphthylamine, do not suffer much loss in intensity.

The red from cumidine is the most brilliant; a red of better shade is obtained from a mixture of three parts of  $\beta$ -naphthylamine and one part of cumidine. Two parts xyldine and one part of  $\beta$ -naphthylamine likewise give a good red.

The colours obtained from the diazo-chloride are somewhat dull. This may be rectified by the addition of sodium acetate (40 grms. per litre of padding) to the padding mixture.

In the association of azo-colours with direct colours and whites, it is necessary to use for the latter a stronger resist than usual, since the fabric is padded with sodium naphtholate.

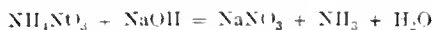
To prepare the padding mixture, the glucose and sodium naphtholate are dissolved and then mixed in the cold, in order to prevent decomposition of the glucose by the soda.

For the same reason, the pieces should not be raised to too high a temperature in drying. In fact, all the precautions that are usual with diazo-colours should be taken. The addition of "oil for red" to the padding mixture produces more vivid and darker blues and a more brilliant resist.—H. I.

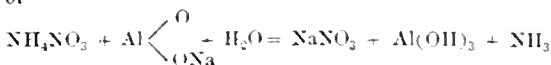
*The Employment of Ammoniacal Salts as Resists for Caustic Soda, Aluminate of Soda, &c.* E. Bontemps. Bull. Soc. Ind. Mulhouse, 1894, 285—286.

When, in printing, a resist is required for caustic soda, aluminate of soda, &c., it is possible to make use of an organic acid, e.g., tartaric or citric. But when these resists have to be applied on a ground of Turkey-red, or on a fabric mordanted with tannin and antimony, these acids are very unsuitable, as they, and also their sodium salts, partially dissolve the alizarin lake or the tannate of antimony.

This defect is not shared by ammonium salts, such as the nitrate, which form the best resists for these alkaline mordants. The reactions which occur are—



or—



In the latter case the metal is precipitated in the form of hydroxide, and is removed during clearing. As there is a



tendency for the resist to spread in the presence of moisture, the steam to which the fabric is exposed in the "Mather and Platt" should be well dried.

A good resist is made thus:—10,000 grms. of nitric acid at 37° B. are neutralised with 7,500 grms. ammonia (sp. gr. 0.92), and the compound poured with constant stirring on to 1,250 grms. of starch, mixed with 1,250 grms. water. The resist is then cooled and is ready for use. Care must always be taken that an excess of alkali is present to prevent the transformation of starch into glucose.—R. B. B.

*Printing Processes of the Japanese.* M. Tétaz. Bull. Soc. Ind. Mulhouse, 1894. Part II., 85.

M. Tétaz communicated to the Society an account, from personal observation, of the printing process employed by the natives of Japan. The process is based on the use of a resist paste prepared from rice, which is spread by means of a palette-knife on to a paper-stencil, and under this the tissue is placed. This reserve is successfully employed for vat blues, for which purpose the Japanese reduce the indigo by means of fermentation.—W. E. K.

*The Production of Fast Reds in Calico Prints without Mordant.* J. Dépierré. Bull. Soc. Ind. Mulhouse, 1894, 201—202.

In the Dutch East Indies the reds in the native calico prints or "batieks" are produced without the use of alumina or other metallic mordant. The fabric undergoes a lengthy preparation with oil, consisting of alternate steeping in an emulsion of sesame oil and alkali, and exposure to sun and air. This may be continued for about nine weeks. A wax resist is then applied by means of an apparatus shaped like a pipe, the bowl forming the reservoir, and the melted wax flowing from the orifice in the stem. The stem is moved about over the fabric to trace out the design, and the "batticker" is often provided with a number of these instruments, some with one orifice, others with two, three, or more.

The resist being applied, those portions of the fabric which are to be coloured are rubbed over with a paste made of 3 parts keedoe, 1 part djirack, and a sufficient quantity of water. This treatment is repeated nine times; clearing and washing with boiling water complete the production of the red.

*Keedoe* (this Journal, 1894, 940) is the powdered root-bark of *Morinda Citrifolia* or Sooranges (Schützenberger, *Traité des Matières Colorantes*, Vol. II., p. 419). The colouring matter of this root does not dye mordanted fibres in the ordinary manner. The colouring principle, morindin, gives a yellow precipitate with alum, a red precipitate with lime or baryta. Probably the lime present in the water plays the part of a mordant.

*Djirack* is an astringent product of the bark of *Symplocos fasciculata*, *S. racemosa*, and other varieties (Wardle, *Catalogue of Dyestuffs and Tannin Matters of India*, 1878).—R. B. B.

#### PATENTS.

*Process for Dyeing Textile Materials in Cops or Spools with Alizarin and other Mordant Dyes.* F. Erban, Hoechst a/Main, and L. Specht, Marienthal, Germany. Eng. Pat. 15,829, August 21, 1893.

THE insoluble mordant dyes have been hitherto considered unsuitable for cops and spools. They can, however, be applied in the form of alkaline solutions. The dyes applicable by this method are the anthracene colouring matters and other mordant dyes, such as gallein, coerulein, galloflavin, gambines, dioxine, &c., &c. The cops are impregnated with a solution of the colouring matter, together with an alkali, e.g., ammonia, sodium hydrate, carbonate, or phosphate, alkaline soap, Turkey-red oil, or an organic base (amine). The fugitive alkaline mediums have the advantage that in drying, they evaporate and leave the colouring matter insoluble on the fibre. Other substances which do not precipitate the colouring matter may be added to the dyebath, e.g., aluminate or stannate of soda, soap, &c. After impregnating the fibre the material is dried or

steamed, and then treated with an aluminium, chromium, iron, or other mordant, preferably in the form of acetate. The formation of the colour lake is completed by steaming; Washing, soaping, and clearing may increase the brightness of the dye. An example of the process is as follows:—

*To Dye Turkey-Red on Cops.*—First bath—

14 lb.  $\frac{1}{4}$  oz. Alizarin R. (20 per cent.)  
5 galls.  $\frac{1}{3}$  pints water (free from lime).  
 $2\frac{1}{2}$  pints ammonia (25 per cent.)  
 $7\frac{1}{2}$  pints Turkey-red oil (80 per cent.)

After impregnation the cops are subjected to centrifugal action and dried at 50° C., then treated with the second bath—

4.52 galls. water.  
 $5\frac{1}{2}$  pints acetate of alumina 15° Tw.  
3 pints acetate of lime 28° 4 Tw.

They are subjected to centrifugal action and steamed one hour without pressure,  $\frac{1}{2}$  hour at a pressure of one atmosphere, and  $\frac{1}{2}$  hour at a pressure of two atmospheres. After steaming, the colour can be soaped, or cleared with a mixture of soap, soda, and tin salt.—R. B. B.

*Improvements in or relating to the Clouding of Textile Materials in Dyeing.* A. J. Boulton, London. From P. V. Renard, Fontenay-s.-Bois, France. Eng. Pat. 22,498, November 23, 1893.

FLAT rings or perforated discs are held apart at fixed distances and in parallel position by suitable bracing. If the discs have a hub they are mounted on a shaft, with smaller rings interposed to keep them in position. The textile material, in the form of sheets or strips of fibres, or spun threads, is arranged in layers of as uniform thickness as possible round the periphery of the discs, and firmly bound to their edges by impermeable cords or ribbons. When the whole is immersed in the dyebath, those portions bound against the discs remain uncoloured, whilst the dye has free access to both sides of the remainder of the layer of material.—R. B. B.

*A Process for Marking Indelibly on Paper, Fabrics, and like Receptive Material.* C. F. Cross and E. J. Bevan, London. From C. Beadle, Boston, U.S.A. Eng. Pat. 6887, April 6, 1894.

THE material is moistened with water or with some other solution conducting electricity, and laid on a metal plate connected with one pole of an electric battery or generator. The other pole is connected to a metallic style or stamp. On writing with this style a current of electricity passes through the material, and an electrolytic action takes place by which portions of the metal or of the style are dissolved and deposited on the material in the form of the writing. If the deposit is not readily visible it may be treated with a suitable reagent; e.g., a deposit of copper or of iron may be developed with potassium ferrocyanide. Or, the paper may be moistened with a solution of the required reagent, or the style may be dipped in such a solution.

In some cases without solution of the metal some change is effected in the material, such that when moistened with water the marking is rendered visible. This occurs when a platinum style or stamp is used.

Paper or fabric may be marked in the same way by passing it in a moist condition between a pair of rollers, each of which is connected to one terminal of the battery.

—R. B. B.

*Improvements in Calico-printing Machinery.* M. MacPhail, Busby, Lanark. Eng. Pat. 17,885, September 20, 1894.

INSTEAD of supporting the colour-furnishing roller in bearings on its colour box, the spindle of the roller is extended, and its ends supported in bearings, capable of delicate horizontal and vertical adjustment, fixed to side brackets which are secured to the machine framing. The result is an increased steadiness and freedom from vibration of the roller.—R. B. B.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Solubility of Certain Inorganic Salts in Organic Liquids.*  
S. von Lascezyński. Ber. 1894, 27, 2285–2288.

100 parts by weight of ethylic ether dissolve 0.11 part of  $\text{CuCl}_2 + 2\text{Aq}$  at 16° C.; 6.35 parts and 6.38 parts of  $\text{HgCl}_2$  at 0° and 35.5° respectively; 0.97 part of  $\text{HgI}_2$  at 36°; and 11.41 parts and 11.38 parts of  $\text{SnCl}_2 + 2\text{Aq}$  at 0° and 35.5° respectively.

100 parts by weight of ethylic acetate dissolve 0.44 part and 0.2 part of  $\text{KCNS}$  at 0° and 79° respectively; 0.08 part and 0.26 part of  $\text{COCl}_2$  at 14° and 79° respectively; 35.53 parts and 73.44 parts of  $\text{SnCl}_2 + 2\text{Aq}$  at 22° and 82° respectively; 28.92 parts and 35.98 parts of  $\text{HgCl}_2$  at 0° and 83° respectively; and 1.56 parts and 4.36 parts of  $\text{HgI}_2$  at 17.5° and 76° respectively.

100 parts by weight of acetone dissolve 2.38 parts and 1.21 parts of  $\text{KI}$  at 22° and 56° respectively; 20.75 parts of  $\text{KCNS}$  at 23°; 4.60 parts and 2.14 parts of  $\text{LiCl}$  at 0° and 58° respectively; 9.28 parts of  $\text{CoCl}_2$  at 22.5°; 17.06 parts  $\text{CoCl}_2 + 2\text{Aq}$  at 25°; 1.40 parts of  $\text{CuCl}_2$  at 56°; 8.92 parts of  $\text{CuCl}_2 + 2\text{Aq}$  at 13.5°; 98.35 parts and 126.8 parts of  $\text{HgCl}_2$  at 0° and 18° respectively; 3.36 parts and 6.07 parts of  $\text{HgI}_2$  at 18° and 58° respectively; 0.35 part of  $\text{AgNO}_3$  at 14° and 59°; 0.02 part of  $\text{PbI}_2$  at 59°; and 41.70 parts of  $\text{Bi}(\text{NO}_3)_3 + 5\text{Aq}$  at 19°.

100 parts of amyllic alcohol dissolve 0.48 part and 3.15 parts of  $\text{KCNS}$  at 13° and 133.5° respectively; 0.66 part and 9.57 parts of  $\text{HgI}_2$  at 13° and 133.5° respectively; 0.02 part of  $\text{PbI}_2$  at 135.5°; 0.54 part and 1.80 parts of  $\text{HgCl}_2$  at 15° and 84° respectively; and 0.22 parts and 1.24 parts of  $\text{HgI}_2$  at 15° and 84° respectively; 100 parts by weight of aniline dissolve 0.50 part and 1.1 parts of  $\text{PbI}_2$  at 13° and 184° respectively.

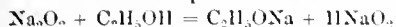
100 parts by weight of pyridine dissolve 0.26 part and 0.11 part of  $\text{KI}$  at 10° and 119° respectively; 6.75 parts and 3.21 parts of  $\text{KCNS}$  at 0° and 115° respectively; 7.78 parts and 14.26 parts of  $\text{LiCl}$  at 15° and 100° respectively; and 0.1 part and 8.6 parts of  $\text{AgI}$  at 10° and 121° respectively.—A. R. L.

*The Behaviour of Sodium Peroxide towards Alcohol.*  
J. Tafel. Ber. 27, 2297.

By pouring alcohol on sodium peroxide, about half of the latter goes into solution. This solution is strongly alkaline; on diluting with water and acidifying with sulphuric acid, it only very slightly reduces permanganate solution, showing that it contains only a trace of "active" oxygen.

The part insoluble in alcohol is a white, sandy, easily filtered powder, which differs materially from the original substance. This difference shows itself most distinctly in their behaviours respectively, when heated. Sodium peroxide may be heated to a high temperature without changing, and begins to give off oxygen only at a red heat; but this new substance liberates oxygen rapidly at a very low temperature. If heated quickly in an open tube, the oxygen is set free with explosive violence and ignition.

At a low temperature the new substance is changed into an orange yellow mass, which melts on further heating, giving off oxygen and a little water, leaving a residue which is chiefly sodium hydrate. Owing to the extreme liability of the substance to decompose at ordinary temperature, even in an atmosphere of carbon dioxide, the author had some difficulty in analysing it. The constitution he assigns to the substance is  $\text{HNaO}_2$ , and the equation showing the action of alcohol on sodium peroxide—



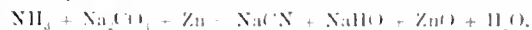
This new body is plainly identical with the hydrate obtained by the author by the action of alcoholic mineral acids on sodium peroxide (this Journal, 1894, 885).—H. M.

## PATENTS.

*Improvements in the Preparation of Cyanogen Compounds.*  
J. J. Hood and A. G. Salmon, London. Eng. Pat. 21,239, November 8, 1893.

As carbon bisulphide and ammonia may be caused to react to produce hydrogen sulphide and sulphocyanic acid, so the inventors find that a similar reaction takes place when carbonic acid is substituted for carbon bisulphide, the products being water and cyanic acid. The same analogy holds good with cyanides of the bases. Just, for instance, as sodium sulphocyanide heated with a reducing agent, such as zinc, gives sodium cyanide and zinc sulphide, so sodium cyanate, similarly treated, gives sodium cyanide and zinc oxide.

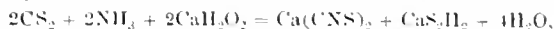
In applying these reactions to the production of cyanogen compounds, it is not necessary to use carbonic acid gas, for which a carbonate of the required base may be substituted, or two carbonated bases may be used. Thus, to obtain sodium cyanide, sodium carbonate or bicarbonate is incorporated with a reducing agent, as zinc, manganese, or lead, and the mixture is heated in a current of ammonia; the sodium cyanide formed is washed out from the zinc oxide, and is purified by suitable means. The reaction is thus expressed:—



As only half the base is, in this process, converted into cyanide, the reaction may advantageously be completed by passing carbonic acid over the heated mass simultaneously with the ammonia. Also, it is sometimes found better to employ zinc in conjunction with carbon, so that the former would reduce the oxy-cyanide to cyanide, and the latter in turn reduce the oxide of zinc formed, to the metallic state, thereby avoiding the action that takes place to a greater or less extent between the sodium or potassium hydrates and the zinc oxide. An oxide or carbonate of zinc, manganese, or lead, with a reducing agent, such as carbon, may be used instead of the metal.—E. S.

*Improvements in the Manufacture of Sulphocyanides, in the Recovery of By-Products, and in Apparatus therefor.*  
J. Brock, A. E. Hetherington, F. Hurter, and J. Raschen, Widnes. Eng. Pat. 21,451, November 10, 1893.

THE process consists in heating together 100 parts of carbon bisulphide, ammonia solution equal (preferably) to 45 parts of dry ammonia, and 200 parts of slaked lime, with water sufficient to admit of suitable agitation. The proportion of ammonia used, is double that required by the equation—



in order to obviate the formation of calcium sulphocarbonate.

The apparatus employed is a horizontal cast-iron or steel cylinder, jacketed for steam except at the ends, provided with an agitator, a pressure gauge, safety valve, sampling tap, a pipe for admission of the liquids, and an opening with lid for the sieved lime. In front of the short, wide pipe for receiving the lime, a plate is attached to the top and sides of the cylinder, reaching below the surface of the liquor, to prevent escape of ammonia when the lime is introduced. The top of the cylinder is connected by a pipe and valve to a cooling worm, and this to a pair of coke-charged condensers on which water or weak ammonia condensed from a former operation is sprayed. The safety valve is so arranged that in case the pressure rises too high, the escaping vapours pass to the ammonia condensers. The heating, as indicated by a permanently fixed thermometer, is carried at least to 100° C., and if the pressure is not too high, live steam may be admitted to the cylinder through a pipe provided with a back-pressure valve. When the reaction is complete, the valve admitting passage to the ammonia-condensers is opened, and live steam is passed in until any excess of ammonia is driven off and collected for future use. The charge is then transferred to a vacuum filter, and the precipitate is washed, the washings being used instead of water in the next operation.



Carbonic acid gas is forced through the filtrate, and the hydrogen sulphide set free, is collected and utilised. Or the gas may be passed through the liquor before filtration, in a suitable apparatus admitting of collection of the hydrogen sulphide in this case derived both from the soluble and insoluble calcium sulphides, and thus all the sulphur not contained in the sulphocyanide may be recovered. In either case the solution of calcium sulphocyanide obtained may be evaporated. But if it be desired to obtain the potassium or sodium salt, the solution is treated with the corresponding carbonate, and the calcium carbonate precipitated is separated. The solution of potassium or sodium sulphocyanide is then dried up or crystallised.—E. S.

*Improvements in obtaining Cyanides and Ammonia direct from the Air, or other Gases containing Nitrogen.* J. Young, Glasgow. Eng. Pat. 24,856, December 27, 1893.

Four parts of an alkali carbonate or oxide, are mixed with one part of the carbonate or oxide of an alkaline earth, and two parts of coke or charcoal, preferably paraffin coke, and the mixture is strongly heated in an iron or other suitable apparatus, through which air, or a mixture of air and a hydrocarbon gas or vapour from hydrocarbon oils or gases containing nitrogen, is passed. If the mixture is sufficiently heated to volatilise the alkali cyanide formed, the cooled gases containing the cyanide are passed through wool or cotton to retain the cyanide.

If it be desired to obtain ammonia, the heated mixture is allowed to cool a little after the cyanide is formed, and steam is then passed through the apparatus. The ammonia evolved is received in an acid to obtain an ammonium salt.—E. S.

*Improvements in or connected with the Manufacture of Bleaching Powder.* H. Gaskell, jun., Liverpool, V. C. Driffield, Widnes, A. Carey, Liverpool, and F. W. Wright, Widnes. Eng. Pat. 25,923, December 29, 1893.

THE inventors have in Eng. Pat. 1214, 1893 (this Journal, 1894, 38), described apparatus for cooling the chlorine used in the manufacture of bleaching powder, but find it desirable that it should be also dried. This is effected by connecting the successive absorption compartments to drying towers filled with coke, flints, or the like, through which, from lutes in the top, sulphuric acid is caused to trickle. The gas from one compartment enters the drying tower by a pipe near the bottom, and another pipe conveys the dried gas, first to a cooler, and then to the next absorption compartment, and so on, successively. Calcium chloride or other suitable hygroscopic substance may be used instead of sulphuric acid in drying the chlorine.—E. S.

*Improvements in or relating to the Manufacture of Chlorine.* H. Gaskell, jun., and A. Carey, Liverpool. Eng. Pat. 25,024, December 29, 1893.

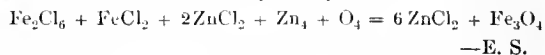
IN the process for manufacturing chlorine by decomposing hydrochloric acid with air in presence of a catalytic or contact substance, the inventors add to the materials containing such substance, a substance having superior affinity for the impurities (as sulphuric and arsenic acids) contained in the acid gas. In the Deacon process for the manufacture of chlorine, in which cupric chloride is used, the substance added may be calcium chloride or magnesium chloride, which may be dissolved in the cupric solution used to impregnate the porous body serving as a vehicle. When such vehicle is burnt clay or bricks, basic material—as calcium oxide, hydrate or carbonate, or magnesia, but preferably impure calcium carbonate resulting from the manufacture of caustic soda or from the "Chance" sulphur recovery process—is used, in proportion not exceeding 7 per cent., to mix with the clay before it is burnt for the purpose stated.—E. S.

*Preventing Gases from Intermingling while passing through Liquids.* J. B. Hilliard, Glasgow. Eng. Pat. 24, January 1, 1894.

CHANNELS, preferably semi-circular in section, are placed in an inclined position in a tank containing a liquid, the lower end communicating with an entrance pipe and the higher end with an exit pipe for the gas to be passed. The channels are arranged in sets, at different elevations, and are shown with three channels to each set. As each set has a different entrance and exit, the gases travelling the higher or lower set do not mingle. The channels may be permanently fixed in the tanks, or supported by floats. The top of the tank may be inclined, so as to act as channel for one gas, while the other gases pass along other submerged channels. The invention is stated to be particularly applicable in the making of chlorine gas, "as it allows the chemist to utilise the oxygen in the atmosphere during the process without allowing the other constituents of the atmosphere to mix with and spoil or adulterate the chlorine gas."—E. S.

*Process for the Separation and Precipitation of Salts of Iron and Zinc from Solutions containing both.* A. J. Boulton, London. From E. Warzee, Brussels, Belgium. Eng. Pat. 4916, March 8, 1894.

TO a solution containing, for instance, ferric chloride, ferrous chloride, and zinc chloride, in the proportions  $\text{Fe}_2\text{Cl}_6 + \text{FeCl}_2 + 2\text{ZnCl}_2 + \text{aq.}$ , just so much of the more electro-positive metal, zinc, as corresponds to the electro-positive salt, is added. Ferrous-ferric oxide is precipitated, and the solution then contains essentially zinc chloride, contaminated, however, by a certain proportion of ferrous chloride, as is indicated by a green coloration of the liquid. Zinc in excess is then added, and the solution is boiled, whilst being subjected to a strong air-current blown into it, the process being continued for 12 to 20 hours. The green coloration then disappears, and the zinc chloride is found to be freed from iron salts. The process is represented as to its termination by the equation—



*Improvements in the Preparation and Manufacture of Hydrosulphurous Acid and Hydrosulphites.* J. Grossmann, Manchester. Eng. Pat. 16,968, August 23, 1894.

HYDROSULPHUROUS acid, or a hydrosulphite, is prepared from monosulphites or metasilphites by acting upon them in solution or suspension in water, with graduated additions of 10 per cent. sulphuric acid, followed by agitation with zinc. For example, sodium mono- or metasilphite, dissolved in sufficient water to give a solution containing from 15 to 25 per cent. of total sulphurous acid, is placed in a suitable vessel fitted with an agitator, and a pipe going to nearly the bottom of the vessel for introduction of the dilute sulphuric acid. So much acid is added, little by little, followed by gentle agitation, as may liberate from one-half to nearly all the sulphurous acid present, the temperature being kept as low as possible. When the reaction is completed, the necessary quantity of zinc or other suitable reducing agent is added with agitation. A series of tests is made to determine the proportion of sulphuric acid to be added, the final hydrosulphite being tested by Berntsen's test as modified by Rawson, or by any other suitable test. Instead of an acid, an acid salt, such as sodium hydrogen sulphate, may be used. The claim covers the use of bisulphites in the described process, as well as mono- or metasilphites or mixtures of these.—E. S.

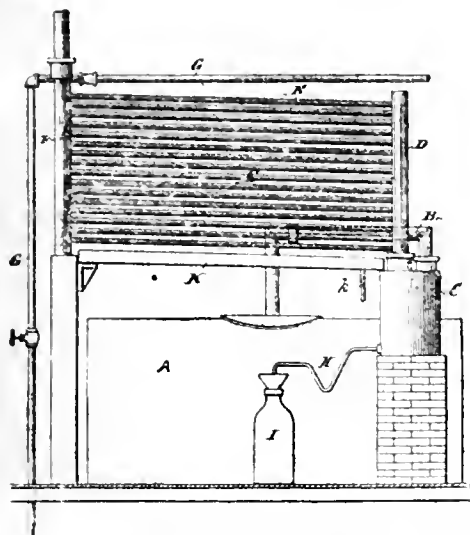
*Improvements in Salt Evaporators.* T. Craney, Bay City, Michigan, U.S.A. Eng. Pat. 17,275, September 11, 1894.

AN upright cylindrical vessel, with ends of conical shape, is fitted with steam pipes for heating the saline liquid contained therein. The heavy crystals as they form, fall down a vertical leg, forming the lower outlet of the vessel, and from thence are removed by means of a conveyer or chute,

while the vapour formed is carried over by a goose neck, and condensed by means of water circulating in a ring pipe upon the goose neck. The condensed water is discharged by means of a pipe depending from the goose neck and dipping under water below, so as to form a water seal. Water is conveyed to the condensing ring by means of a supply pipe in connection with a centrifugal or other pump.—E. G. C.

**Improved Apparatus for the Condensation of Nitric Acid and other Vapours.** H. E. Newton, London. From E. Hart, Easton, U.S.A. Eng. Pat. 17,289, September 11, 1894.

The apparatus has a series of pipes arranged one above the other, preferably slightly inclined, in combination with a retort and receiver, whereby the vapour is divided on its path, and condensed by the cooling effect of water or other liquid sprinkled upon the pipes.



In the above drawing the vapour passes from the retort A through the pipe B into the vessel C, and thence through the vertical pipe D into the series of slightly inclined tubes E, having exit through the vertical pipe F. The supply of water through the pipe G, perforated or slotted above the condensing pipes, may be so regulated as to completely evaporate on these pipes; or there may be a surplus, carried off by the trough K and pipe k. The liquid condensed in the pipes E flows into the vessel C, and is thence conveyed by the siphon H into the receiver I, which, in the case of nitric acid, it reaches boiling hot, so that "no red oxides of nitrogen or chlorine shall be dissolved in it." The apparatus may be varied in some respects, as by arranging the tubes E horizontally, or inclined towards the pipe F, in which latter case that pipe must communicate with the vessel C. The pipes E are preferably of glass, and the vessel C and the vertical pipes of stoneware, glass, or other acid-resisting material. Although the apparatus has been described in connection with the preparation of nitric acid, it is applicable also to the condensation of other similar vapours.—E. S.

**Improvements in the Manufacture of Sodium Diborate or Borax.** J. Ascough, Birmingham. Eng. Pat. 17,556, September 15, 1894.

Caustic soda is stirred in a circular pan, to which heat is applied, with a stated proportion of water, and dissolved, when boracic acid is added by degrees, the agitation being maintained till the combination is complete; the liquid is then evaporated at 100° C. until a trial sample on being stirred while cooling is in a granular condition. The source of heat is then withdrawn, agitation being maintained in a circular direction until the mass is granulated. If the borax

is to be produced in a semi-pulverulent state, the agitation is more vigorous and longer continued. The proportions of the materials, estimated as if pure, and proportionably if impure, are caustic soda 20 parts, boric acid 62 parts, and water 120 parts. The proportion of water used is larger than is required by the equation—



in order to facilitate combination. Part of the caustic soda may be replaced, in equivalent proportion, by dry sodium carbonate, crystal sodium carbonate, or by both, and quantitative examples are given.—E. S.

## VIII.—GLASS, POTTERY, AND ENAMELS.

**R. Engelmann's Process for the Decoration of Glass.** L. Appert. Bull. Soc. d'Encouragement, 9, August 1894, 472—474.

The inventor has adapted the ordinary method used in applying colours to pottery to the case of glass. The colours are transferred from prepared paper on to the glass and are made to adhere firmly by the use of linseed oil which has become oxidised to the consistency of caoutchouc. The paper is removed by soaking for some hours in water. The principal difficulty to be overcome in the process is due to the liability of the fatty and resinous matter to spoil the surface of the colour by cracking or blistering during carbonisation. To avoid this the temperature must be raised gradually, maintained steadily, and lowered slowly. This is effected by the use of a long chamber heated by gas burners arranged at right angles to the axis of the chamber, the articles being slowly moved throughout the length of the chamber by mechanism actuated by revolving cylinders. As the articles approach the centre they are gradually exposed to higher temperatures, and as they pass the centre the temperatures decrease at the same rate. In this way glass articles of large dimensions can be decorated in colour without risk of breakage.—V. C.

## PATENTS.

**Improvements relating to the Manufacture of Pottery.** J. E. G. Méran, Paris. Eng. Pat. 24,283, December 16, 1893.

The invention consists in the employment of minerals containing magnesia as a basis for the ware, e.g., magnesite, tale, steatite, or serpentine. The material is very refractory at high temperatures, and in the porous or biscuit state is especially suitable for electrolytic purposes, being unattacked by acids. Binding material, such as clay, may have to be added to the magnesia paste, but should be in as small proportion as possible and not exceeding 10 per cent. The baking is carried out at about 1,200° C. If done at 1,800° C. a porcelain-like material, which also has industrial applications, is obtained.—V. C.

**Improvements in the Manufacture of Pottery or Earthenware.** Worcester Royal Porcelain Company, Lim., E. P. Evans, A. Rushton, and H. Hawker, Worcester. Eng. Pat. 24,682, December 22, 1893.

This process is designed as an improvement upon the old method of employing modelled pieces of clay to give the necessary impress of a design in the mould in which "ware" is to be shaped. In carrying out the invention "raised colours" mixed in the ordinary way are printed upon blocks or moulds, from which reverse moulds in plastic material are then formed. In these reversed moulds the ware is formed as usual.—V. C.

*Improvements in the Manufacture of Enamel Glass Letters and other Devices.* E. Böhm, London. Eng. Pat. 878, January 15, 1894.

THE enamel glass letters are formed by direct pressure, thereby avoiding the employment of the metallic backing, which has hitherto been generally used and which frequently becomes unfastened. Molten glass is poured into a mould of the required shape, which is made in two pieces. The mould is then heated and subjected to strong pressure, the result being that each letter is stamped out. The letter or device can then be attached to windows, sign tablets, and the like.—V. C.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*The Effect of Stress on the Corrosion of Metals.*  
T. Andrews. Proc. Inst. C.E. 118, IV.

See under X., page 1200.

### PATENTS.

*Improved Process for Transforming Roasted Cements into Powder and in Apparatus therefor.* D. Belloe, and E. Benard, Paris. Eng. Pat. 2247, February 1, 1894.

CEMENTS which have been roasted to exactly the proper extent disintegrate rapidly, upon cooling, into a fine powder. Those parts which are insufficiently roasted disintegrate more slowly, and those which have been over-roasted do not disintegrate, but remain in lumps. In this invention the automatic disintegration is utilised, both for obtaining a fine powder composed of the properly-roasted cement, and for separating the properly-roasted cement from the remainder by "screening." If the screening be done without delay the under-burnt may subsequently be separated from the over-burnt cement by a second screening. The regular and uniform roasting of the materials is secured by the use of ovens in which combustible gases circulate through the charge, and in which the raw material, in lumps or powder, is incorporated with comminuted fuel. The subsequent cooling is allowed to proceed in a slow and regular manner.—V. C.

*Improvements in the Utilisation of Precipitated Sewage Matters for the Manufacture of Cement.* W. Bruch, Wiesbaden, Germany. Eng. Pat. 12,350, June 26, 1894.

THE precipitate obtained by the addition of lime to sewage is dried and heated to redness, when, among other things, "a basic salt of lime with hydraulic properties, is formed from the phosphoric lime, the carbonic lime, the caustic lime, and the organic substances."

The inventor claims that this compound, possessing hydraulic qualities, may be employed for preparing cement for use in the construction of subaqueous works.—L. de K.

## X.—METALLURGY.

*A Method of Treating Auriferous Ores with Bromine, with Regeneration of the Bromine Employed.* C. Lossen. Ber. 1894, 27, 2726—2727.

THE use of bromine in treating gold ores, mainly as a substitute for chlorine, has generally been abandoned after trial, on account of the loss of bromine, which amounts to at least 1.5 lb. per ton. The author proposes to regenerate the bromine electrolytically. After passing a current through a solution of potassium bromide, the resulting alkaline solution contains no free bromine, but is capable of dissolving gold. The ore, either raw or roasted, is treated with this alkaline brominated solution in a rotating cylinder until the gold is dissolved; more of the solution is added if the liquid be not still alkaline. The solution is then filtered to separate the hydroxides of iron and other metals; and the gold, which remains in solution as aurate, is reduced by passing the filtrate through vessels filled with fragments of iron and charcoal, or coke. The solution, now consisting mainly of potassium bromide, is run into troughs, where it is re-electrolysed and thus fitted for the treatment of a fresh batch of ore.—W. G. M.

*Composition of Newly-discovered and Ancient Copper from Chaldea and Egypt.* M. Berthelot. Bull. Soc. Chim. 1894, 11, 859—863.

THE author adduces the following analyses of ancient metallic objects in support of the theory that an age of pure copper preceded that of bronze, the proof of which is much obscured by the loose nomenclature adopted by antiquarians in regard to the composition of such specimens. All the objects described were much altered by exposure to salt water. No. 1 was from a small votive figure found in the foundation of a Chaldean house of the 40th century B.C. (estimated), and was almost entirely converted into oxychloride and a mixture of metallic copper and cuprous oxide; it contained no tin, and must originally have been pure copper. No. 2 was from a vase found in a tomb on the site of ancient Memphis, and is referred to the time of Seneferu of the fourth Dynasty. This also was oxidised through and partially chloridised, and gave no evidence of tin. No. 3 was from a bracelet or anklet, found in the same tomb as No. 2, but in such a position that it may have been left there by ancient depredators, and cannot therefore be accepted as evidence that bronze was known in the reign of Seneferu.

	No. 1. Chaldean.	No. 2.* Egyptian.	No. 3 Egyptian.
Copper.....	77.7	79.9	76.2
Tin.....	..	..	8.2
Lead.....	Trace	..	5.7
Oxygen as CuO.....	6.1	4.2	9.4
"    Cu <sub>2</sub> O.....		6.1	
Chlorine as oxychloride	1.1	6.2	
Water.....	3.9	6.3	
Sulphur.....	Trace	0.15	
Arsenic.....	Trace	..	
SiO <sub>2</sub> , CaCO <sub>3</sub> , Al <sub>2</sub> O <sub>3</sub> , &c.	11.2	5.15	

\* These numbers add up to 108, as in the original.

—W. G. M.

*Manganese Steel.* H. Le Chatelier. Comptes rend. 1894, 119, 272—274.

THE author recapitulates the properties of manganese steel (containing 13 per cent. Mn), which are of importance in connection with the present investigation. The alloy is non-magnetic and has the highest electrical resistance

(about 1 ohm for a wire 1 m. long and 1 mm. in diameter) of all the alloys of iron. It becomes softer on heating and quenching.

Hadfield has lately discovered the existence of an allotropic modification of this alloy, which is magnetic, this variety having been obtained by annealing the alloy at a high temperature for about 10 days. The author has defined the conditions governing this transformation. The transformation of the non-magnetic into the magnetic variety is effected by annealing it at 500—650 °C., the operation being complete in one or two hours, when the most favourable temperature (550 °C.) is adopted. To reverse the change the alloy is heated to a temperature not lower than 800 °C., and cooled rapidly enough to prevent alteration to the magnetic variety, occurring between 500 and 600 °C. Seeing, however, that the change is slow, a correspondingly moderate rate of cooling is sufficient to prevent the change. In this respect manganese steel is analogous to certain "self-hardening" steels containing chromium or tungsten, which harden on being allowed to cool spontaneously and need no quenching. The sole difference is that in the case of the last two materials, the transformation prevented by tempering, is one into an iron carbide, whilst in the case of manganese steel it is the allotropic change of the non-magnetic to the magnetic alloy which is impeded.

The following tables give the variation of the electrical resistance and the dilatation with alteration of temperature, for the two allotropic modifications of manganese steel:—

*Electrical Resistance of a Wire 1 m. in Length and 1.4 mm. in Diameter.*

Temp. °C.	15.	90.	300.	500.	635.
Resistance in ohms—					
(1.) Non-magnetic.....	1.06	1.19	1.44	1.65	..
(2.) Magnetic.....	0.88	0.99	1.27	1.50	1.70

Temp. °C.	730.	850.	965.	1020.
Resistance in ohms—				
(1.) Non-magnetic.....	..	1.88	..	1.97
(2.) Magnetic.....	1.79	..	1.93	1.97

*Longitudinal Expansion of a Rod 100 mm. in Length.*

Temp. °C.	250.	500.	680.	830.	960.	1060.
Expansion mm. (both kinds)	0.35	0.67	1.05	1.43	1.97	2.00

The two curves of electrical resistance meet at about 740° C. and become identical for higher temperatures. This temperature is that at which pure iron passes from the magnetic to the non-magnetic state. This leads to the hypothesis that manganese steel is a mechanical mixture of iron and a definite compound of iron and manganese, which would not be magnetic. The iron itself would undergo its ordinary change by rise of temperature, but the rate of change would be retarded by the presence of the compound of iron and manganese which is mixed with it. The expansion of the two modifications of manganese steel has been found to be identical, which fact excludes the possibility of a change of dimensions at the moment of transformation. Manganese steel quenched in water undergoes on annealing a contraction of 0.4 mm. on a length of 100 mm.—B. B.

*The Impurities of Commercial Aluminium.* H. Moissan. Comptes rend. 119, 1894, 12—15.

ALUMINIUM produced by electrolytic processes is always impure and variable in composition. Hitherto only two impurities have been generally recognised, namely, iron and silicon. The former has an unfavourable influence on the

physical properties of aluminium, but its presence may be avoided by due care in the choice of the raw material, crucibles, and electrodes. Silicon, being chiefly derived from the alumina employed, is more difficult to eliminate. For many applications of aluminium the presence of this impurity is of no moment; but, according to the author, its amount may be readily reduced, by fusing the metal under a layer of an alkaline fluoride. In addition to the above, the author finds that the aluminium of commerce always contains nitrogen and carbon. The former probably exists as a nitride dissolved in the excess of aluminium, the breaking strain and elongation of which it appears to reduce considerably. The nitrogen may be detected by dissolving the sample in 10 per cent. pure potash solution and passing the gases evolved into Nessler's reagent. Carbon may be separated by dissolving aluminium in hydrochloric acid, when it is left behind in the form of a maroon-coloured light powder quite free from graphite. For quantitative purposes potash is the best solvent, the residual carbon being finally estimated by combustion in the usual way. In this manner the author has obtained the following values:—0.104, 0.108, 0.080 per cent. of carbon. As regards the effect of carbon on the physical properties of aluminium, the following figures are given:—

	Limit of Elasticity.	Breaking Strain.	Elongation.
	Kilos. per Sq. mm.	Kilos. per Sq. mm.	Per Cent.
Aluminium of good quality..	..	11.1	9
The same, carburetted and cast.	..	8.6 to 9.5	3 to 5
The same, carburetted and rolled.	20	20.8	2.5
The same, carburetted, rolled, and annealed.	7.7	13.8	26.5

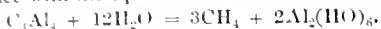
—H. T. P.

*Preparation of a Crystalline Carbide of Aluminium.* H. Moissan. Comptes rend. 119, 1894, 16—20.

THE electrical furnace described by the author (Comptes rend. 117, 679) may be conveniently employed. A number of carbon cells, each charged with 15—20 grms. of aluminium, are placed in the carbon tube of the furnace and submitted to the action of a current of 300 amperes at 65 volts, hydrogen being passed through the tube during the operation, and until the apparatus has become cool. The greyish fused mass formed in the cells when fractured will be found to be interspersed with brilliant yellow crystals of the new compound. Similar crystals are obtained by electrolysis a mixture of kaolin and carbon. They are best separated from unaltered aluminium by repeatedly treating the melt, 2—3 grms. at a time, with strong hydrochloric acid in a test tube kept cool by means of ice. When hydrogen is no longer disengaged, the crystals are rapidly washed with cold water, followed by alcohol, and finally ether. Thus obtained, aluminium carbide forms fine, yellow, transparent crystals, occasionally 5—6 mm. in diameter, and hexagonal in shape, having a density of 2.36, and the composition expressed by the formula  $C_3Al_4$ .

Heated to a dull red heat in an atmosphere of chlorine, the carbide is decomposed with incandescence, aluminium chloride is formed, and carbon liberated, the latter being amorphous and lamellated in character, and retaining the form of the original crystals. At temperatures above 700° C., bromine has a similar influence, whilst iodine is without action. Under the same conditions oxygen effects only a slight superficial change; but sulphur violently attacks the crystals, much heat being evolved, and aluminium sulphide and traces of carbon bisulphide produced. The carbide is completely oxidised with more or less violence when heated in contact with oxidising agents, such as potassium permanganate or bichromate, oxides of lead, &c. Potassium nitrate and chlorate, however, form exceptions. Strong acids in general have little or no action (boiling sulphuric acid is decomposed with evolution of sulphur dioxide); whilst weak acids dissolve the compound. The

most characteristic reaction, however, is that with water; methane is slowly formed in quantitative amount in accordance with the equation—



Under similar circumstances the carbides of the alkaline earths— $\text{M}''\text{C}_2$ —give off pure acetylene.—H. T. P.

*A New Method of Producing Metallic Deposits, especially adapted to the Plating of Aluminium.* C. Göttig. Ber. 1894, 27, 1824—1826.

THE difficulty in obtaining adhesive deposits of most metals upon aluminium, whether by simple immersion or by using a separate current, is well known, and appears to be dependent to some extent upon the nature and quantities of the impurities in the aluminium. The author finds that he can obtain a good deposit of certain metals by immersing the aluminium in such a solution of the metal as will not deposit by simple immersion, and rubbing the surface of the aluminium with a brush made of wires of a more electro-positive metal; and that the deposit is most rapid when the aluminium is relatively most electro-negative. Thus copper is deposited from a copper sulphate solution by rubbing the aluminium with tinfoil, but not with platinum; or tin from an ammonium stannic chloride solution by rubbing with brass wire, but not with copper. The deposit of copper may be thickened by immersing the thinly-coated aluminium in a dilute copper bath. Rubbing the copper solution on to aluminium with red-lead powder does not produce a satisfactory result, whilst with whiting a deposit is obtained instantaneously; the latter material, however, fails to give a deposit of tin from its ordinary solutions. The author intends to apply the process to other metals besides aluminium.—W. G. M.

*The Effect of Stress on the Corrosion of Metals.* T. Andrews. Proc. Inst. Civil Eng. 118, 1V.

THE author investigated the influence of stress upon the corrosion of metals, with the view of determining to what extent the alteration of rate of corrosion thus induced might account for the local attack sometimes observed in metallic structures. The metals used were wrought-iron bars and hammered shafts, and Bessemer and Siemens steel forged shafts. Soft Bessemer steel, hard Bessemer steel, Siemens steel, both soft and hard, soft and hard cast steel, and special steels, e.g., those containing Al, Ni, Cr, Si, and Cu, were also employed. The metals were in the form of bars  $3\frac{1}{2}$ — $3\frac{3}{4}$  in. in diameter or shafts  $4\frac{3}{8}$ — $5\frac{1}{2}$  in.; the plates were  $\frac{3}{16}$  in. in thickness. Full tables of the mechanical properties and chemical composition of the test pieces are given in the appendix to the paper. The method of experiment adopted, consisted in preparing ordinary test pieces, subjecting them to considerable stress in the testing machine, but not necessarily to the point of fracture, and turning from the stressed and elongated specimen, two similar pieces, one of which was from the turned-down centre of the bar where most of the stress had taken effect, and the other from the thicker end, which had been gripped in the jaws of the testing machine and had not been stressed to any considerable degree, the maximum stress being well below the yield point. The two similar pieces were placed in an electrolyte, usually a solution of common salt, and the difference of potential between them taken as a measure of their relative corrodibility. Precisely similar arrangements were used in those cases in which the stress was torsional or flexional instead of tensile.

The results of the whole of the series of experiments were nearly identical; they may be summarised thus:—

(1.) The effect of tensile stress was to cause the production of an average electromotive force of 0.016 volt between the "strained" and "unstrained" portions of the metal, the unstrained electrode being positive, and therefore considered by the author to be more corrodible.

(2.) The effect of torsional strain was to produce an average electromotive force between the electrodes of 0.018 volt, the "unstrained" metal being again electro-positive.

(3.) Flexional strain on plates gave an average of 0.012 volt in the same direction.

(4.) Flexional strain on shafts gave an average of 0.010 volt.

The author has drawn the following conclusion from his recent work on the corrosion of metals. Wrought iron, when exposed to the corrosive action of sea-water for long periods without being subjected to other causes of corrosion than such as are local, is more resistant than steel, the difference sometimes amounting to as much as 25 per cent. The more carbon present in the steel the greater the corrosion. Galvanic corrosion may be set up in metallic structures exposed to tidal flow, by reason of the local variations of the electrolyte (sea-water) in contact with them. The electromotive force existing between strained and unstrained metal in the same structure may induce local corrosion.—B. B.

*Hard Solders for Brass.* R. Schwirkus. Dingl. Polyt. J. 293, 64—67 and 89—93.

ORDINARY commercial hard solder is generally either too infusible or too brittle to be used for soldering commercial brass hammered-ware, and, as the composition of the latter is very variable, there is no solder in the market which may be safely applied indiscriminately. Exhaustive experiments have been tried with various soldering mixtures, under six heads, which are dealt with separately, as follows:—

1. *Influence on the Method of Alloying.*—The usual solder consists of copper and zinc, with or without the addition of other metals to promote fusibility. The common method of adding the zinc to the molten copper causes a loss of the former, which may amount to 25 per cent. of the zinc used, if small quantities only are being handled; and the use of brass instead of copper is scarcely an improvement. This variable loss of zinc leads to irregularity both in composition and fusibility. The method recommended by the author practically overcomes this difficulty: he heats the zinc to a temperature just above its fusing point, and then, little by little, and with constant stirring, introduces granulated copper in the form of fine grains (passing a 1.5 mm. sieve), no further quantity of copper being added until the previous addition is quite dissolved; the temperature is raised gradually as the melting point of the alloy rises. With the copper is always added one-third of its bulk of powdered sal-ammoniac, and a further quantity of the salt is introduced before pouring; for this reason the melting should be effected under a hood with a good draught to carry off the fumes. A third metal, if fusible, is added initially to the zinc (silver is introduced in the form of this strip with the copper). Brass in equivalent quantity may be substituted for copper, and is introduced in the same way; but its use is not to be recommended by reason of the incertainty of its composition. When traces of iron are to be excluded, pure materials must be used, and the stirring must be effected with wood or a clay pipe stem, the fusion being made in a non-ferruginous vessel.

2. *The Fusibility of the Solder.*—To estimate this, the author has determined the point at which the granules run together (the *flowing-point*). He places a mixture of 0.5 gm. of the solder and 0.3 gm. of powdered borax, moistened with two drops of water, in a small flat copper vessel 12 mm. in diameter, provided with a handle; the mixture is heated until the last trace of the dull white appearance of the borax is just vanishing; a too prolonged heating here causes oxidation, while insufficient heating leads to an incorrect result afterwards. The mixture is then cooled completely and placed in a constant predetermined position in a gas-blast flame maintained by regulators at a constant temperature. The exact time required to produce union of the grains is determined in half-seconds with the aid of a metronome. Five determinations were made with each mixture, and a maximum variation of 1.5 seconds in any series was held to invalidate the five trials, which were then started anew. The practical importance of a bold and rapid heating of the solder mixture is great; if heated for long to the fusing point of borax, but short of that of the solder (as by an inexperienced workman afraid

of burning his "work"), the borax runs off the corners of the solder particles, which then oxidise and may so far prevent subsequent union that the brass work to be joined is fused first. For this reason heavy work, which rapidly conducts the heat away from the intended joint, is more liable to be burnt than light work, and therefore demands the use of a more fusible solder.

3. *Estimation of Malleability.*—Tubes of brass or copper 100 mm. long by 35 mm. diameter, and 1 mm. thick, were made up from sheet metal with the solder to be tested, by means of a butt joint. One end of the tube was then carefully worked out under the hammer into a flange 7.5 mm. broad at right angles to the tube, so that the solder was submitted to a combined hammering and tensile test. If the joint gave way at once, the solder was considered non-malleable and marked 0; if the flange had turned over to an angle of 45°—60° before rupture occurred, it was marked  $\frac{1}{2}$ ; if only when brought to a right angle, it was marked 1. But if even then it remained sound, the flange was cut off, and another 10 mm. wide, was started in the same way; if this gave way at once, or between 45° and 60°, or when just complete, the solders were marked respectively 1,  $1\frac{1}{2}$ , or 2, as before. If still unbroken, the second flange was also cut off and a third was made 12.5 mm. wide; if necessary other flanges, each 2.5 mm. wider than the last, were similarly made, until at last the joint ruptured. In this way 8 degrees of malleability, corresponding to flanges from 7.5 to 25 mm. wide were obtained. Test-bars, 100 mm. long by 10 mm. diameter, were cast from the solder itself. Non-malleable solders may sometimes have to be used, but their tenacity should never be less than 2 kilos. per sq. mm. (= 1.27 tons per sq. in.).

4. *Use of Hard Solders for Various Qualities of Brass.*—Brass samples supplied by nine different makers were tried with brass and zinc, and copper and zinc solders. The flowing points of the brasses themselves were reached after from 16.8 to 23 half-seconds; and the only solders which could with safety be applied to all, were those containing 77 or less per cent. of brass (to 23 of zinc) or 46 or less per cent. of copper (to 51 of zinc), the flowing points of each of these solders being 14.8 or lower. A solder containing 51 per cent. of copper could just be used for eight of the samples, but the brass was in each case on the verge of destruction.

5. *Simultaneous Lowering of the Fusing Point and Increase in Malleability.*—By comparing Nos. 1, 2, 3, and 4 with Nos. 16, 18, 19, and 20 respectively in the annexed table, it will be seen that the substitution of silver for a small proportion of the copper is accompanied by a very marked increase both in fusibility and in malleability. Nos. 18 and 19 worked well with all the brass samples above alluded to, and No. 17 could just be used with extreme care.

6. *The Influence of an Addition of Tin upon the Malleability of the Solder.*—Nos. 26 to 32 in the table show clearly that even a small percentage of tin decreases the malleability to a greater extent than it lowers the fusing point of the solder, and the addition of this metal is not to be recommended.

In the table (page 1202) are included the results of the various tests made with alloys of the pure metals. (In the original table tests of 20 other solders made up with "brass" are included; but these are here omitted on account of the uncertainty of their composition.)

The author lays down 14 half-seconds as the high limit for the flowing point, and grade 2 (= flange of 10 mm.) as the low limit for the malleability of a generally useful solder for brass. Excluding those alloys which do not fall within these limits, it will be seen that only the solders containing silver are to be recommended for workshop use. Nos. 16 to 19 can well be used for soldering copper, but Nos. 19 to 22 are so trustworthy for all kinds of brass, and behave so well under the hammer, that the slight extra cost due to the addition of silver is more than compensated for. It is noteworthy that the silver solder, No. 23, containing 62 per cent. of the precious metal, is distinctly inferior to No. 22, containing only 12 per cent. No. 19 is especially recommended for the first soldering of brass, No. 20 for sheet and

wire, No. 21 for second solderings (on pieces which have been already soldered with a less fusible alloy) and for ordinary use, and No. 22 both for third solderings and as a substitute for fusible solders containing tin.—W. G. M.

*The Testing of Zinc Ores and Products of Manufacture.* F. Meyer. Zeits. angew. Chem. 1894, 394.

See under XXIII., page 1226.

*Separation and Estimation of Tin and of Antimony in Alloys.* Mengin. Comptes rend. 1894, 119, 224.

See under XXIII., page 1224.

## PATENTS.

*Improvements in the Manufacture and Purification of Iron, and in Means or Apparatus employed thereon.* W. J. Chapp and S. D. Williams, Newport. Eng. Pat. 20,450, October 30, 1893.

The lime or limestone required for fluxing is introduced into the furnace in the ordinary way, but the excess required for eliminating the sulphur is blown in through the tuyères in an extremely fine state of division.

Or, the lime (or limestone) is volatilised by the electric arc, and the "calcigen" thus produced is blown into the molten metal. Liquid fuel is also blown in when required. A specially-constructed revolving tuyère, carrying radial arms with channels or blades, and shaped screw or propeller fashion, dips into a trough or container holding the supply of pulverised lime. It is thus scooped up and delivered at an orifice, where it meets the blast and is carried into the furnace.

The furnace is lined with a flux of borax and chloride of sodium, or with bauxite, clay, silicate of soda, or potash, or a suitable mixture of these, thus setting up a hard, firm, glazed surface.—J. H. C.

*Improvements in Coating Iron, Steel, or other Metallic Plates with Tin, Terne, and other Mixtures.* F. Watkins, Llanelly. Eng. Pat. 23,048, December 1, 1893.

The plates having been "pickled," are passed through hard paraffin or ozokerite, or a mixture of the two, then into the tin, terne, or other metallic bath, and finally through palm-oil or palm-oil mixture.—J. H. C.

*Process for the Elimination and Recovery of Arsenic from Copper Precipitates.* E. G. N. North, Rio Tinto, Spain. Eng. Pat. 23,770, December 9, 1893.

AFTER being washed and dried, the precipitate is heated to a temperature somewhat below redness, in a closed muffle, which is provided with a small outlet-pipe for the escape of arsenical fumes. Stirring is effected without allowing air to enter, by means of a poker, working through a hole in a small sliding plate let into the sliding door. The arsenic is condensed in separate chambers, and removed at intervals.—J. H. C.

*The Manufacture of Ferro-Boron.* C. J. L. Lettler, Shetfield. Eng. Pat. 24,230, December 16, 1893.

FERRO-BORON is obtained by mixing boric acid and any suitable ore or artificial oxidised compound of iron in the requisite proportions, and heating them in a crucible or furnace, from which the resulting borate of iron may be poured. The borate of iron thus produced, or a natural borate such as *Lagonite*, is then mixed with carbonaceous matter and again strongly heated, thus producing ferro-boron or boron-iron containing from 2 per cent. to 20 per cent. of boron.—J. H. C.

TABLE OF RESULTS.

No.	Percentage of Composition.						Colour of Grains.	Character of Crystals.	Result of Soldering Brass.	Relative Grade of Fusibility.	Phasing-Point in Bulk-Secords.	Behaviour under Hammer.		Tensile in Tensile Machine.
	Cu	Zn	Sn	Bi	Ag	Pb						Material of Tube.	Grade of Malleability.	
1	61	39	..	..	..	..	Yellow	Soft	Burnt	Very infusible	19.2	Copper	2	..
2	56	44	..	..	..	..	Yellow-grey	" but harder	"	"	19.0	"	2	..
3	51	49	..	..	..	..	Clear yellow-grey	"	"	Difficultly fusible	15.6	"	2	..
4	46	54	..	..	..	..	Grey-yellow	"	Good	"	14.8	Brass	3	..
5	45	55	..	..	..	..	"	"	"	"	14.5	"	2½	..
6	33	67	..	..	..	..	Dark yellow	Friable	"	Good	14.0	"	0	0.9
7	72	26	3.0	..	..	..	Yellow-grey	Soft	Burnt	Exceedingly infusible	24.0	Copper	1½	..
8	48	49	3.0	..	..	..	Clear yellow-grey	"	Good	Good	13.6	Brass	1½	4.4
9	57	28	15.0	..	..	..	" grey	Pulverisable	"	Difficultly fusible	11.8	"	0	0.75
10	60	20	20.0	..	..	..	"	Friable	"	"	11.4	"	0	1.3
11	72	18	4.0	8	..	..	"	Brittle	Burnt	Very difficultly fusible	19.2	Copper	1½	1.3
12	53	45	..	..	..	0.5	Yellow	Soft	"	"	15.2	"	2½	..
13	53	45	1.5	..	..	0.5	Dark yellow	"	Nearly burnt	Difficultly fusible	14.2	"	1½	..
14	44	51	3.5	..	..	1.5	Blue-grey	"	Good	Good	13.8	Brass	1½	..
15	33	51	..	2	..	..	Yellow-white	Friable	"	Very rapidly fusible	10.4	"	0	..
16	39	39	..	..	2	..	Clear green-yellow	Soft	Burnt	Difficultly fusible	15.6	Copper	3	..
17	50	46	..	4	..	..	Green-yellow	" but harder	Good (very thin flowing).	"	15.2	Brass	5	..
18	53	43	..	4	..	..	Dark green-yellow	"	"	"	14.5	"	5	..
19	48	48	..	4	..	..	Gold-yellow	"	"	Good	13.8	"	5	..
20	42	52	..	6	..	..	Dark gold-yellow	"	"	"	13.4	"	6	..
21	43	48	..	9	..	..	Red-yellow	"	"	Easily fusible	12.9	"	4	..
22	38	50	..	12	..	..	Clear red-yellow	Friable	"	Rapidly	11.0	"	3½	..
23	26	12	..	62	..	..	White-yellow	(Plate)	"	"	11.5	"	2	..
24	French brass solder.						Gold	Soft	Burnt	Difficultly	15.7	Copper	4	..
25	White solder.						White	Pulverisable	Good	Good	13.6	Brass	0	..
26*	76	24	..	..	..	..	Red-grey	Soft	"	Difficultly fusible	14.8	"	2½	..
27*	75	24	1.0	..	..	..	Grey-yellow	" but harder	"	Less difficultly fusible	14.2	"	2	..
28*	75	23	2.0	..	..	..	"	"	"	"	13.8	"	1½	..
29*	74	23	3.0	..	..	..	Dark grey	Friable	"	Fair	13.6	"	1	..
30*	74	22	4.0	..	..	..	"	Brittle	"	"	12.8	"	½	..
31*	73	22	5.0	..	..	..	Darker	Friable	"	Good	12.6	"	0	..
32*	73	21	6.0	..	..	..	"	Pulverisable	"	Easily fusible	12.2	"	0	..

\* Note by Abstractor.—The numbers are quoted as for Cu + Zn + Sn in Nos. 26—32; but this appears to be a misprint for Brass + Zn + Sn.



*Processes of and Converters for Smelting and Refining Ores.* C. M. Allen, Butte City, Montana, U.S.A. Eng. Pat. 8586, May 1, 1894.

The raw materials are reduced to a molten condition in a suitable converter, which is so mounted, on trunnions or otherwise, that it can be tipped for drawing off any required quantity from the contents, and then restored to its original position for further charging with a suitable quantity of fresh material, and so on. The converter is water-jacketed, and the blast is usually introduced through one of the trunnions. The dust and fume are collected in separate chambers, and returned to the converter at suitable intervals.

—J. H. C.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

*The Scientific and Technical Electrochemistry of the Present and Future.* W. Ostwald. Zeits. f. Electro-technik u. Electrochem. 1894, 122—125.

The author, in this address, discusses some problems, to-day of speculative scientific interest only, but which may in the near future be of technical and manufacturing importance.

The great waste of energy in the steam engine, owing to the very great loss of heat between the fireplace and the boiler and again between the boiler and the engine, is first mentioned. It is pointed out that of the energy of the coals consumed, only about 10 per cent. is realised in the form of mechanical work in the very best cases. Gas-engines may remedy this to some extent, but the author looks to the electrical transformation of the energy of the fuel. In this connection he points out that attempts at direct union of coal and atmospheric oxygen must fail. He illustrates his argument by an experiment with a divided cell containing zinc and platinum plates in potassium sulphate; no permanent current is set up till acid is added to the liquid, and this acid, which is to act on the zinc, must be added to the compartment in which the zinc is not, for electrochemical reasons set forth in the paper. He then indicates the coal-cell of the future, containing coal in the one part, the oxidising material in the other (which must be atmospheric oxygen or something easily obtained from this), and containing possibly an electrolyte for purposes of conduction, which must undergo no permanent change.

Secondary batteries are then referred to, and the author points out that as the energy in an accumulator is proportional to the quantity of electricity and the E.M.F., and as the former depends on the electrochemical equivalent of the material employed, attempts should be made to utilise in accumulators a metal of low equivalent, in order to economise bulk and weight.

Here also he points out that the ordinary distinction between primary and secondary reactions in electrolysis is to a great extent arbitrary and meaningless, for the E.M.F. needed for electrolysis is always that agreeing with the actual products, not that calculated for the imaginary ions, which are supposed afterwards to form the actual products by "secondary actions." He distinguishes between the electrolytes which aid in conveying the current and those which are separated at the poles; the current strength will depend on the resistance, to which all the substances present contribute, according to their mass or concentration and their ionic speeds, but the polarisation at the electrodes is conditioned only by what deposits there.—J. H. D.

*Darriens' Theory of the Lead Secondary Battery, and the Life of Lead Accumulator Plates.* P. Schöpp. Zeits. f. elektrotech. u. elektrochem. 1894, 294—298.

The experiments of Darriens, as described in his *Essai de Théorie Chimique sur les Accumulateurs au Plomb* (Bull. de la Soc. Internat. des Electriciens, 1892), render the generally accepted sulphating theory of Gladstone and Tribe untenable; the apparent confirmation of their theory that lead sulphate is formed upon both plates by normal discharge, which is afforded by the agreement of calculated and observed E.M.F., being purely accidental. If the positive\* plate be removed from a cell which has just been fully charged, and be plunged into water, the presence of persulphuric acid may be proved. The proportion of this acid present in the liquid increases with the strength of the sulphuric acid, being almost *nil* in a 5 per cent. solution; and it rapidly diminishes when the charged accumulator is left at rest. A determination of the persulphuric acid from a freshly charged plate, effected by measuring the oxygen produced on dilution with water, showed it to be equivalent to that given by 0.15 ampère-hour per kilo. of plate. The active material of the charged positive plate was not affected by nitric acid, indicating the absence of oxides of lead lower than  $PbO_2$ , whilst after washing and drying *in vacuo* it evolved 31 per cent. of chlorine from hydrochloric acid (pure  $PbO_2$  gives 29.7 per cent.); when partially dried it smelt strongly of ozone. The E.M.F. of a charged positive plate, purified from persulphuric acid by washing with water, when opposed to a normally charged negative plate, was 1.92 volt, whilst a plate filled with chemically prepared peroxide gave 1.88 volt.

After normal discharge (stopping when the E.M.F. falls to 1.8 volt), the positive plate is partly soluble in ammonium acetate solution, and therefore contains lead oxide and sulphate; mixed with hydrochloric acid it evolved from 18 to 24 per cent. of chlorine. The quantity of sulphate found by boiling a portion of the well-washed material with sodium hydroxide and precipitating with barium chloride, is given in the following table, from which it will be seen to be small, and quite disproportionate to the ampère-hours of discharge.

Expt.	After Charging.		After Discharge.		Formed.		Ampère-hours.	$PbSO_4$ corresponding to Amp.-Hrs.
	$PbO$ .	$PbSO_4$ .	$PbO$ .	$PbSO_4$ .	$PbO$ .	$PbSO_4$ .		
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.		Per Cent.
1	2.33	3.60	8.72	4.41	6.39	9.81	86	21.12
2	0.21	0.47	6.97	4.40	6.76	3.93	114	28.00
3	0.40	0.90	8.40	2.18	8.00	1.28	120	29.47
4	2.44	0.95	10.92	4.01	8.48	3.06	121	29.71
5	1.47	0.19	6.65	1.16	5.18	0.97	76	18.66

Hydrometric determinations of the strength of the electrolyte before and after discharge, confirm the results of these experiments and show the sulphating to be confined to one plate. This is contrary to Kohlrausch's experience; but

\* Throughout this abstract the plate on which peroxide is formed during charging is termed the *positive*, and that on which lead is reduced the *negative* plate. And for sake of brevity either plate is said to be "charged" or "discharged" when it has been taken from a cell which has been charged or discharged.



Darriens discharged his cells rapidly, and then at once made his determinations, because a slower discharge or a period of rest subsequently would allow the sulphating to take place by chemical action. But the sulphate formation, even on the negative plate, is due to secondary reactions:—A negative plate, after discharging 58 amp.-hrs. with an average E.M.F. of 1.80 volts, contained only a trace of sulphate; but the sponge, when heated before the blow-pipe, suddenly glowed, and on cooling was shown to be a mixture of litharge and lead globules, and thus behaved as lead sub-oxide would. If sub-oxide were the first product of discharge it would quickly react with sulphuric acid to form lead sulphate and metallic lead, so that the sulphating would be proportional to the current, and might at first be ascribed to direct electrolytic action. The presence of lead molecules side by side with those of the sulphate accounts for the rapidity of the action of charge and discharge, which is so inexplicable by the old theory, when the insolubility and low conductivity of lead sulphate are considered. On long standing after discharge, especially if left short-circuited, the admixed spongy lead itself becomes sulphated, and then the cells cannot be again charged; this is confirmed by the fact that the plates lose "capacity" when exposed freely to air:—A plate, normally giving 320 amp.-hrs. afforded only 32 amp.-hrs. after a month's free exposure to the air.

The rationale of charge and discharge appears to be as follows:—On charging, persulphuric acid, formed at the positive electrode, there converts lead oxides and sulphate into peroxide, and, when this conversion is complete, is broken up into sulphuric acid and oxygen, the escape of which gas marks the completion of charging; whilst on the negative plate lead sulphate is reduced to spongy lead, and finally hydrogen is evolved after a small proportion has been occluded by the sponge. Then, on discharging, the occluded hydrogen is oxidised in the one plate, and the persulphuric acid is reduced in the other, thus accounting for the initially high E.M.F. of the cell; next a part of the spongy lead of the negative plate is converted into sub-oxide, which reacts with the electrolyte to give lead and lead sulphate. Meanwhile the peroxide on the positive plate is reduced to a lower oxide, which in time is partly transformed into sulphate; spontaneous discharge ("local action") of the positive plate, owing to the presence of  $PbO_2$ ,  $PbO$ , and  $H_2SO_4$ , may result in the complete sulphating of the plate, as it only ceases when all peroxide has been reduced. The E.M.F. on first discharging, due to the oxidation of nascent hydrogen and decomposition of persulphuric acid, is theoretically 2.07 volts ( $+34.5 + 13.2 = 47.7$  kilo.-calories), but by applying Helmholtz' correction it approximates to that observed, viz., 2.24 volt; subsequently it is 1.9 volts. In making thermal calculations it must be remembered that the published heats of formation of lead salts refer to dense, not spongy, lead; and these two substances give with a peroxide plate in dilute sulphuric acid 1.6 and 1.93 volts respectively; chemically prepared lead sponge behaves precisely like that formed on the negative plate, and both may perhaps be considered a true allotropic modification of the metal.

The complete sulphating of the negative plate on continued short-circuiting may be explained by the electrolysis of persulphuric acid, which continues so long as any peroxide exists on the positive plate. But it should be noted that solid lead sheet exposed to a solution of potassium persulphate is gradually covered with a film of oxide, whilst if in contact with platinum wire, it will be encrusted with white lead sulphate in less than a day. It is the action of persulphuric acid on massive lead which causes the often observed rapid destruction of the lead plates; and as the production of this acid is essential to the action of the battery, it may be said that every lead accumulator inevitably carries within it the means for its own destruction; nevertheless circulation of the liquid, even while the battery is at rest, may possibly prolong the life and increase the capacity of the cell.—W. G. M.

*The Electromotive Force of Alloys in a Voltaic Cell.*  
A. P. Laurie. Proc. Chem. Soc. 1894 [142], 173.

THIS paper contains the results of determinations of the electromotive force of 16 of the 19 alloys alluded to in Matthiessen's paper on the conductivity of alloys (*Phil. Trans.* 150). The results obtained confirm Matthiessen's conclusion that only one compound, the tin-gold alloy, exists among the 16, which are:—

Bismuth-tin, bismuth-lead, bismuth-gold, bismuth-silver, gold-silver, antimony-lead, cadmium-zinc, antimony-tin, lead-gold, lead-silver, lead-tin, lead-zinc, lead-cadmium, cadmium-tin.

In some cases the addition of a metal to the alloy results in displacement. Thus mercury decomposes the gold-tin compound, and zinc, added to a copper-tin alloy, appears to replace the tin.

*The Electrolysis of Copper Sulphate.* A. Chassy. Comptes rend. 1894, 119, 271—272.

ON electrolysing a hot solution of copper sulphate a violet-red deposit of cuprous oxide is frequently obtained. At 100° C., with a current density of  $\frac{1}{100}$  ampère per sq. cm., a saturated solution of copper sulphate gives on a platinum electrode a deposit which, when examined under the microscope, is seen to consist of bright red crystals in the form of cubes or octahedra. The deposit is not always uniform, for, if the temperature of the electrolyte be lowered some of the crystals have the yellowish-red colour of copper, their proportion increasing with diminishing temperature. At 40° C. only a few isolated red crystals are obtained, the bulk of the deposit being metallic copper. Raising the current density and diminishing the concentration of the electrolyte have the same effect as decreasing its temperature. In order to obtain red crystals the electrolyte must be nearly neutral. Removal of oxygen by previous ebullition of the liquid does not prevent the formation of the red crystals, which upon analysis are found to be cuprous oxide, thus ranking as artificial cuprite.

A comparison was made between the weight of the deposit thus obtained and that of one of copper thrown down in a voltmeter in series with the first. The deposit of cuprous oxide was always greater in quantity than corresponded with the weight of the copper thrown down in the second voltmeter (calculated into  $Cu_2O$ ). The relation to be expected is expressed by the ratio 1:1.12, whereas that actually obtained was 1:1.35, when the conditions were favourable to the production of cuprous oxide. If a plate of copper of the same dimensions as the platinum electrode be kept in the cell for the whole period during which electrolysis is proceeding, it suffers an oxidation so slight as to be negligible compared with that of the deposit on the platinum plate. The loss of weight of the anode of a voltmeter used hot is smaller than the increase of weight of the cathode. It is obvious that measurement of current by means of a voltmeter containing a warm solution of copper sulphate is inaccurate; a slight error exists even when the solution is feebly acid and when the current density is fairly high, but the inaccuracy becomes of moment when the solution is neutral and the current density small.

—B. B.

*The Electro-Nickeling of Metallic Surfaces.* Dingl. Polyt. J. 293, 69—70.

THE following baths (most of them well known) have all given good results, but require careful handling:—

1. 8 kilos. nickel ammonium sulphate in 100 litres of water, made slightly alkaline with ammonia, and then weakly acidified with citric acid.

2. 5 parts nickel sulphate neutralised with ammonia, 3.75 ammonium tartrate, and 0.025 gallotannic acid per 100 of water. This gives a homogeneous white and smooth reguline deposit, even when of great thickness.

3. 2.75 nickel acetate, 2.5 calcium acetate, and 100 of water, afterwards mixed with 0.7 part of acetic acid (sp. gr. = 1.017) and filtered. (Potts' formula.)

4. 5 nickel ammonium sulphate, 2 ammonium sulphate, 0.5 citric acid, and 100 of water. Boil and filter.

5. 8 nickel ammonium sulphate, 1 ammonium chloride in 100 of water, with or without the addition of 0.5 part barium oxalate.

6. 6 nickel ammonium sulphate, 3.5 ammonium chloride, and 2.5 ammonium sulphate per 100 of water.

7. 5 nickel ammonium sulphate, 1 ammonium sulphate, and 100 of water. Specially suitable for cast iron.

8. 5 nickel ammonium sulphate, 2.5 boric acid, 100 water.

Powell has found that the addition of not more than 1 to 8 grms. per litre of benzoic acid or of a benzoate to a suitable nickel bath produces a good and pure deposit. Baths containing boric acid, such as are commonly employed, give a good deposit upon smooth surfaces, but refuse to cover cavities or hollows; this difficulty may be removed by the addition of sodium chloride to the bath. Such a bath may be made by dissolving 5 kilos. nickel ammonium sulphate in 100 litres of water, adding 2.5 (or 1.25) kilos. of boric acid and 1.25 kilos. of sodium chloride, boiling, acidifying with citric acid, neutralising with ammonia, and filtering.

So also nickel chloride with boric acid in the proportion of 5 : 2 or 2 : 1 gives a good bath, but it is not suitable for depositing upon iron or steel, as all baths containing chlorine are apt to cause rusting of these metals. The use of citric, benzoic, tartaric, or other weak acids is to be recommended for them, except upon the score of expense. —W. G. M.

*Klein's Method of Compressing and Shaping Electrolytically Deposited Metals.* G. Langheim. Zeits. f. Electrochem. u. Electrochem. 1894, 161—165.

THE object of the apparatus here described is the electrolytic production of articles (such as candlesticks or stair-rails) the shape of which is that of surfaces of revolution round a central straight axis, and the longitudinal section of which may have any profile. The cathodes are built up on central "cores" which consist of strong rods or tubes, coated with a layer of easily fusible substance; these, if tubes, are stopped at the ends by caps, and in any case are provided with short axles at the ends. A core is covered with (for example) clay, and is then worked to the right shape by being rolled on a plate the surface of which is cut in parallel ridges and hollows of the required form; it is then baked and finished by being again rolled under pressure on the plate, guides being used to ensure the right motion. Its surface having been rendered conducting, it is placed in a bath of the electrolyte, on a glass or porcelain plate of the same shape as that on which it was formed, confronted with an anode also shaped to correspond to it, and rolled back and forth during the whole time of the deposition, more or less pressure being applied to it according to circumstances. A number of objects of the same or of different shapes can be worked at the same time; and the paper gives diagrams illustrating various forms of apparatus, in which the cathodes are arranged side by side on the horizontal or inclined plate, and pressure and motion applied by means of a framework laid over them, provided with slots open below in which the axles work, or in which the motion is guided by wheels rolling against the sides of the trough, while pressure is got by elastic pressure-rollers working against the top or against upper ledges. In other forms the plate is a vertical cylinder with horizontal corrugations, round which the cathodes roll continuously as they rotate round a central vertical axis; or a flat plate with circular corrugations, on which the cathodes, radially disposed round a central axis, roll like the spokes of a wheel. Metal can thus be deposited on both the inside and outside of an annular cathode, and is obtained in an exceedingly tough and resisting state; whilst by substituting polishing arrangements for the shaped "matrix-plates," the same apparatus can be used for silver or nickel plating. The author points out the advantage of Klein's process over Elmore's, in that so much more work can be done in the same space and with the same amount of electrolytic fluid, and that not only cylindrical articles, but objects of

any profile, can be produced. He suggests the manufacture of boiler tubes as one of the large uses to which the method may shortly be put. J. T. D.

*The Analysis of American Refined Copper.* H. F. Keller. J. Franklin Inst., July 1894, 54.

See under XVIII., page 1222.

*Quantitative Analysis by Electrolysis.* I. R. Schott. Zeits. angew. Chem., 1894, 388.

See under XVIII., page 1224.

## PATENTS.

*Improvements in Voltage Batteries.* J. A. S. Gregg, New Rochelle, U.S.A. Eng. Pat. 20,975, November 4, 1893.

THE apparatus described is for automatically supplying a cell with fresh liquor at the rate required by the use of the cell and in a continuous manner.

The cell is provided with a water-tight cover, through which a wide vertical tube passes to the bottom. A second tube passes just through the cover and is bent to act as an overflow. Above the wide tube is mounted a reservoir, mouth downwards, the mouth being fitted with a short length of pipe and a stopcock. The end of this pipe dips just below the level of the liquor in the wide tube. During use the spent liquor, being of lower density, collects at the top and flows away by the overflow pipe. The level falls in the wide tube, and bubbles of air enter the short pipe and pass into the reservoir, a corresponding quantity of fresh liquor flowing from the latter into the wide tube. The reservoir can be raised or lowered so as to regulate the point at which feeding shall go on. The short pipe from the reservoir has the end which enters the wide tube, cut off obliquely.—E. T.

*Improvements in Electrolytic Apparatus.* W. H. Caldwell, Cambridge. Eng. Pat. 21,531, November 13, 1893.

THESE improvements relate to a diaphragm for use in electrolytic tanks. This diaphragm is made up of the loose crystals of the salt which is being decomposed. A vertical wall of these crystals is maintained by horizontally placed strips of glass, porcelain, or other suitable material, arranged vertically one above the other, from the bottom of the tank to the surface of the solution, after the manner of the laths of a Venetian blind. Apparatus is shown for the electrolysis of sodium chloride solution, narrow spaces being left between the screens for the electrodes.—J. C. R.

*Improvements relating to the Electrolytic Preparation of Oxygen and the Halogens, and to the Simultaneous Production of Electrodes.* A. Cohn, Berlin, Germany. Eng. Pat. 23,478, December 6, 1893.

THE inventor proposes to make use of the hydrogen set free in the electrolytic production of oxygen and the halogens, by employing a cathode capable of absorbing it. The compound electrode thus produced is employed in the generation of current in primary or secondary batteries. "Negative accumulator plates work advantageously as cathodes. These absorb the evolved hydrogen, as in their usual accumulator charge, until they reach a saturation limit, which is indicated by a violent ebullition of free hydrogen."

Batteries are formed with this hydrogen-charged lead, representing the zinc, and copper, separated by a diaphragm, in sulphate of copper, as in the Daniel's cell. Single fluid batteries are constructed with negatives of carbon or copper in dilute sulphuric acid. Polarisation is prevented by constant circulation of the electrolyte, or by blowing air through it. Cells thus set up can be employed as generators of electric current for a further electrolysis, in order to produce oxygen or halogens thereby.—J. C. R.

*Improvements in Process and Apparatus for Electrolytical Decomposition of Alkali Salts.* A. Sinding-Larsen. Christiania, Norway. Eng. Pat. 13,499, July 12, 1894. (Under International Convention, December 12, 1893.)

This invention relates to apparatus in which mercury is employed as a cathode for transporting the metallic constituent separated from the salt in solution under treatment. Chloride of sodium is the example given. The characteristic feature of the process is the use of an amalgamated metal body (tinned iron for example), having a greater part of its surface exposed to the solvent agent, a part of its surface in contact with mercury serving as a cathode, and being moved in such a manner that the amalgam in the cathode is pushed through the solvent in the form of a thin layer adhering to the surface of the moving body.

The illustrations show metal discs or drums mounted on shafts journaled vertically and horizontally across, or in tanks divided by non-porous walls extending into the mercury. These discs or drums dip into the mercury. In one case the mercury is contained in a bowl perforated at the bottom, by the rotation of which the mercury is continually carried up centrifugally and falls back again over a flanged edge, re-entering the bowl by the perforations. The anode chamber walls dip into the mercury in the centre of the bowl. The outer portions of the bowl are in the tank containing the solvent liquor.

In another description it is proposed to separate the mercury from the anode by the electrolyte under pressure forced in between them, the mercury normally resting on the anode, but a piece of thin cloth may be inserted between them. Methods, electrical and otherwise, are explained for rendering the apparatus automatic.—J. C. R.

*Improvements in the Manufacture of Carbon for Electric Lighting and other Electrical Purposes.* R. Niewerth, Berlin, Germany. Eng. Pat. 24,081, December 14, 1893.

THE inventor claims the heating of carbons after moulding, by means of an electric current in place of baking. During this heating the carbon is supported in a holding sleeve of non-conducting material to prevent warping. Ammonious earths, magnesia, and other metalliferous compounds, which are added to the mixture before moulding, are reduced when the carbon is subsequently raised to a "red heat." Carbons thus prepared have less resistance and more illuminating power when used in arc lamps, as the metal, burning simultaneously with the carbon, gives a very great illuminating effect.—J. C. R.

*Improvements in Apparatus for Electrolytic Purposes.* H. Guthrie, Manchester. Eng. Pat. 21,276, December 16, 1893.

THE inventor describes apparatus for the decomposition of brine by electrolysis. Electrodes of sheet or flat material are placed between frames carrying porous partitions. These are arranged in series to form electrode compartments, the whole being screwed up together, filter press fashion, to form tight joints, in a containing tank for the electrolyte. Suitable feed and exit ducts or passages for the products are fully detailed and shown.—J. C. R.

*A New or Improved Preparation of Active Material for Electric Accumulators.* C. K. Mills, London. From C. J. Barbier, Lyons, France. Eng. Pat. 24,353, December 18, 1893.

THESE improvements relate to the preparation of active material for electric accumulators. Lead or an alloy in a liquid state is projected upon a rapidly revolving wheel, provided with floats or blades, which acts by shocks upon the molten metal and produces particles of peculiar texture. This wheel is fixed on a shaft or spindle journaled in a tank containing water, the sides of which are kept wet by a perforated water-coil. By projecting the lead from the wheel on to solid metallic or other surface, to which is given movement of variable speed, agglomerates are formed, the metal being partially soldered to it. In this agglomerated

condition it is applied to form negative plates without supports. In the finely divided condition, the material produced is applied as a paste, or under pressure in proportion to the porosity required.—J. C. R.

*Improvements in Electrode Plates for Secondary Batteries.* Dr. A. Lehmann, Berlin, Germany. Eng. Pat. 24,389, December 19, 1893.

A LEAD plate is so corrugated as to present the appearance of a series of gutters of semicircular section placed one above the other, with their axes horizontal. The top gutter is turned round its axis apparently 30° or 40° degrees to the right: its right-hand horizontal edge is the right-hand edge of the second gutter, which, running below it, is inclined towards the left: the left-hand edge of this is the left-hand edge of the third gutter, which is turned towards the right: and so on to the bottom of the plate. The active material is placed in these gutters, and is locked in place independently of the action of gravity, by passing into numerous perforations made in the metal. If any material should fall out of these perforations, it will fall into the gutter below.—E. T.

*A New or Improved Electrical Accumulator Plate.* C. K. Mills, London. From C. J. Barbier, Lyons, France. Eng. Pat. 24,442, December 19, 1893.

A NEW accumulator plate in which the active material is enclosed in a flat box of lead or lead alloy, the flat sides of which are perforated and provided on the interior with pins or projections passing through the active material, and extending to within a short distance of the opposite side.—E. T.

*Improvements in Primary Voltaic Batteries.* W. Walker, jun., Birmingham. F. R. Wilkins, Handsworth, and J. Lones, Smethwick. Eng. Pat. 616, January 11, 1894. Improvements on Eng. Pats. 23,007 of 1892 and 10,942 of 1893.

(THIS Journal, 1893, 1042; 1894, 529.) The porous pot, containing a zinc cylinder, is placed in a perforated vessel of earthenware, the annular space between them being packed with carbon granules, fine next the porous pot and coarser next the perforated outer vessel. Carbon electrodes make contact with these granules, which are thus kept continually moist by the leakage from the porous pot, and are yet bathed in air. It is the air which acts as depolarizer. Caustic potash or caustic soda are preferred as electrolytes. The cell, of course, stands in a tray, to collect the liquor flowing from the porous pot. A perforated metal cylinder is in some cases placed between the porous pot and the earthenware vessel, to make contact with the carbon granules, in place of the carbon electrodes.—E. T.

*Improvements in Electrical Accumulators or Storage Batteries.* D. Young, London. From the Hess Storage Battery Company, Springfield, U.S.A. Eng. Pat. 16,425, August 28, 1894.

THE electrode preferred by the patentees consists of a plate of active material clamped between two perforated lead sheets. The perforations and the spaces between the separate electrodes are filled with quartz, sand, or some such non-conductor, the separate grains of which are held together by some adhesive material like india-rubber or shellac, such material not being allowed to fill the interstices. The parts of the lead sheets not immediately in contact with the active material are varnished or otherwise protected.

The use of quartz sand or similar material, treated as above, is claimed for cells of any type, as well as for the one described.—E. T.

*Improvements in Secondary or Storage Batteries.* C. Rindon, Toronto, Canada. Eng. Pat. 18,288, September 22, 1894.

EACH electrode consists of a number of plates or strips of metal laid horizontally, and separated by layers of active

material. The side-edges of the strips are turned up or down, to keep all in position horizontally. Clamping-plates of insulating material are placed at top and bottom, and are furnished with side lugs, through which bolts pass to bind the whole together. Each bolt is provided with a springy washer—made, for example, of india-rubber—which, while constantly exercising pressure, allows for expansion of the electrode. At one end the metal strips are continued beyond the active material and bent upwards; they are united above the surface of the electrolyte by a saddle-piece carrying a terminal.—E. T.

*Improvements in and connected with Carbons for Electric Lamps, or for other Purposes.* E. G. Acheson, Monongahela, U.S.A. Eng. Pat. 18,339, September 27, 1894.

For arc lamps, the inventor uses rods made of pure carbon to which has been added about 10 per cent. of carbide of silicon; or the core of an ordinary carbon may be made of carbide of silicon.

For incandescent lamps, the carbide is suspended in the oil-bath used for building up the carbons, and gets bound up in the carbon deposited from the oil on the filament; or other methods may be employed to construct a filament containing carbide of silicon.

The inventor claims that the above additions to the pure carbon have the effect of increasing the number of light-waves produced at a given temperature.—E. T.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*The Clarification of Oils.* Chem. and Druggist.  
November 24, 1894, 740.

MANY methods have been tried, some of which are very slow. In the *Corps Gras Industr.* cellulose and asbestos are especially recommended, filters similar to those used for beer, being used. The best results are said to be obtained by first clarifying by means of decantation, and finally filtering through cellulose.

*The Extraction of the Free Acids of Beeswax.* T. Marie.  
Comptes rend. 1894, 119, 428—431.

THE free acids characteristically present in beeswax, were considered by Brodie to be a chemical individual to which he assigned the name cerotic acid,  $C_{26}H_{52}O_2$  (melting point  $78^\circ\text{C}.$ ). Schalfew in 1875 showed that this substance was a mixture, containing a small quantity of an acid (melting point  $91^\circ\text{C}.$ ) of the formula  $C_{34}H_{68}O_2$ ; the more recent values of Nafzger for the same acid are melting point  $90^\circ\text{C}.$  and formula  $C_{30}H_{60}O_2$  or  $C_{31}H_{62}O_2$ . The author has endeavoured to supplement existing knowledge on this subject by working on large quantities of beeswax and submitting the products to various methods of fractionation, and has found that fractional solution in boiling methyl alcohol yields the best results, always provided that it be applied to mixtures of acids free from substances of alien character. In the case of beeswax, however, boiling alcohol dissolves certain hydrocarbons, fatty products and colouring matters which may be present, and also myricin in addition to these. (The last-named substance is almost insoluble in alcohol *per se*, but is taken into solution by the wax acids). The separation of the first two impurities is effected by taking advantage of the solubility of fatty and colouring matters in cold alcohol, and of the ease with which the hydrocarbons can be extracted from the salts of the wax acids. Myricin is removed by heating the crude wax acids with potash lime (containing half its weight of caustic potash), whereby it is saponified and the alcohols thus liberated oxidised and fixed as the corresponding acids. The

acids thus formed are found by the author to be identical with the free acids of the wax. The application of the foregoing method is made in the following way.—The wax is extracted with boiling alcohol, the bulk of which is distilled off. The residue is cooled and pressed to remove colouring and fatty matter. The cake of wax thus obtained is washed with boiling water, decolourised by charcoal and filtered. It is then heated with potash lime until hydrogen ceases to be evolved; the mass is powdered and boiled with a large quantity of water. The solution is saturated with dilute hydrochloric acid, the wax acids, in the presence of soluble calcium salts, being completely transformed into insoluble calcium salts, which are precipitated. These are washed, dried, and extracted by boiling alcohol and light petroleum (to dissolve impurities). The acids are recovered from the purified calcium salts and after the removal of a little palmitic acid, derived from the decomposition of the myricin, are found to fuse at  $79-80^\circ\text{C}.$  The mixed acids are gently heated with 30 times their weight of methyl alcohol, and filtered at  $60^\circ\text{C}.$ ; the filtrate, on cooling, deposits cerotic acid. The process is repeated until the fusing point falls to  $76^\circ$ ; a single recrystallisation of the product in ethyl alcohol, raises the fusing point to  $77.5^\circ\text{C}.$  This acid appears to be pure cerotic acid, several dry methods of fractionation having failed to prove it non-homogeneous. The crude acid on the other hand contains 30—40 per cent. of acids similar to cerotic acid.—B. B.

*The Behaviour of the Alkaline Salts of Fatty Acids and of Soaps in the Presence of Water.* I. and II. F. Kraff and A. Stern. Ber. 27, 1747—1761.

AN historical and critical account of the various theories of the action of soap and of the decomposition by water of the alkaline salts of the fatty acids of which it is composed. Chevreul's researches, the results of which were published in 1823 (*Recherches chimiques sur les corps gras d'origine animale*) still remain the most important ones on this subject. His views regarding the behaviour of the alkaline salts of the solid fatty acids and of oleic acid, when dissolved in a large amount of water, although now generally abandoned in favour of those of Rotondi and others must, according to the authors' investigations, still be accepted as correct. According to Chevreul a solution of normal potassium stearate in 20 parts of boiling water gives on dilution with 1,000 parts more of hot water and subsequent cooling a precipitate of an acid stearate, potassium hydrate and only an almost imperceptible trace of stearic acid remaining in solution. Potassium oleate, on the other hand, shows far greater stability, and in order to effect its decomposition it is necessary to use a very large quantity of water and to expose the solution to a low temperature for a considerable time. Chevreul made use of this difference in the behaviour of the stearates (and margarates) and oleates for the separation of the respective acids. An acid potassium oleate (bioleate) was obtained by the same investigator by the combination of weighed quantities of oleic acid and caustic potash, as an almost insoluble substance. Rotondi (1883) assumes that the soaps are split up by water into basic and acid salts, the former being readily soluble in hot and cold water. His experiments were made with a solution of purified Marseilles soap. Subjecting this to dialysis he obtained besides the insoluble acid salt, an alkaline solution, in which, after evaporation on the water-bath, he estimated the amount of sodium, and, overlooking the fact that the supposed alkaline soap consisted of nothing but a mixture of free alkali and sodium oleate, he arrived at his erroneous conclusions. Basic alkaline salts of the fatty acids and of oleic acid have never yet been obtained. The authors likewise did not succeed in preparing them. Thus, by dissolving equal parts of elaidic acid and caustic soda in hot alcohol, and pressing and drying the crystals *in vacuo* which separated on cooling from the filtered solution, it was found that only normal (neutral) sodium elaidate had been formed. A further proof of the non-existence of basic soaps is the fact that from highly alkaline soap solutions only neutral soap is precipitated by the addition of common

salt. This has long been recognised by practical soap-makers, and was verified by the authors by accurate experiments.

For the study of the hydrolysis of soap the authors used pure sodium palmitate. It was found that about 900 parts of water are required to separate all the palmitic acid as acid sodium palmitate,  $C_{15}H_{31}O_2Na$ ,  $C_{15}H_{31}O_2$ , and that by reducing the quantity of water less acid palmitates of varying composition can be obtained. The dilute hot solution of sodium palmitate or stearate shows the remarkable property of possessing a strongly alkaline reaction, and at the same time containing minute globules of free fatty acid in suspension. By extraction of the hot solution with toluene, the fatty acid can be obtained in a state of perfect purity. Formation of the acid salt takes place on cooling the solution, by the combination of the acid set free with the neutral salt remaining undecomposed in solution. The filtrate from the acid salt never became strikingly turbid by the addition of a mineral acid, and therefore could not contain a basic salt, but consisted simply of a solution of alkali. It seems probable that not only the free alkali but also the finely suspended free fatty acid plays a definite part in the washing process with hot soap solutions. The difference in the action of water on neutral sodium oleate as compared with sodium palmitate and stearate is shown clearly by the fact that 200 parts of hot water are necessary to produce even a faintly turbid solution of sodium oleate. This turbidity is scarcely increased in a solution of 1:900, and immediately disappears on addition of a small quantity of alkali. The acid sodium oleate behaves in a different manner, being immediately split up by an excess of cold water, with separation of minute globules of free oleic acid, causing a turbid solution. Sodium elaidate closely resembles sodium stearate in its behaviour in dilute aqueous solutions.

Regarding the cleansing action of soap the authors point out that its emulsifying capacity for greasy substances, and the important part which this plays, was fully recognised by Chevreul, Berzelius (1828), and Persoz (1846), whilst more recently purely chemical theories of its action held the field. By more recent research the truth of Chevreul's theory has become fully established.—F. M.

*Action of Hydrogen Sodium Sulphite and Sulphurous Acid on Oleic and Erucic Acids.* M., C., and Alex. Saytzeff. *J. Prakt. Chem.* 50, 1894, 73—80.

DESIRING to extend the field of research opened up by the investigations of Strecker and Messel and others on the direct union of unsaturated carbon compounds with bi-sulphites of the alkalis, the authors experimented on oleic and erucic acids, but with results differing from those obtained by their predecessors with such bodies as fumaric acid, acrolein, &c.

On gradually heating oleic acid with an equal bulk of  $NaHSO_3$  solution (saturated in the cold) in sealed tubes up to  $180^\circ C.$ , the liquid separated when cooled into a crystalloid fatty layer, a watery liquor containing  $H_2SO_4$  and free sulphur. The fat, after repeated recrystallisation and purification by alcohol and ether, was found to be identical in composition with oleic acid, and from observation of its melting and congealing points, behaviour in combination with alkalis and silver, oxidation by potassium permanganate, and results when tested by an alcoholic solution of iodine in presence of  $HgBr_2$ , was considered to be elaidic acid, which view was confirmed on comparison with this acid prepared by the action of nitrous acid on oleic acid.

In order to determine whether the transformation was attributable to the action of the hydrogen sodium sulphite itself or to the sulphur dioxide evolved therefrom at the high temperature, the experiment was repeated separately with neutral sulphite of sodium and with a solution of  $SO_2$  saturated in the cold. In the former case no detectable amount of elaidic acid was produced, even with a temperature of  $200^\circ C.$  and over, but with the  $SO_2$  solution (kept at  $200^\circ C.$  for 24 hours) the same phenomena were observed as in the original experiment and the resulting fat exhibited

on purification all the characteristics of elaidic acid. The amount of this acid produced was increased when a larger initial volume of  $SO_2$  solution was employed.

Erucic acid yielded brassic acid on being treated with hydrogen sodium sulphite or sulphurous acid under the same conditions as the oleic acid, and the brassic acid thus obtained corresponded in all particulars with the same acid prepared by the action of nitrous acid on erucic acid. Almond oil also yielded elaidic acid on exposure to the same treatment, and in addition the oil underwent saponification with formation of fatty acids and glycerol.—C. S.

*A Simple Viscosimeter.* M. Wendriner. *Zeits. angew. Chem.* 1894, 545.

See under XXIII., page 1220.

## PATENTS.

*Improvements in Bleaching, Sweetening, and Purifying Oils and Fats.* F. B. Aspinall, R. W. Hoar, and G. H. Wise, Hull. Eng. Pat. 18,767, October 6, 1893.

THE application of an electric current is claimed for this purpose. A tank divided into two parts by a porous partition is used. In one compartment a carbon electrode is placed in a solution of salt at  $8^\circ T.$ , and in the other compartment a copper electrode, with a mixture of equal parts of oil and salt solution. A continuous electric current is then generated by a dynamo, the oil and salt solution being agitated by mechanical means at the same time. The treatment of oils and fats by means of the light and heat generated by incandescent electric lamps immersed in the oil is also claimed.—J. J. K.

*Process of Distilling Glycerin and similar Liquids, and of Treating the Residue Obtained after Distilling Glycerin.* J. Van Ruynebeke, New York, U.S.A. Eng. Pat. 24,556, December 20, 1893.

THE first part of this invention consists in the use of a closed vessel, containing an expansion coil of larger diameter than that of the steampipe from the boiler, for the purpose of allowing the steam to be expanded in the coil, and reheated at the same time by steam outside the coil, supplied by the same boiler. This reheated steam at a temperature of  $300^\circ F.$  is then blown into the still containing the crude glycerin or glycerin residues, which is also heated by a steam coil. A high vacuum is maintained in the still during the distillation.

The second part of the invention consists in treating the "foots" left in the still, with sulphuric or hydrochloric acid, to decompose the sodium salts of the non-volatile organic acids, separating the precipitate formed, then volatilising the acetic acid, and distilling off the glycerin from the residual liquid in the manner described above.

—J. J. K.

*An Improved Soap.* H. J. W. Taylor, Scarborough. Eng. Pat. 25,001, December 29, 1893.

THE alleged improvement consists in the admixture of about one part of chocolate, cocoa, or the extracts thereof with three parts of ordinary soap, whereby it is claimed that a soft and pleasing effect is produced on the skin.—J. J. K.

*Improvements Relating to the Manufacture of Soaps.* J. O. Klimsch, Vienna. Eng. Pat. 15,228, August 9, 1894.

THIS relates to the production of a hard resia soap. 100 kilos. of resin and 120 kilos. of sodium carbonate are mixed and ground to a powder, and the mixture is moistened with a binding agent, such as alcohol, benzene, glycerin, starch, glue, or other material, and pressed into tablets or bars. This soap is said to be soluble in cold water.—J. J. K.

### XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

#### (A).—PIGMENTS, PAINTS.

##### PATENTS.

*Improvements in Washing Blue.* K. Ammann, Lenzburg, Switzerland. Eng. Pat. 17,630, September 17, 1894.

THREE hundred parts of ultramarine in fine powder are mixed with 150 of powdered and warmed glucose and five parts of sodium thiosulphate (hypo-sulphite), perfume being added if desired. It is claimed that this device renders spotting of the goods impossible, and that the balls may be repeatedly used until they are completely consumed without suffering any decomposition and without the appearance of fungoid growths.—F. H. L.

*An Improvement in and relating to Compounds for Destroying or Removing Old Paints.* E. Tessen, Rostock, Germany. Eng. Pat. 17,544, September 14, 1894.

It is claimed for this composition, that it will remove old paint or lacquer from wooden surfaces completely, in a short time without injuring the wood itself. This is effected by introducing into the substance a quantity of "unsaponified" paraffin oil, "said oil having the property of filling the pores of the wood, and of protecting thereby the fibres of the latter." 30 kilos. of linseed oil are saponified with 20 of caustic potash, 100 of paraffin added, and the mixture stirred into 50 of slaked lime. When homogeneous, 500 kilos. of caustic soda, 200 of potash, 200 of chalk, and 25 of starch or flour are mixed with it, and the whole is ready for use.

—F. H. L.

#### (B).—RESINS, VARNISHES.

##### PATENTS.

*Improvements in Varnish.* R. G. Bennett, London. Eng. Pat. 19,115, October 11, 1893.

LINSEED oil is mixed with half its weight of water and 2 per cent. of sulphuric acid and an electric current passed through the liquid which is kept in the form of an emulsion by means of a stream of air or ozonised air, one or more of the electrodes being made in the form of a revolving disc to assist in the agitation. Sodium peroxide in small quantities may be added to hasten the oxidation, but in the latter case, care must be taken to avoid fire. Gum or resin varnishes in oil or turpentine may be prepared in a similar manner after the addition of 10 per cent. of water.—F. H. L.

*New or Improved Manufacture of Varnish, or of a Substance for Use in Making Varnish.* L. L. B. Meyer, Hamburg, Germany. Eng. Pat. 9883, May 21, 1894.

FAT-TAR, a kind of pitch which remains in the still after the dry distillation of fatty acids, is heated for a few minutes to 500° C. with 5 or 6 per cent. of litharge, and 5 or 4 per cent. of lead peroxide. When cold it is dissolved in a mineral "oil" such as benzene, and diluted to a proper varnish strength with turpentine or other diluent.—F. H. L.

#### (C).—INDIA-RUBBER, &c.

##### PATENT.

*A New Elastic Material or Manufacture, composed chiefly of Gutta Percha, India-Rubber, or other Analogous Substance, and the Application of the Same to various Useful Purposes.* E. M. Desprez, Paris. Eng. Pat. 23,028, November 30, 1893.

THIS invention consists primarily in the manufacture of a strong or tough elastic material by taking gutta-percha, &c. in the form of a sheet and covering it on one or both sides

with a more or less close-meshed fabric or wire gauze, the whole being agglomerated by simple pressure under heat. With the gutta-percha may be incorporated saw dust and zinc oxide for various purposes, and the material may finally be cut up into strips. Its chief value lies in the ease with which it can be moulded by heat into any shape, the curvature being retained when cold.—F. H. L.

### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

*The Estimation of the Sugar Content of Tanning Materials, Tanning Extracts, Tanning Liquor and Weighted Leather.* A. Schroeder, A. Bartel, and W. Schmitz-Dumont. Dingl. Polyt. J. 1894, 293, 234.

See under XXIII., page 1227.

##### PATENTS.

*Improvements in the Treatment of and Improved Products from the Seed of the Locust Bean.* J. Pearson, Liverpool, and F. H. Taunton, Waterloo, Lancaster. Eng. Pat. 20,368, October 28, 1893.

THE kernel of the locust bean consists of two equal parts, which are secured together by a considerable thickness of matter different, as regards its construction, from that of the halves forming the kernel. If this "division" substance, which includes the germ, be removed from the kernel, the products made from the bean will keep for a much longer time than would otherwise be the case. The bean is accordingly decorticated, cracked, and cleansed to remove this division substance: it is then heated in water, milk, or other suitable liquid (3 or 4 gallons per pound of seed), for 5 or 6 hours with occasional stirring. The decoction is strained and the liquid extract concentrated, when it becomes useful as a lubricant or sizing material; if it be poured upon a hot plate a dry cake is obtained, and may be substituted for gelatin, isinglass, glue, &c. The solid matter strained from the extract is boiled with water until it becomes a firm jelly; this is suitable as a basis for soap, or as a toilet requisite, or as a gelatinous substance to be used in cookery.

The necessary machinery consists of rollers or grinding tools, such as emery wheels, designed to excoriate the division substance from the surface of the half bean.

—A. G. B.

*Improvements in Machines for Removing Stiff Water or Waste Hairs from Pelts.* L. Werner, New York, U.S.A. Eng. Pat. 17,710, September 18, 1894.

VARIOUS machines have been made for removing the stiff water or waste hairs from pelts, notably one which separates the soft fur from the water hairs by a blast of air, so that the shearing mechanism can remove the water hairs without damaging the fur. In the present modification the pelt is fed intermittently over the edge of a plate where the fur is turned aside by a brush, and the water hairs made to stand erect on the sharp edge of the plate; a metallic conductor, heated to redness by an electric current, is then brought in contact with these hairs so as to burn them away. The appropriate mechanism is described and depicted in the specification.—A. G. B.

## XV.—MANURES, Etc.

*The Valuation of Artificial Manures.* E. Henfeldt.  
Zeits. angew. Chem. 1894, 383.  
See under XXIII., page 1224.

*Estimation of Phosphoric Acid by the Citrate Method.*  
F. Glaser. Zeits. angew. Chem. 1894, 543.  
See under XXIII., page 1225.

*The Agricultural Value of certain Natural Phosphates.*  
G. Paturel. Comptes rend. 119, 1894, 119—122.

THE fertilising power of mineral (fossil) phosphates varies considerably, although so far as richness in phosphoric acid is concerned, there may be little difference between good and indifferent samples. In order to throw light on the matter, the author, starting with the idea that the assimilation of phosphoric acid is due to the solvent action of acids contained in the roots of plants and in the soil, has investigated the solubility of two different samples of phosphate in solutions of organic acids (citric and acetic acids). From the results it is concluded that the solubility of natural phosphates depends almost entirely on the amount of chalk (calcareous gangue) associated with the mineral; large quantities of chalk, by reason of its neutralising action on the acids of the soil, &c., having the effect of almost completely preventing solution. It is to this fact that the comparative inutility of certain phosphates (for instance, the granular phosphate from the Somme district, which contains much chalk) must be ascribed. Determinations of the commercial value of mineral phosphates should not, therefore, be based solely on their richness in  $P_2O_5$ ; account must be taken of the chalk present, and a reduction, proportionate to its amount, made in the price.—H. T. P.

## XVI.—SUGAR, STARCH, GUM, Etc.

*Losses by Concentrating Beetroot Juice.* Degener.  
Deutsch Zuckerind. 1894, 19, 1210.

DESTRUCTION of sugar occurs during the boiling process, and indeed principally by the protracted contact of the liquor with heated surfaces; the compounds formed in this way are of a dextrin-like nature, but their properties are as yet unknown. Their presence in technical products renders uncertain the analytical results obtained by the application of the raffinose formula (inversion method), but besides this, since they may be melassigenic at high temperatures and concentrations, a fresh difficulty is introduced in the calculation of the losses.—A. R. L.

*Decomposition of Sugar during Evaporation.* Strohm.  
Oesterr. Zeit. Zuckerind. 1894, 23, 456.

THE author again directs attention to this important question. Herzfeld has observed that the losses in practice (for example, in the boiling of heavy massecuites) are much greater than were those in his own laboratory experiments, in which much lower concentrations were dealt with. Claassen's values are perhaps also too low.—A. R. L.

*Purification of Beetroot Juice.* Bouvier. Sucr. indigène, 1894, 43, 724.

THE author explains the advantages arising from completely freeing the raw juice from albuminous matters and pulp, and recommends defecating the juice at a temperature of 80° C. with dry lime, which presents advantages.—A. R. L.

*Diffusion in Vacuo according to Heckmann's Process.*  
Mügge. Zeits. Zuckerind. 1894, 44, 596.

THE author has obtained good results with this process. He points out, however, that it was patented as long ago as 1867. Beauvais and Vier have also found that the process yields good practical results.—A. R. L.

*Purification of Beetroot Juice.* Sucr. indigène, 1894, 44, 2.

THE method advocated here is to treat the juice in the cold with dry lime, to saturate with carbonic acid in closed centrifugal machines either at a high or low temperature, and finally to remove the last traces of lime in pans.  
—A. R. L.

*Estimation of the Quantity of Crystalline Sugar contained in Massecuites.* Vivien. Sucr. indigène, 1894, 44, 234.

THE author effects this by systematic washing of the massecuites with saturated chloride. The percentage of water in the resulting white sugar is estimated, and this deducted, together with double its amount of sugar.—A. R. L.

*Yield of Sugar from Massecuite.* Claassen. Deutsch Zuckerind. 1894, 19, 956.

THE author shows conclusively that the yield of sugar is augmented much more rapidly when the purity of the massecuite is increased than when the amount of drainage syrup is diminished, and it is not, therefore, as a rule, to be recommended to allow the quotients of the drainage syrups to fall below 70—73, which can with certainty be accomplished by means of crystallisation in motion.—A. R. L.

*Grey Colour of Raw Beetroot Sugars.* Drenckmann.  
Zeit. Zuckerind. 1894, 44, 632.

THE grey colour and sensitiveness towards light of many massecuites and raw sugars met with during the last campaign is, among other causes, to be attributed to the presence of citric acid or citrate of iron. Citric acid and its amide were found frequently in large quantities in the products manufactured during the present year.—A. R. L.

*Valuation of Raw Sugars and After-products on the Basis of their Content of Crystalline Sugar.* Karcz. Oesterr. Zeits. Zuckerind. 1894, 23, 557.

THE so-called "Rendement" gives no idea of the real yield of refined sugar obtained in practice, because the latter does not depend upon such illusory values, but (other things being equal) it depends upon the amount of crystalline sugar contained in the raw sugar and upon the purity of the syrup which drains from it. By the aid of the author's glycerin process the amount of crystalline sugar may be determined, and from this the yield can be calculated.

A refinery, for example, worked 330,000 metre-centners of raw sugar of Pol. 95·3, water 1·8, ash 1·25, and organic matter 1·65 per cent., having thus a "Rendement" of 89·05. The percentage of crystalline sugar was, however, only 87·85, besides which the sugar contained 12·15 per cent. of syrup having a purity of 71·96. The known loss of sugar in the refinery was 0·69 per cent., due to decomposition by boiling, and 0·21 per cent. by washing, making a total of 0·90 per cent. on 100 parts of sugar or 0·61 + 0·19 = 0·80 per cent. on 87·85 parts of sugar. The remaining syrup comprised 12·15 + 0·61 = 12·76 per cent. of 67·96 purity, yielding according to experience 1·95 per cent. of after-product containing 1·56 per cent. crystalline sugar. The calculated yield is therefore 87·85 - 0·80 + 1·56 = 88·61 or 0·44 per cent. below the "Rendement." In reality the yield amounted to 286,950 metre-centners or 86·159 per cent. of white sugar, and 9,726 metre-centners of after-product containing 2·490 of crystalline sugar,



making a total of 88.649 per cent., thus 0.039 per cent. above the calculated; this difference would probably vanish when the after-product was manufactured into refined sugar.

—A. R. L.

*Inversion of Sugar by Glycerol.* Bordt. Zeits. Zuckerind. 1894, **44**, 701.

THE author shows that Donath's observations (this Journal, 1894, 823) are erroneous and that aqueous solutions of saccharose are more easily inverted when heated alone at 130° C. than in presence of glycerol.—A. R. L.

*Remarks on the Alcoholic Extraction of Beetroots.* Wiskirchen. Zeits. Zuckerind. 1894, **44**, 700.

See under XXIII., page 1226.

*Contributions to the Chemistry of Cellulose. 1. Cellulose, Sulphuric Acid and the Products of its Hydrolysis.* A. L. Stern. Proc. Chem. Soc. 1894, [112], 186.

See under Analyt. and Scient. Notes, page 1230.

*The Gravimetric Method of Estimating Cupric Reducing Sugar with Fehling's Solution.* C. Killing. Zeits. angew. Chem. 1894, 431.

See under XXIII., page 1226.

*Gravimetric Estimation of Cupric Reducing Sugars by means of Fehling's Solution.* A. Prager. Zeits. angew. Chem. 1894, 520.

See under XXIII., page 1227.

*The Influence of the Acid Molybdates of Soda and Ammonia on the Rotatory Power of Rhamnose (Isodulcitol).* D. Gernez. Comptes rend. **119**, 1894, 63.

See under XXIII., page 1226.

*A New Gravimetric Method of Estimating Glucose.* Fernand Gaud. Comptes rend. **119**, 1894, 478.

See under XXIII., page 1227.

*Determination of Glucose by Means of Alkaline Copper Solutions.* F. Gaud. Comptes rend. 1891, **119**, 650.

See under XXIII., page 1229.

*New Method of Estimating Cupric Reducing Power.* Alleino and Gaud. J. Pharm. Chim. 1894, **30**, 305.

See under XXIII., page 1229.

*Behaviour of Various Sugars towards Pure Yeast Cultures.* E. Fischer and H. Thierfelder. Ber. 1894, **27**, 2031.

See under XVII., page 1211.

*Behaviour of Normal and Basic Lead Acetate towards Sodium Carbonate, Sulphate and Phosphate in presence of Invert Sugar.* A. Bornträger. Zeits. angew. Chem. 1894, 521.

See under XXIII., page 1230.

*Ratio of Saccharimeter Degrees on the Ventzke Scale to Angular Degrees for Sodium Light.* E. Rimbach. Ber. 1894, **27**, 2282.

See under XXIII., page 1222.

#### PATENTS.

*Process for Purifying and Decolourising Saccharine Juices and Sugar Solutions.* C. Steffen, Vienna, Austria. Eng. Pat. 20,397, October 28, 1893.

THE patentee claims a process for obtaining decolourised and purified sugar solutions by treatment of the juices of

beetroot and cane-sugar manufacture after they have been defecated in the usual way, and in some cases saturated with carbonic acid, freed from defecation scum, and cooled below 60° C. (preferably, however, between 30 and 40° C., or lower) with sulphurous acid. He also claims the application of the same process to medium and thick juices of any concentration as well as to all syrups of the sugar manufacture; furthermore, the repeated application of the process to sugar solutions which have been partially decolourised, as also to the *chareux* or fine liquors of refineries. The sulphurous acid is employed in sufficient quantity so that the colouring and other non-saccharine matter are as much as possible made to form sulphite or acid sulphite compounds, being thereafter precipitated together with the acid in excess, by the action of suitable quantities of lime (or of baryta, strontia, alumina, &c.). The resulting neutral or slightly alkaline liquors being freed from the precipitates and, if desired, subsequently filtered through small quantities of animal charcoal, in order to obtain a still higher degree of decolourisation and purification.—A. R. L.

*An Elastic Composition for Moulds.* E. Hérisse, London. Eng. Pat. 5440, March 15, 1894.

FOUR parts of gelatin are softened in water, melted by heat, and added to one part of glucose previously dissolved in sufficient water to give it the consistency of "boiled sugar." The mixture is well stirred, allowed to set, again melted to remove air bubbles, and poured into suitable matrices. When cold, it is stained orange, by being dipped in a five per cent. solution of potassium permanganate, and after drying it is ready for employment as a flexible mould for casting articles of confectionery.—F. H. L.

*Improved Process for the Treatment of the Juice of Sugar Canes.* H. H. Leigh, London. From G. Cambray, Paris, France. Eng. Pat. 11,812, August 2, 1894.

THIS process consists in the subjection of cane juices to a double treatment with sulphurous acid (SO<sub>2</sub>), as follows:—The juice, after treatment with lime, is fed into tanks, where it is saturated to such an extent, either hot or cold, with sulphurous acid (SO<sub>2</sub>) that it allows the separation from it of the precipitates, either by decantation or by filtration under pressure. The clear juice is then again treated with sulphurous acid, until it is neutral or slightly acid, after which it is filtered through any type of mechanical filter. A perfectly colourless highly pure liquor is thereby obtained.

—A. R. L.

#### XVII.—BREWING, WINES, SPIRITS, Etc.

*Behaviour of Various Sugars towards Pure Yeast Cultures.* E. Fischer and H. Thierfelder. Ber. 1894, **27**, 2031—2037.

THE older experiments on the fermentability of various sugars were carried out with ordinary brewery yeast. A description of the results obtained by operating in this manner is to be found in the paper of Stone and Tollens (Liebig's Annalen, **249**, 257). According to the observations of these chemists, glucose, fruit sugar and galactose are completely fermentable; arabinose is, however, unfermentable. Sorbose is also stated to ferment but slowly and incompletely with ordinary yeast. The investigations of Hansen have shown that the yeasts formerly employed in the industry are mixtures which, by systematic cultivation are separable into a large number of sharply-defined varieties. The behaviour of a number of pure cultivations of yeast towards glucose, saccharose, maltose, and milk sugar has been studied by Hansen himself (see Jørgensen, Mikroorganismen der Gährungsindustrie, page 131). According to his observations, we have to do with three different classes of Saccharomycetes, of which the members

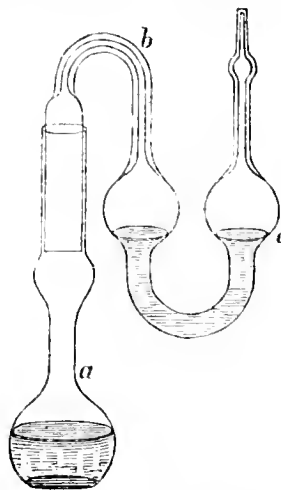


of the first and most numerous, ferment glucose, saccharose, and maltose. In this class are included *Saccharomyces cerevisia*, I.; *S. Pastorianus*, I., II., and III.; and *S. Ellipsoides*, I. and II. The second class embraces the class whose members ferment glucose and saccharose but not maltose (*S. Marxianus*, *S. Ludwigii*, and *S. Eriguns*). The third class is represented by a single species (*S. Membranifolius*), which, strangely enough, does not exhibit the phenomenon of alcoholic fermentation. Milk sugar is not affected by any of the above-mentioned *Saccharomyces*.

*S. apiculatus* ferments glucose, d-mannose, and d-fructose (Chemer, Zeits. Biol. 29, 525), but not galactose (Voit, *ibid.* 29, 149), saccharose, milk sugar, and maltose (Hansen, Anthor. Zeit. physiol. Chem. 12, 563). Finally, Daclaux (Annal. Institut. Pasteur, 1, 573), Adametz (Centralbl. Bact. und Paras. 5, 116), Grolenfeld (Fortschr. d. Mediz. 1889, 121), Byerinck (Centralbl. Bact. und Paras. 6, 44), and Kayser (Annal. Inst. Pasteur, 1891, 395), have described some yeasts which ferment milk sugar.

The authors have investigated the action of 12 different yeasts on various sugars, including some of the synthetical sugars and glucosides obtained by Fischer. In order to economise the material, the experiments were carried out on a small scale, making use of the flask which is shown in the cut in its actual size. The flask *a*, having a capacity of about 1 cc., is closed with a wadding stopper, sterilised, and about two-thirds filled with a mixture of equal parts of a 20 per cent. aqueous sugar solution and yeast extract. The latter is prepared by boiling well washed and pressed pure yeast with four parts of water, and, after repeatedly filtering the extract so obtained, adding a small quantity of citric acid thereto. To the carefully sterilised liquid about 0.013 gm. of the pure yeast cultivated on beer-wort gelatin is then added by means of a platinum wire furnished with a loop, observing the usual precautions; the tube *b*, which is filled to the mark *c* with baryta water and also sterilised, is then inserted, the joint being rendered gas-tight by smearing the inner portion with vaseline and melting paraffin round the outer portion. The apparatus so charged is allowed to remain 3—10 days in an incubator, the temperature being

maintained at 24°—28° C. In all cases when the sugar is unfermentable, and even when no sugar is present beyond



that contained in the yeast extract, the surface of the baryta water is covered with a thin layer of barium carbonate. If, however, the carbohydrate be fermentable, the baryta water not only becomes turbid, but the whole of the barium is precipitated as carbonate, and finally no reduction by Fehling's solution takes place by the liquid which contained the sugar in solution. In cases in which the material is not directly fermentable, but only so after hydrolysis, such as with the glucosides, and where, on this account, fermentation proceeds slowly, and probably with a limited quantity of yeast is never complete, the amount of evolved carbonic anhydride is always sufficient to indicate whether fermentation has or has not taken place.

The results obtained are shown in the following table:—

	d-Mannose.	d-Fructose.	d-Glucose.	d-Talose.	L-Mannose.	L-Glucose.	Sorbose.	L-Arabinose.	Rhamnose.	α-Glucoheptose.	α-Glucooctose.	Saccharose.	Maltose.	Milk Sugar.	Methyl Glucoside. <sup>c</sup>	Ethyl Glucoside. <sup>c</sup>	Glucose Resorcinol.	Glucose Pyrogallol.	Glucose Ethyl Mercaptal.
<i>S. Pastorianus</i> I. . .	+++	+++	+++	..	..	..	..	..	..	..	..	+++	+++	..	+	+	..	..	..
<i>S. Pastorianus</i> II. .	+++	+++	+++	..	..	..	..	..	..	..	..	+++	+++	..					
<i>S. Pastorianus</i> III. .	+++	+++	+++	..	..	..	..	..	..	..	..	+++	+++	..					
<i>S. Cerevisia</i> I. ....	+++	+++	+++	..	..	..	..	..	..	..	..	+++	+++	..					
<i>S. Ellipsoides</i> I. . .	+++	+++	++	..	..	..	..	..	..	..	..	+++	+++	..					
<i>S. Ellipsoides</i> II. .	+++	+++	++	..	..	..	..	..	..	..	..	+++	+++	..					
<i>S. Marxianus</i> . . . .	+++	+++	+++	..	..	..	..	..	..	..	..	+++	+++	..					
<i>S. Membranifolius</i> .	..	..	..	..	..	..	..	..	..	..	..	..	..	..					
Brewery yeast <sup>2</sup> . . .	+++	+++	+++	..	..	..	..	..	..	..	..	+++	+++	..	+	+	..	..	..
Distillery yeast . . .	+++	+++	+	..	..	..	..	..	..	..	..	+++	+++	..	+	+	..	..	..
<i>S. productivus</i> . . .	+++	+++	..	..	..	..	..	..	..	..	..	+	+++	..	+		..	..	..
Milk sugar yeast . .	++	+++	+	..	..	..	..	..	..	..	..	+++	..	+++	..				

Explanation of the table:—

.. denotes that the solution no longer reduces Fehling's solution at the end of eight days; thus fermentation was complete.

++ denotes a very slight reduction after eight days; thus almost complete fermentation.

+++ denotes distinct reduction after eight days, but undoubted fermentation.

.. denotes no fermentation.

<sup>1</sup> Obtained from C. E. Hansen.

<sup>2</sup> This was Töberg yeast, or yeast No. 10 (see Irmsch. Wochensh. Brauw. 1891, No. 39—46); P. Lindner, *ibid.* 1893, 692; 1894, 381.

<sup>3</sup> This is described in the literature as yeast No. 128, variety 2 (see Zeit. Spiritusind. 1892, 361).

<sup>4</sup> Obtained from B. Yerinck.

<sup>5</sup> This yeast has not yet been morphologically defined.

The test after complete fermentation was omitted in these cases, as the detection of the glucoside, which only reduces Fehling's solution after hydrolysis, is rendered difficult by the presence of yeast glycogen.

<sup>6</sup> After 11 days the sugar had all disappeared.

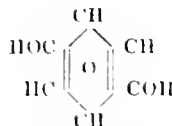
These experiments, so far as they are a repetition of the results of others, confirm all the older observations with the exception of that of Stone and Tollens, who found that sorbose was fermentable, although incompletely so—a result which must have been due to the presence of foreign organisms in the yeast. Sorbose is unaffected by pure yeast.

The authors then discuss the bearing of these results on the configurations of the various sugars.

It is shown, furthermore, that it is possible to ferment away, by means of *S. Pastorianus* L., the whole of the glucose from mixtures of that sugar with *D*-mannose, the latter remaining unattacked by the yeast at the end of three months.—A. R. L.

#### Maltol. J. Brand. Ber. 27, 806–810.

An aqueous solution of the so-called "crystal malt" gives a similar reaction to salicylic acid with ferric chloride, leading in the past to the erroneous conclusion that beers made from this class of malt contained salicylic acid. On carefully heating crystal malt in a test-tube the whole of the substance which gives this reaction is volatilised, and hence it is that patent black malts do not give this coloration with ferric chloride. By treating the aqueous extract of crystal malt with ether it was obtained as needle-shaped crystals, melting at 148–150° C., and soluble in water. It yields no coloration with Millon's reagent—a reaction which differentiates it from salicylic acid. The author prepared it in large quantity from the steam issuing from the roasting cylinders employed in the manufacture of "coffee-malt." By this method it was obtained as large, shining, yellowish leaflets which were odourless, and had a melting point of 159° C. They were very soluble in hot water, in chloroform, and in acetic acid. Analysis showed its formula was  $C_6H_8O_3$ . It shows some phenol characteristics, gives some but not all the aldehydic reactions, and is not a true acid, so that the author concludes it is derived from a sugar by the abstraction of water, and gives the following as the formula of "maltol":—



Maltol is thus of great interest, as it is allied to cincol and its derivatives, and also to the terpenes.—J. G. W.

#### Employment of Selected Yeasts in Wine Production. Charles Fabre. Comptes rend. 119, 1894, 373–375.

The author carried out experiments during 1891–93 in order to find to what extent the character of wine from a given must, may be altered or improved by the use of selected yeasts. The musts used in the laboratory had been previously heated to 70° C., and were sown with selected yeasts obtained from the Institut de la Claire. For instance, must from Cabernet-Sauvignon was sown with yeast from Margaux; Sauterne must with *Sémillon* yeast; and Vongont must with yeast from Pineau noir.

The results obtained show that the same must behaves differently with different yeasts, in spite of the sterilisation of the must at 70° C., but the flavour of a wine, although depending partly on the yeasts used, also largely depends on the composition of the must. Experiments were conducted on the large scale, using 3 hectols. of must, and the following conclusions are to be drawn therefrom:—

(1.) A selected yeast will not yield a fine wine with every must.

(2.) The must used should be obtained from grapes which were grown in and were well acclimatised to the district from which the selected yeasts used were obtained.—T. A. G.

#### Phenomena accompanying the Dialysis of the Cells of Beer Yeast. E. Ommus. Comptes rend. 1894, 119, 179–180.

If a solution of sugar in boiled water be separated from an aqueous yeast extract by means of a membrane of parchment paper, the sugar is found to be inverted after 15–20 minutes. No cells can, however, be discerned under the microscope, but only some microzymes; and it is only after two or three hours that a few isolated yeast cells make their appearance. A solution of sugar freely exposed to the air was found to undergo no alteration during this period. In order to completely exclude air, the author placed a sugar solution in a small metallic cup, the orifice of which was covered with parchment paper held in position by a metallic band. The solution was boiled and the whole treated with beer yeast. On the following day the sugar was inverted, and small isolated cells could be discerned in the liquid.

These experiments show that beer yeast secretes a dialysable substance, and that the inversion of sugar precedes the appearance of new cells. The medium is at first modified by the enzyme, and only after this process does it become nutritive for whatever can give birth to the cells.—A. R. L.

#### Yeast Gum. E. Salkowski. Ber. 27, 925–926.

The author considers that his formula for this body,  $C_{12}H_{22}O_{11}$ , is more probably correct than Hesseland's,  $C_{12}H_{20}O_{10}$ , as the products of its hydrolysis were found by Hesseland to be *D*-mannose and a little dextrose, which renders it probable that his preparation was not homogeneous.—J. G. W.

#### The Power in Ferments of becoming Inured to the Action of Antiseptics, and the Influence of the Inurement upon their Chemical Work. J. Filron. Comptes rend. 119, 169.

In his preceding work the author showed that the inurement of brewer's yeast to the antiseptic action of fluorine compounds, brings about a manifest change in the chemical work effected by these organisms of the yeast; such change, indeed, that the secondary fermentation, which consists in the formation of glycerol and succinic acid from the sugar, diminishes, gradually and proportionally, according as the yeast has become more or less accustomed or inured to fluorine antiseptics; so that after a sufficiently lengthy inurement, its chemical work consists almost exclusively in the transformation of sugar into alcohol and carbon dioxide.

The author has now extended his work to the cases of the lactic acid and butyric acid ferments, and to that of *mycoderma aceti* (acetic acid ferment).

In the first place the author established—by inoculating with pure cultures of the lactic and butyric ferments, sterilised malt infusions, to which varying quantities of hydrofluoric acid had been added—that 25 and 12 mgrms. of hydrofluoric acid per 100 grms. of infusion were respectively sufficient to arrest the lactic and butyric fermentations.

He then proceeded to inure the organisms to the hydrofluoric acid by a series of cultures, in which the amount of hydrofluoric acid present was gradually increased until the organisms were able to withstand the antiseptic (inhibitory) influence of 30 mgrms. of hydrofluoric acid in 100 grms. of malt infusion.

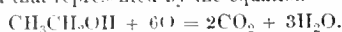
In such infusions the ferments produced 1.5 grms. of lactic acid and 0.86 grm. of butyric acid per 100 grms. of infusion—quantities which are approximately equal to those which, with like environment, but with absence of hydrofluoric acid, are produced by the respective ferments. Microscopic examinations showed that, just as was the case with yeast, less than the normal amount of new cells had been formed; so that, in the presence of hydrofluoric acid, though their powers of increase had been curtailed, the fermenting power of the cells had proportionally increased.

In ordinary lactic and butyric fermentations, just as in alcoholic fermentation, secondary products are undoubtedly

formed, and that this is so may be proved by estimation of the sugar before and after fermentation; the acid formed being found not to account for the whole of the sugar which has disappeared. Little, however, is as yet known about these secondary products of the lactic and butyric fermentations.

With the *mycoderma aceti* fermentation the change in the character of the chemical work accomplished is much more strongly marked. Here the author employed a mixture containing in 100 parts, 6 parts of alcohol, 1 of acetic acid, and 5 of a sterilised malt infusion, the specific gravity of which was 7° B. In the presence of 50 mgrms. of hydrofluoric acid, such a mixture, after being inoculated with the acetic ferment, developed no mycelium, nor was there increase in the amount of acetic acid; in control experiments, where the antiseptic was absent, the development of the *mycoderma* was quite regular; the production of acid normal.

A series of cultures was now undertaken with increasing quantities of hydrofluoric acid present, and it was noted that as the increment (power of resistance to the antiseptic) of the fungus increased, so proportionally the oxidation approached that represented by the equation—



A series of determinations of the alcohol and acetic acid, in liquids which had been submitted to acidification during three days, gave the following figures per 100 grms. of liquid:—

HF Present.	C <sub>2</sub> H <sub>5</sub> HO Remaining.	CH <sub>3</sub> .COOH Formed.
0·0	1·55	4·32
0·025	1·75	3·27
0·050	1·95	1·31
0·120	1·40	0·12

So that, as against the disappearance of 100 parts of alcohol, there is an appearance of acetic acid of 97·08 parts (HF present = 0), of 76·94 parts (HF = 0·025), of 32·34 parts (HF = 0·05), and of 2·62 parts (HF = 0·120) respectively.

In conclusion the author states that the study of the increment of ferments to antiseptics has a very special interest from the point of view of bacteriological pathology; he suggests that perhaps certain pathogenic ferments may lose some of their virulence under an antiseptic treatment by fluorine compounds, and he further states that he has already commenced work in this direction.—E. R. B.

*Ratio of Saccharimeter Degrees on the Ventzke Scale to Angular Degrees for Sodium Light.* E. Rimbach. Ber. 1894, 27, 2282.

See under XXIII., page 1222.

*Removal of the Lead from Musts and Wines which have been Clarified with Lead Acetate.* A. Borntraeger. Zeits. angew. Chem. 1894, 554, 579, and 583.

See under XXIII., page 1230.

#### PATENTS.

*Improved Manufacture or Production of Extracts from Malted Grain.* F. Faulkner, Birmingham, and J. E. J. Johnson, Old Ford. Eng. Pat. 23,846, December 11, 1893.

The patentees claim the invention of certain fluid extracts from malted grain denoted respectively:—*Digestive malt extract*, *saccharine malt extract*, and *amide yeast food*.

In the manufacture of so-called *digestive malt extract* one quarter of malt may be heated at 110° F. for 50 minutes with, say, 108 gallons of water. The extract so obtained is separated from the draff or grain, and the liquid concentrated *in vacuo* at a temperature not exceeding 118° F. until it has the consistency of about a 14-lb. syrup, or 14 lb. to the gallon. This extract is suitable for medicinal purposes, and also for use in breweries and distilleries as a nutritive material for yeast.

*Saccharine malt extract* is prepared by mixing the primary or partially extracted draff from the last-mentioned operation, with about 15 or 20 per cent. of fresh crushed malt, and adding the mixture to water in the proportion of about one quarter of mixed draff and fresh malt to about 72 galls. of water at about 110° F., maintaining the mash at this temperature for about 50 minutes, and finally raising it to 150—160° F. for 10—15 minutes. The liquor filtered from the draff is then concentrated to a 14-lb. syrup, and thus forms an extract practically equivalent to entire malt extract existing in a brewery wort, but without the enzymes.

*Amide yeast food* is manufactured from the draff left in the second operation (manufacture of saccharine malt extract) by extracting about one quarter of it under pressure with, say, 90 galls. of water at a temperature of 250° F. for 2—3 hours. The filtered liquor is then concentrated as in the other cases, the finished product being specially suitable for brewery operations.—A. R. L.

*Improvements in Drying Brewers' and Distillers' Grains and the like, and in the Means or Apparatus employed therein.* H. Fielder, South Hampstead and the Dailuaine-Glenlivet Distillery, Limited, Carroo, Strathspey, N.B. Eng. Pat. 24,949, December 28, 1893.

THE patentees claim the combination or arrangement and use, for drying distillers' grains and the like, of apparatus comprising a series of communicating tortuous unjacketed chambers, through which the material to be dried is caused to travel by means of scoops radially flanged on one side thereof, carried on rotary shafts suitably mounted and driven. Through the drying chambers mentioned heated air is caused to pass in the earlier sections of the chambers against, or alternately with and against, the flow of the material, and in the later sections, where the final drying is effected, *with* the flow of the material. There are also claimed, means for heating the drying air by tubes below the drying chambers, which tubes effect direct transmission of heat thereto, as well as by communication to the interiors thereof. Furthermore, the combination or arrangement and use of apparatus for drying brewers' or distillers' grains and the like, whereby the amount of inlet of hot air to each section of the chambers can be separately regulated, and the direction of the currents of air and of the flow of the material is alternately the same and in opposite directions. Access to the material is facilitated by removable covers, and increased surface is exposed to the heat of radiation of the air-heating tubes.—A. R. L.

*Improvements in the Treatment of Yeast.* G. G. Cave, Penny Stratford, Bucks. Eng. Pat. 2889, February 9, 1894.

THIS specification relates to a process whereby yeast may be preserved, it is said, for any length of time in an inert state without its vitality being destroyed. The washed yeast is mixed with finely pulverised malted grain, which has been roasted, in the proportion of five parts of yeast to six parts of malt, after which the dough is dried by the action of heat. The grain used is preferably barley, but may also be rye, oats, maize, &c., which, after being malted in the usual way, is roasted or carbonised in a manner similar to that adopted in the case of coffee beans, and subsequently ground to an almost impalpable powder.—A. R. L.

# XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY AND WATER PURIFICATION, DISINFECTANTS.

## (A.) CHEMISTRY OF FOODS.

### PATENTS.

*Improved Manufacture and Production of Extracts from Malted Grain.* F. Faulkner, Birmingham, and J. E. J. Johnson, Old Ford. Eng. Pat. 23,816, December 11, 1893.

See under XVII., page 1214.

*Improvements in Food Preparations for Man and Animals.* H. P. Madsen, London. Eng. Pat. 1772, January 26, 1894.

THIS is an extension of previous patents (this Journal, 1886, 675). For food for man 150 parts of blood mixed with 600 parts of water are boiled while well stirred; when boiling, sufficient vinegar is added to "cause extraction of the albumen, the fibrin, and the alkaline and other salts." After sifting, the parts remaining on the sieve are mixed with condiments and made into a dough, which is fashioned into biscuits and baked at 200°–300° F.

A number of recipes, in which the above blood-substance is mixed with other foodstuffs to make food suitable for diverse animals, is given. A drink for man is made by mixing "albumin fibrin and the salts obtained from blood" with water, flavouring with cinnamon, orange juice, pineapple juice, cardamom, and sugar, boiling and filtering.

—A. G. B.

*Improvements in Apparatus for Use in the Sterilisation of Materials in Bottles, Jars, or other Vessels.* A. Hüssener, Gelsenkirchen, Germany. Eng. Pat. 4178, February 27, 1894.

THE inventor claims the hermetical sealing of vessels, in which foods have been sterilised, by means of the pressure of the outer air. The vessels are placed on a base plate and suitable covers are held down on their mouths by spiral springs. The apparatus is immersed in water up to the necks of the vessels and heated. This causes the central spring to lengthen and lessen the pressure, so as to allow the heated air to escape from the vessels. On removing the source of heat, the pressure is again increased, and when the vessel is cold, the external pressure of the air is sufficient to keep the covers in position.—L. de K.

*Improvements in the Manufacture of Feeding Cakes Suitable for Human or Animal Use.* A. W. Rehnström, Målhammer, Rekarne, Sweden. Eng. Pat. 5373, March 14, 1894.

SKIMMED milk is more or less concentrated and mixed with residues obtained in expressing vegetable oils, or with meal or bran of cereals, maize, rice, peas, vetches, brewers' grains, beet-pulp, reindeer-moss, or oil-seeds; the mass is made into cakes and baked. The most concentrated foods will contain the milk substance evaporated to dryness, and the proportion added will vary with the requirements of the animal in respect of nitrogen.—A. G. B.

*Improvements in Pasteurising Apparatus for Milk, Cream, or the like.* W. Paaseh, Horsens, Denmark. Eng. Pat. 18,143, September 24, 1894.

THE apparatus consists of a pasteurising vessel immersed in a steam-bath, and containing a rotatory agitator which, by centrifugal action, shall raise the milk to a higher level than that of influx. The agitator mentioned is mounted so

as to rotate on a vertical axle, and it is furnished with broad blades or pallets, so that in the agitating, centrifugal force is communicated to the milk, and the latter whilst rotating, is thus raised above the level of the vat, without producing foam or froth.—A. G. B.

## (B.)—SANITARY CHEMISTRY.

*Examination of the Products of Combustion of Coal Gas when Burned by Argand and by Auer Burners respectively.* N. Crochant. Comptes rend 119, 146.

THE author has minutely examined the products of combustion for carbon monoxide in the two cases, both chemically and physiologically.

The chemical examination consisted in passing the products of combustion, first carefully freed from carbon dioxide, over red oxide of copper and then through baryta water.

In the physiological examination a dog was caused to breathe the products of combustion for half an hour; samples of its blood were analysed for combustible gas before and after breathing the products of coal-gas combustion.

The results obtained were as follows.—For the Argand burner, by the chemical process, 1.2 cc. of  $\text{CO}_2$  from 73 litres of the products of combustion, equal to a trace of  $\text{CO}$ , say,  $\frac{1}{75,000}$ ; by the physiological process, nil. For the Auer burner, "which gives so white and dazzling a light," they were very different. Here 60 litres of combustion products gave 23.2 cc. of  $\text{CO}_2$ , equal to 23.2 cc. of  $\text{CO}$  or of formene, or a mixture of these two substances—say  $\frac{1}{23.2}$  of the volume of the products of combustion. By the physiological method 100 cc. of blood yielded 1.3 cc. of  $\text{CO}$ , equal to  $\frac{1}{1300}$  of the air inspired.

It is pointed out that these results are most interesting from a hygienic point of view; the products of combustion of lighting gas should be carefully avoided, especially those emitted by the Auer burner.—E. R. B.

*Attempts to Estimate Sulphur Compounds in the Atmosphere.* W. H. Oates. Proc. Chem. Soc. 1894 [113], 218.

See under XXIII., page 1222.

### PATENTS.

*Improvements in connection with the Precipitation of Sewage and Polluted Waters, and in Precipitation Tanks, and in Filtering Apparatus, and in Filter Beds for the Purification of Sewage and Polluted Waters.* F. P. Candy, London. Eng. Pat. 19,560, October 18, 1893.

THE sewage first passes through a settling-tank to remove heavy matter, and then, after mixing with the precipitant or deodoriser, through the inventor's improved mixing-tank. The latter is furnished with variously arranged partitions bearing flanges so placed as to cause as much disturbance of the liquid as possible. The sewage should flow at such a rate that any sediment is carried through. The improvement claimed in the filter-tank is that the bed can easily be cleaned by an upward flow of water of small pressure without disturbing the filtering material. This is accomplished as follows:—On an iron plate with large perforations rests a non-corroding sheet with smaller holes, and on this is placed a sheet of copper gauze. The three plates are fastened to a flange on the side of the tank, so that all liquid must pass through the holes. Resting on the gauze is a coarse layer of iron ore, above which comes a layer of coarse sand, and on the top is a layer of coal or coke granules. The difference between their specific gravities keeps the layers in their proper positions when being cleaned. The large openings of the lowest plate may be provided with automatic valves, on the principle of a ball-cock, to prevent any return of the sewage.—L. de K.

*Improvements in Mechanism for Controlling and Regulating the Discharge of Liquids from Tanks.* W. G. Strype, Dublin. Eng. Pat. 22,529, November 24, 1893.

See under L, page 1180.

*Improvements in the Precipitation of Compositions adapted for Use in the Treatment of Sewage and Foul Waters.* H. B. Sharp and J. B. Petrie, both of Smithy Bridge, Lancaster. Eng. Pat. 25,049, December 30, 1893.

Poor iron-ore largely, if not mainly composed of alumina, is reduced to a very fine powder and then thoroughly mixed up with water. Hydrochloric and sulphuric acids are next added and the mixture boiled with constant stirring until the solution is so concentrated that on cooling it will set in blocks or masses. At this stage the contents of the boiling-pot is run into suitable moulds.

The inventors claim that this preparation is superior to either sulphate of iron or sulphate of aluminium when applied to the disinfecting of sewage.—L. de K.

*Improvements in and connected with Iron Compounds for Use in Precipitating Sewage, Purifying Waste Liquors, Colouring, and the like Purposes.* I. Levinstein, Manchester. Eng. Pat. 1415, January 23, 1894.

SUITABLE bisulphates and oxides of iron, especially ferric oxide, are heated together to about 300° C. with stirring, until quite dry, water having been previously added to promote the reaction. The preferred materials and proportions are equal parts of sodium acid sulphate and ferric oxide, with one-fourth part water. It is defined that the term "bisulphate" "is intended to cover and include sulphates having an additional molecular proportion of sulphuric acid mixed therewith or added thereto." The compound, obtained as described, containing per salts of iron, is used for the purposes stated in the title.—E. S.

*Improvements in or relating to the Recovery of Sediment from Clarifiers.* O. Schmidt, Berlin, Germany. Eng. Pat. 2051, January 30, 1894.

These improvements are included in a process for the separation of sediment in a thick and semi-fluid state from water in clarifying apparatus, with a siphon-vessel or deep well, so arranged that the sediment in the mud-collector is removed by an exchangeable pump working in the collector itself and driven from above, whilst at the same time it is prevented from accumulating and consolidating itself on the walls of the mud-collector, by the use of an agitator rotating round the axis of the pump.—L. de K.

*An Improved Continuous Apparatus for the Sterilisation of Water or other Liquid by Ebullition.* E. Herscher, Paris, France. Eng. Pat. 16,938, September 5, 1894.

The apparatus consists essentially of a reservoir in which a constant level is maintained by a ball-cock. The water passes thence through a valve, which is only opened when the water in the boiler reaches 100°, into the outer part of the condenser, and from there, part passes to the boiler and another part flows away through a waste-pipe. From the boiler, water and steam pass through a pipe, which is above the level of the reservoir into the inner part of the condenser. The boiler contains a system of bells and inverted bells to keep the water for some time at boiling point before allowing it to pass into the condenser, and the high-level pipe ensures the temperature, whilst the steam-worked valve prevents loss of water when the apparatus is not in use.

—L. de K.

## (C).—DISINFECTANTS.

### PATENTS.

*An Improved Anti-fouling Composition for the Prevention of Marine Growth in or on Wood or Iron.* R. Hingston and J. P. Wernegreen, Brisbane, Queensland. Eng. Pat. 17,662, September 17, 1894.

SIXTY-NINE pounds of purple oxide of iron are ground into 3 gallons of boiled linseed oil, and, in succession, 10 lb. of "patent driers," 5 lb. of mercuric chloride in fine powder, and 1 lb. of mercuric oxide are added and the whole well mixed in a wooden or iron vessel.—F. H. L.

## XIX.—PAPER, PASTEBOARD, Etc.

### PATENTS.

*Improvements in the Manufacture of Cellulose Pulp.* J. Y. Johnson, London. From P. Sobège, Paris, France. Eng. Pat. 24,503, December 20, 1893.

THE inventor claims the addition of tannic acid to the cellulose intended for sizing with gelatin, and the employment of celluloses containing tannin, either in a natural state or in the state of tan, for making pulp for the production of paper or card, in which the gelatin is rendered insoluble, inaptesible, and very slightly liable to swell and soften by the action of damp.—J. C. C.

*Improved Process for Making Paper Pulp Boards or Paper in Imitation of the Grain, Knots, or Burls of Natural Woods.* W. N. Cornell, Brownville, New York, U.S.A. Eng. Pat. 13,790, July 17, 1894.

THIS process consists in winding convolutionally on a paper machine a pulp board of less than the desired thickness; then in adding to the partially finished pulp board a separate piece or pieces of pulp of greater density, or drier, or of a darker colour than the pulp board; then finishing by winding to the required thickness, whereby the pieces of pulp cause the pulp board to slip and wrinkle on the forming roll; finally in removing the board from the machine, drying, and sandpapering it. An article is produced, which is said to be similar in appearance to natural wood.—J. C. C.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*The Emetics.* Paul Adam. Bull. Soc. Chim. 1894, 11, 597—602.

IN the emetics (of which tartar emetic is the type) the antimonyl (SbO), arsenyl (AsO), "or boryl (BO)" groups are generally regarded as being substituted in the carboxyl group of the organic acid. The author brings forward various facts in support of the view, originally put forward by Jungfleisch in 1883, that the substitution really takes place in the alcoholic or phenolic hydroxyl.—T. E.

*The Alkaloids of Corydalis Cava.* Corybulbine. J. J. Dobbie and A. Lander. *Proc. Chem. Soc.* 1894 [143], 217.

This alkaloid is obtained from the commercial corydaline supplied by Schuchardt of Grlitz, by either of the following methods:—

1. The crude material is dissolved in hydrochloric acid, and the solution mixed with excess of soda, which throws down the corydaline only. The filtrate is then saturated with carbon dioxide, when the corybulbine separates out.

2. The crude corydaline is repeatedly exhausted with hot alcohol, which dissolves out the greater part of the corydaline. The residue is dissolved in a large quantity of boiling alcohol, from which the corybulbine separates on cooling as an exceedingly fine crystalline powder. It is purified by conversion into the hydrochloride, which, after repeated recrystallisation from water, is decomposed by ammonia.

Corybulbine is nearly insoluble in water, soluble with difficulty in methyl and in ethyl alcohol, and insoluble, or nearly so, in ether. It dissolves readily in carbon bisulphide, chloroform, and hot benzene. It is also soluble in solutions of the caustic alkalis. An alcoholic solution of the alkaloid rapidly reduces a warm solution of silver nitrate. When heated, corybulbine softens at 210°, but does not melt till 238°–240°. A solution of the alkaloid in chloroform is dextro-rotatory.

Corybulbine has the formula  $C_{21}H_{25}NO_4$ . The hydrochloride,  $C_{21}H_{25}NO_4 \cdot HCl$ , is obtained in clusters of prismatic needles by dissolving the alkaloid in hot dilute hydrochloric acid. This salt is remarkable for the difficulty with which it dissolves in water.

The acid sulphate,  $C_{21}H_{25}NO_4 \cdot H_2SO_4$ , is prepared in the same manner as the hydrochloride. It dissolves with difficulty in hot water, from which it slowly separates on cooling, in long prismatic crystals.

*Corydaline. Part IV.* By J. J. Dobbie and A. Lander. *Proc. Chem. Soc.* 1894 [143], 218.

It is probable that corydaline is an alkaloid of the same type as papaverine, narcotine, and hydrastine, containing an isoquinoline and a benzene nucleus, but of simpler constitution than these alkaloids, inasmuch as the two nuclei appear to be united directly to one another and not through an intervening carbon-atom.

*Alkaloids of the Bark of the Pomegranate Root.* G. Cianaicari and P. Silber. *Ber.* 1894, 27, 2850–2861.

THE paper deals with the alkaloid "*Norgranatoline*" previously obtained by the authors by the reduction of granatoline with phosphorus and hydriodic acid (this *Journal*, 1894, 417). This product forms small white needles, melting between 50 and 60°C., which absorb carbon dioxide very readily from the air. So greedily does this absorption take place that the melting point could not be determined. Distilled over zinc-dust,  $\alpha$ -propyl pyridine is formed, a decomposition corresponding to one in which  $\alpha$ -ethyl pyridine is formed from norhydratropidine.

Granatoline, upon oxidation with an alkaline potassium permanganate solution, forms *Norgranatoline* under similar conditions to the formation of trepigenine from tropine. The properties and some derivatives of this new base are described. Distilled over zinc-dust, pyridine is obtained. The paper concludes with remarks on the probable constitutional formula of granatoline and its relation to tropine, of which it is the next higher homologue.

The group of bases obtained by the authors from Granatoline are now:—

Granatoline.....	$C_{17}H_{19}ON \cdot CH_3$
Granatoline.....	$C_{17}H_{19}OH \cdot N \cdot CH_3$
Granatoline.....	$C_{17}H_{19}N \cdot CH_3$
Granatoline.....	$C_{17}H_{19}N \cdot CH_3$
Norgranatoline.....	$C_{15}H_{17}(OH)N \cdot H$
Norgranatoline.....	$C_{15}H_{17}N \cdot H$
Norgranatoline.....	$C_{15}H_{17}N \cdot H$

Norgranatoline is isomeric with A. W. Hofmann's Coniceine.—C. A. K.

*Piceol. A Glyceride obtained from the Root of the Pine.* *Pharm. Ztg.* 1894, 39, 119, 120.

THIRTY hopped pine twigs are boiled for a short time with water. From a grams of solid material, the particles of twigs, filtered off, and digested for an hour in ether. The extract is poured into a beaker, successively with 10 cc. of benzene and lead acetate, and the precipitate produced by the addition of concentrated sulphuric acid. The resulting solid is exactly neutralised with sodium carbonate. The solid is then again subjected to a similar weight to one third of the original weight in ether, which is exhausted by repeated extraction. The ether extract is then distilled off, and the residue is taken up in alcohol, again evaporated to expel the last traces of acetone, and finally dissolved in a small quantity of warm absolute alcohol. The liquid is then set to a pasty mass, which is washed by suction with a solution of alcohol camphoraceous residues are thus removed and dissolved in boiling absolute alcohol or water. The solid mass, on cooling, deposits crystals of *piceol*. The yield is about 10 per cent.

*Piceol*,  $C_{10}H_{16}O_2$ , crystallises from water with one molecule of water of crystallisation, anhydrous from alcohol, and in either case forms a very fine, needle-shaped, transparent, of boiling water, for cold water, 500 of cold water, 100 of boiling absolute alcohol. It possesses a bitter flavour. *Piceol* is dextro-rotatory,  $\alpha_D^{20} = +84$  in  $CHCl_3$ , and  $+78$  in alcohol solution. Anhydrous *piceol* melts at 104°C.

*Reactions.*—A small fragment of *piceol* dissolved in strong sulphuric acid imparts a coloration of a reddish-brown tint. This fact serves to distinguish it from *pinic acid*, which yields a violet coloration. *Piceol* is not precipitated by tannin or basic lead acetate, but with the addition of lead acetate a precipitate of the compound  $C_{10}H_{16}O_2 \cdot Pb(OAc)_2$  is obtained. Under the influence of emulsion of lime acids, *piceol* is split up into glucose and *pinic acid*.



*Piceol* melts at 109°C. It is soluble in 100 parts of cold, or 14 of boiling water. From the latter it separates, on cooling, in oily drops which gradually become crystalline. With ferric chloride, its solution yields a violet coloration. *Piceol* appears to be a monatomic phenol and forms crystalline compounds with caustic alkalis, acetic and benzoic acids, &c.

—H. T. P.

*Conine Alkaloids.* R. Wolfenstein. *Ber.* 27, 2611–2615.

A SAMPLE of Merck's pure conine, boiling at 165–169°C., showed an unusually high rotatory power, which was found to be owing to the presence of methyl conine. The latter was isolated from the mother-liquor left after separation of the crystals of conine bitartrate from a solution of 135.5 grms. conine and 160 grms. tartaric acid in 450 grms. of water. By treatment of the mother-liquor with caustic potash, separation of the conine as nitro-compound from the resulting mixture of bases (by addition of potassium nitrite to the aqueous solution of their hydrochlorides) and subsequent supersaturation of the remaining solution with caustic alkali, pure methyl conine was obtained. This proved to be identical with *n*-methyl conine obtained synthetically and showing a specific rotation of  $-81.33$  and boiling point of 173–174°C. Pure conine, prepared from the crystallised bitartrate boils at 165.7–165.9°C. (759 mm.), and has a specific rotation of  $-15.6$ . Merck's conine contains about 95 per cent. of pure dextro-conine and 5 per cent. of *n*-methyl conine.—F. M.

*The Technical Preparation of Conine from the Alkaloids accompanying it.* A. Linhorn and R. Willstätter. *Ber.* 1894, 27, 1523.

THE alkaloids are boiled with a solution of sulphuric or hydrochloric acid in methyl alcohol, whereby they are converted into their mother-substance, the methyl ester of econine. This is then converted, by the introduction of a benzoyl group, into cocaine.—T. E.

*Note on the Paper of Einhorn and Willstätter on the Technical Preparation of Cocaine.* C. Liebermann. Ber. 27, 1894, 2051.

THE author denies that the process described by Einhorn and Willstätter (see foregoing abstract) is any improvement on or materially differs from the process described by Giesel and himself. (This Journal, 1889, 131.)—T. E.

*Physiological Action of Compounds of the Cocaine Series.* P. Ehrlich and A. Einhorn. Ber. 1894, 27, 1870—1873.

THE investigations of Filehne, Ehrlich, and Poulsson have shown that neither benzoylcegonine, ecgonine methyl ester, nor ecgonine have any anæsthetic action, whilst these substances are about 20 times less toxic than cocaine.

MacLagan first observed in 1857 that the alkaloid from *Erythroxylon coca* produced a sensation of numbness when placed on the tongue; von Anrep found in 1879 that cocaine has the property of a local anæsthetic, but this property was first made use of in medical practice by Koller (Wiener. med. Woch., 1884, 1276). Knapp (New York Med. Rec., 1884) and Zieminski (Inaug.-Diss., Dorpat, 1884) observed the mydriatic action of cocaine.

Ehrlich (Deutsch. med. Woch. 1890, No. 32) discovered that cocaine produced an extraordinary increase in the volume of the liver and exerted a specific decrease on the cells of that organ; these phenomena are not exhibited by *ecgonine*, *ecgonine ester*, and *benzoylcegonine*. Falek (Inaug. Diss., Kiel, 1886) had already observed that the homologues of cocaine—*esters of benzoylcegonine* containing a higher alkyl group in the place of methyl—have a like physiological action. Derivatives containing other *acidyl* groups in the place of benzoyl, such as *isatropyl*, *cinnamyl*, *phthalyl*, *phenylacetyl*, &c., act in the same manner on the liver as cocaine, but with the exception of the *phenylacetyl* derivative, none of them are anæsthetics, and the anæsthetic property of this is much weaker than in the cocaine. It is worthy of note that both Poulsson and Ehrlich agree that the anæsthetic action of *R-cocaine* is more rapid than that of (ordinary) *L-cocaine*.

The *orthochloro* and *meta-nitro*-derivatives of *L* and *R*-cocaine have only slight anæsthetic action, but exhibit the typical physiological action on the liver; whilst the *metamido* derivatives exhibit neither actions. The *meta-hydroxy* derivatives occupy as far as their physiological properties are concerned, a position intermediate between the *nitro*- and *amido*-cocaines. The *acetyl* and *benzoyl* derivatives of the *meta-amido*-cocaines are not anæsthetics but act on the liver, whilst the cocaine urethanes obtained by the action of ethyl chlorocarbonate on *R*- and *L*-amidococaine are much more powerful anæsthetics than cocaine, exert the characteristic action on the liver, and are highly toxic. Since *metabenzenesulphamido-R-cocaine* and *R-cocaine-urea* have no anæsthetic action, the physiological inactivity of the *amidococaines* cannot be due to the neutralising (basic) effect of the *amido* group.

The colouring matters *R*-cocaine-azodimethylaniline hydrochloride, and *R*-cocaine-azo- $\alpha$ -naphthylamine hydrochloride produce slight anæsthesia but exert no action on the liver.

The *norcocaines* are more powerfully anæsthetic than the *cocaines* themselves and act on the liver, but are far more poisonous.

Cocaine methiodide exhibits neither anæsthetic properties nor action on the liver.

Besides the benzoylated alkaloids examined by Filehne (Berl. Klin. Woch., 1887, 107), benzoylmorphine, benzoylhydrocotarine, benzoylquinine, benzoyleinchonine, &c., it may be mentioned that *ethoxycaffeine*, *eugenolacetamide*, *orthonitrophenyl- $\beta$ -hydroxypropionic ester*, *benzoylquinoyl- $\beta$ -lactic ester* and other substances, an account of which will be given later, are more or less anæsthetics.—A. R. L.

*By-products obtained in the Preparation of Butyltoluene.* A. Baur. Ber. 27, 1894, 1606—1609.

IN the preparation of butyltoluene, the parent hydrocarbon of artificial musk, by the method already described (this Journal, 1891, 1024), the crude reaction product contains the following other hydrocarbons:—

The fraction boiling at 160°—180° C. contains butylbenzene, and, inasmuch as pure toluene was made use of, a conversion of the latter hydrocarbon into benzene must have taken place under the influence of aluminium chloride, as Friedel and Crafts have already observed. It is interesting to note that the author shows in another paper (Ber. 27, 1610) that the *nitro*-derivatives of butylbenzene do not possess the odour of musk.

A conversion of toluene into xylene must also have taken place in the above-mentioned synthesis (*loc. cit.*), for the fraction boiling at 190°—220°, which consists chiefly of butyltoluene, contains also *butylxylene* boiling at 202°.

The fraction boiling at 220°—250° consists of *dibutylbenzene* melting at 76°, and *dibutyltoluene*, an oily hydrocarbon, boiling at 240°—245°. When *dibutylbenzene* is warmed with a mixture of nitric and sulphuric acids, *dinitrobutylbenzene*,  $C_{11}H_{16}(NO_2)_2$ , is obtained; it crystallises from alcohol in white needles, has a faint odour of musk, and melts at 167°—168°. *Dibutyltoluene*, on nitration, yields the trinitro derivative,  $C_{11}H_{14}(NO_2)_3$ , which melts at 152°—153°, and has a faint odour of musk.—A. R. L.

## PATENTS.

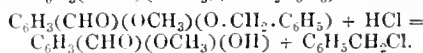
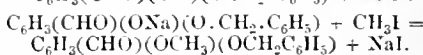
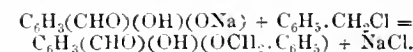
*Improvements in the Manufacture of Aromatic Sulphuryl Derivatives of Protocatechuic Aldehyde and their Conversion into Vanillin.* Sidney Pitt. From the Chem. Fabrik auf Aetien vorm. E. Schering, Berlin. Eng. Pat. 24,177, December 15, 1893.

PROTOCATECHUIC aldehyde is treated with benzene (or toluene) sulphonic chloride and caustic soda or other alkali, under conditions which are more fully described in the patent. Considerable quantities of *p*-benzene sulpho-protocatechuic aldehyde and smaller quantities of the *meta* compound are formed, the reaction being similar to that given below (See patent following). The *para* compound is methylated, and the benzene sulphonic acid group then split off by boiling with potash, vanillin being formed.

—T. E.

*Improvements in the Manufacture of Vanillin.* Sidney Pitt. From the Chem. Fabrik auf Aetien vorm. E. Schering, Berlin. Eng. Pat. 24,178, December 15, 1893.

THE claim is for the manufacture of vanillin from protocatechuic aldehyde,  $C_6H_3(CO\cdot H)(OH)_2$  1.3.4, by transforming a mono-metallic salt of the aldehyde into the *p*-benzyl ether by the action of benzyl chloride, methylating this ether, and then separating the vanillin. The following equations will make the reactions clear:—



—T. E.

*The Preparation of a Crystalline Polymeric Base, suitable for Medical Use, from Amido-Acetic Aldehyde.* S. Pitt. From The Chem. Fabrik auf Aetien vorm. E. Schering, Berlin. Eng. Pat. 24,179, December 15, 1893.

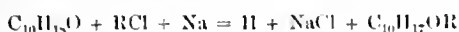
EMIL FISCHER has recently shown that amido acetal is converted into a salt of amido-acetic aldehyde by the action of a strong acid. On standing at the ordinary temperature this salt passes over into a polymeric. The substance is made as follows:—Amido-acetal is mixed with hydrobromic acid (sp. gr. 1.49), and after standing for 5 hours at the ordinary temperature the mixture is evaporated *in vacuo* at



a temperature not exceeding 60° C. The syrup obtained is allowed to stand at 25° C. until it crystallises. The crystals are the hydrobromide of the new base and possess the composition  $C_{10}H_{10}N_2O_2(HBr)_2$ . The free base is very soluble in water, has an alkaline reaction, and is not altered by boiling with alkalis or with Fehling's solution. It is intended for therapeutical use.—T. E.

**Manufacture of Ethers of the Alcohols contained in Natural Essences and of their corresponding Alcohols.** M. Otto and A. Verley, Paris. Eng. Pat. 18,509, September 29, 1894.

CERTAIN natural essences contain isomeric alcohols of the formula  $C_{10}H_{18}O$ , mixed with terpenes, camphenes, &c. To prepare the ethers of these alcohols the patentees treat the essence, in presence of some neutral solvent, with an acid or alcoholic chloride, and sodium. The reaction which occurs is—



where R is any acid or alcohol radical. The ether is separated from other bodies by fractional distillation. The alcohols may be obtained by saponifying the ethers. Several ethers of the above-mentioned alcohols, obtained from the essence of geranium are described; they possess delicate and agreeable odours, and can be used in perfumery.—T. E.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

### PATENTS.

**Method and Apparatus for Timing Photographic Development.** A. Watkins, Hereford. Eng. Pat. 23,894, December 12, 1893.

As a result of numerous experiments the inventor has ascertained that the total length of time of development required to bring a correctly exposed plate to a given density, is generally a definite multiple of the time which elapses between the pouring on of the developer and the first appearance of the image. Also that the multiplying factor is always the same for a particular make of plate, but varies slightly with different makes, and also with different kinds of developers. If, therefore, the time between the pouring on of the developer and the appearance of the image be determined, this time forms a reliable basis to determine the total time required for leaving the plate in the particular developer, as it makes correct allowance for variations in temperature, or in amount of bromide or alkali. Two instruments are described for these timing operations; one is similar to a double-cased hour glass (sand-glass) with two orifices, the lower orifice being capable of alteration in size by means of a regulating screw-plug. Thus, relative variations in the time which the same amount of sand will take in falling through the two orifices may be observed at will. On pouring on the developer the instrument is placed with the sand-chamber uppermost and on the appearance of the image is reversed, the development being finished when the sand has finished running back. The second instrument described is of the nature of a clock.—J. C. C.

**Improvements in producing Photographic Prints.** J. Packham, Croydon. Eng. Pat. 24,963, December 28, 1893.

For the production of warm or pleasantly-tinted photographic prints, platinum prints are treated in a bath of tannin, catechu, &c., either alone or along with deoxidising agents. If after this bath the high lights are degraded, the print is further subjected to the action of a soap- or acid-bath. Several different methods and modifications of this process are described.—J. C. C.

**Improvements relating to the Development of Photographic Pictures.** M. Andresen, Berlin, Germany. Eng. Pat. 25,002, December 29, 1893.

THE inventor claims the following naphthalene derivatives as photographic developers—

1.  $\alpha$ -amido- $\beta$ -naphthol- $\alpha$ -mono-sulphonic acid. 2.  $\alpha$ -amido- $\beta$ -naphthol- $\alpha$ - $\beta$ -disulphonic acid, prepared by the action of sulphurous acid on the nitroso compound of the  $\beta$ -naphthol monosulphonic acid of Schaefer. This acid is obtained as fine white crystalline needles, easily soluble in water, insoluble in alcohol, and sparingly soluble in concentrated hydrochloric acid. The alkaline salts are very soluble in water, and their solution turns brown after exposure for some time to air. The acid solution turns yellow on the addition of ferric chloride. 3.  $\alpha$ -amido- $\beta$ -naphthol- $\alpha$ - $\beta$ -disulphonic acid, prepared by the action of sulphurous acid on the nitroso compound of the  $\beta$ -naphthol- $\beta$ -mono-sulphonic acid. The properties of this body are similar to those described above. 4.  $\alpha$ -amido- $\beta$ -naphthol- $\beta$ -carbo- $\alpha$ -sulphonic acid, prepared by the action of sulphurous acid on the nitroso compound of the  $\beta$ -oxy-naphthol acid having the melting-point 216° C. The properties of this body are, again, similar to those described, with the exception that the acid is sparingly soluble in water.—J. C. C.

## XXII.—EXPLOSIVES, MATCHES, Etc.

### PATENTS.

**An Improved Manufacture of Explosives.** Hans Ritter von Dahmen, Castrop, Westphalia. Eng. Pat. 23,579, December 7, 1893.

ANTHRACENE (eight parts) is melted in a steam-bath and mixed with a powdered mixture of ammonium nitrate (45 parts) and potassium nitrate (44 parts), which salts have previously been melted together at 150° C. About three parts of potassium chlorate are preferably added to the final mixture. Other proportions may be used, larger quantities of ammonium nitrate increases the bursting force. The explosive does not ignite by blows, friction, or heat.—T. E.

**A New Explosive.** H. Boyd, Calcutta. Eng. Pat. 24,425, December 19, 1893.

THE patentee claims the use, in the manufacture of explosives, of oxide of iron which "has passed through the retorts at gasworks, and become impregnated with sulphurous and chemical gases," along with other substances, viz., potassium nitrate, powdered kerosene shale, commercial sulphur, wood dust, and barium nitrate.

A number of valuable properties are attributed to this explosive; among others, that it cannot be exploded by concussion or friction, nor does it explode in the open air. It generates very little smoke or fumes, and is not affected by keeping or by climatic changes. The composition mentioned is oxide of iron, four parts by weight; nitrate of potash, four parts by weight; powdered kerosene shale, three parts by weight; commercial sulphur, two parts by weight; wood dust, two parts by weight; barium nitrate, one part by weight.—T. E.

**Improvements in Explosives.** H. Maxim, New York. Eng. Pat. 18,682, October 2, 1894.

THE object of the invention is to provide a fulminating compound for detonating large masses of high explosives, and which can be thrown from ordnance in considerable quantities with safety. For this purpose nitro-glycerin is thickened with pyroxylin to the consistency of raw rubber. This is done by employing about 75 to 85 per cent. of nitro-glycerin in admixture with 15 to 25 per cent. of pyroxylin, according to the stiffness or elasticity of the compound that may be desired. To facilitate the above



admixture, usually a sufficient quantity of a solvent of the pyroxylin in conjunction with the nitroglycerin, to make the mixture sufficiently pasty to admit of the mixture therewith of a desired quantity of fulminate of mercury. The solvent preferred for the pyroxylin is acetone. With the pasty mass fulminate of mercury is now mixed, to the extent of about 75 to 85 per cent. of the entire compound. The compound can be made less sensitive to shocks by giving it a porous consistency, in agitating it with air while it is still in a syrupy condition. The nitro-glycerin, especially in this latter case, may be omitted. In some cases it is desirable to add a deterring medium, such as nitrobenzene or some suitable gum.—T. E.

*Improvements in the Process of Nitrating Cellulose.*  
Robert Charles Schippaus, Brooklyn, U.S.A. Eng.  
Pat. 18,683, October 2, 1891.

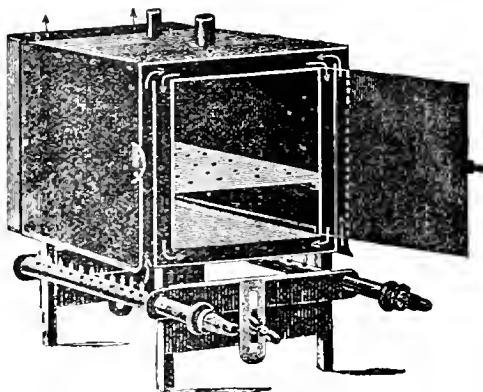
THE improvements consist in adding sulphuric anhydride and nitric acid to the acid bath which has been weakened by acting on the cellulose, and thereby restoring it to its original strength. Water is formed by the action of nitric acid on cellulose, the sulphuric anhydride added converts this water into sulphuric acid; the nitric acid added makes up for that which has combined with the cellulose.—T. E.

## XXIII.—ANALYTICAL CHEMISTRY.

### APPARATUS, ETC.

*An Aluminium Drying-Oven.* L. Meyer. Ber. 1894, 27, 2769—2770.

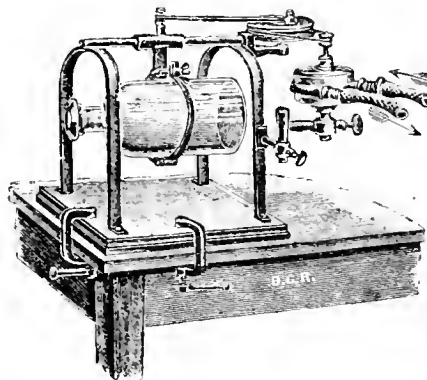
ON account of its high conductivity and comparative permeability in air, aluminium is a very suitable material for the construction of drying-ovens. It possesses over copper the advantage, that the film of oxide formed remains very thin and does not scale off. The author has designed a very convenient form of air-bath, the details of which are



shown in the accompanying drawing. The oven is surrounded throughout (door included) by a double casing through which circulate the products of combustion derived from two perforated tube burners, one at each side of the apparatus, and so arranged that their distance from the bath may be regulated. Two interchangeable sets of burners are provided, the tubes of one set having six, of the other 22 gas outlets each. Using either the small or large burners, according to requirements, it is stated that any temperature between 70 and 330° C., may readily be obtained and kept absolutely constant for any length of time, providing, of course, that a gas-pressure regulator be employed.—H. T. P.

*A New Apparatus for Shaking Bottles.* C. Maull. Ber. 1894, 27, 1732.

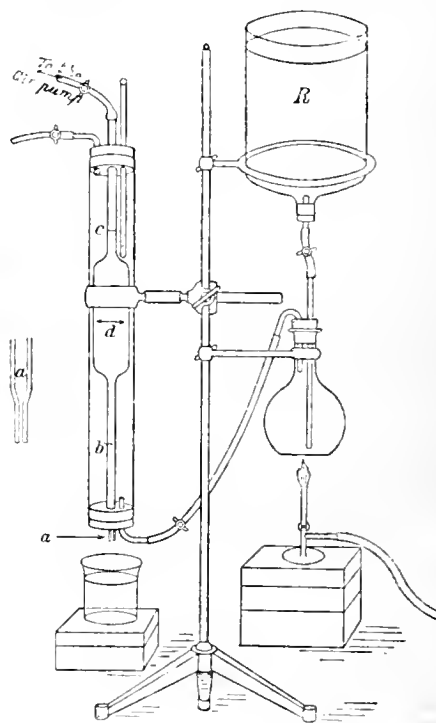
THE apparatus, which is shown in the figure, is said to have the advantage that it can be set in motion by a small



Rabe's turbine, worked by water and consuming 12 litres per minute, and will shake bottles containing as much as 500 cc. Larger instruments are also made.—T. E.

*A Simple Viscosimeter.* M. Wendrin. Zeits. angew. Chem. 1894, 545—547.

THE apparatus shown in the cut consists of a pipette of 50 cc. capacity, the lower end of which is drawn



out to a cylindrical point as shown in *a*: the stem has two marks upon it at *b* and *c*. The pipette is held in position within a cylindrical tube by means of caoutchouc stoppers, and this tube is supported vertically by a clamp. Water is

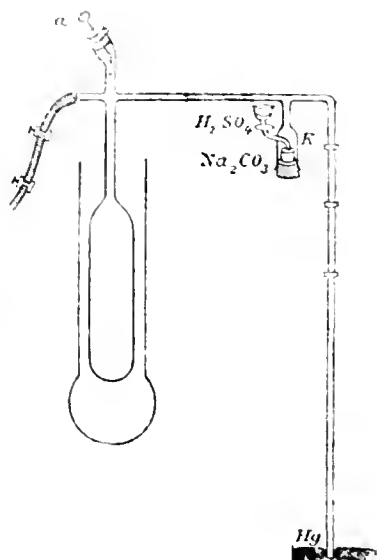
passed through the cylindrical tube from a reservoir R; it may be raised to any desired temperature by the Bunsen's lamp. A thermometer placed through the upper caoutchouc stopper serves to register the temperature of the water passing through the cylindrical tube.

The liquid of which it is required to determine the viscosity is sucked into the pipette somewhat above the mark *c*, and after being adjusted to this mark at a known temperature, the time is noted that elapses while the liquid drops out until it reaches the lower mark *b*. The oily liquid collected in a beaker may be weighed and its specific gravity deduced from this; the estimation of acidity may also be made in the same quantity.—A. R. L.

*Determination of Vapour Density, and a Process for Exhausting without an Air or Water-Air Pump.* C. Schall. J. prakt. Chem. 50, 1894, 87—88.

THE vessel K (Fig. 1), in which the sodium carbonate is decomposed (this Journal, 1892, 370), is now fused on to

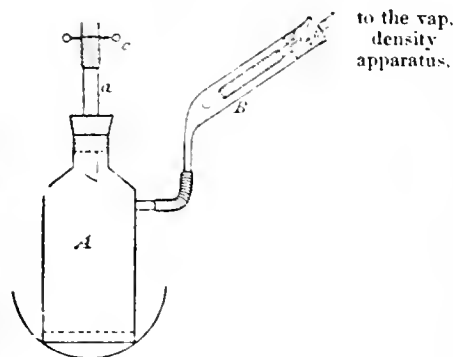
Fig. 1.



the apparatus, the acid being slowly run in—and preferably at not less than 300–400 mm. pressure—through a funnel tube, fitted with a tap, and fused on to the soda vessel. The tube containing the substance under examination is held by a hooked wire *a*, and is released on turning this round by means of the bowed handle.

In the absence of a pump for exhausting, the apparatus in Fig. 2 may be employed. This consists of a tin bottle,

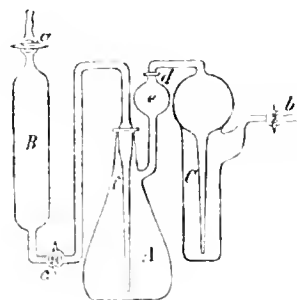
Fig. 2.



A, such as is often used for chemical preparations, which is fitted with a Bunsen valve, B (made of air pump tubing, and slightly increased on the outer edge), and connected with the density apparatus. The air is expelled from A through *c* by boiling water, *c* being closed by sliding a pinchcock from *a* at the moment when the lamp is removed from A. As the vessel cools, which can be accelerated by laying a cloth on A, and afterwards immersing the latter in cold water, the mercury in the density apparatus quickly rises. With a capacity for A of 2½ litres, and of 100–110 cc. for the density apparatus, an attenuation of between 60 and 70 mm. of mercury is soon produced, and this is in most cases sufficient. By repeating the operation (or, less preferably, using a larger vessel) the attenuation can be increased in a manner readily calculable. The vessel A may also be of glass, but in that case the operation of cooling must be carried on more gradually. The soda vessel and the substance tube are weighed together, and, by pouring the soda into the vessel until the required weight is approximated, and then making up exactly by the aid of the rider, the three weighings can be performed almost as quickly as the customary two.—C. S.

*A New Apparatus for the Valuation of Manganese Ores, Estimating Carbon Dioxide, &c.* A. C. Christomanos. Ber. 27, 2748.

THIS is a modification of the well-known Bunsen's apparatus. The carbon dioxide is generated in an Erlenmeyer flask, A, of 55–60 cm. capacity, and about 4½ cm. wide at the bottom, B being the acid vessel, of about 25 cm. capacity, and C the absorption tube, of about 16 mm. diameter, fitted with a bulb of about the same capacity as the



tube, to prevent the sulphuric acid being drawn into the flask A. Sulphuric acid is used for absorption in preference to calcium chloride. B and C are closed by glass Geissler taps, and there is also a tap at *c*, to regulate the supply of acid into A.

The method adopted is as follows:—The substance is first weighed out into the flask A. The vessel B is filled up to the tap *a* with dilute hydrochloric acid, sp. gr. = 1.085, obtained by diluting strong fuming acid with an equal volume of water, and then connected with A, the joint being greased with vaseline. The absorption tube C is connected with A in a similar way. The whole apparatus is then weighed with taps closed, and the weight recorded. The ends of the apparatus, *a* and *b*, are next connected by means of rubber tubing with calcium chloride tubes. First, the tap *b* is opened, then *a*, and lastly *c*, very gradually, so that the acid only drops slowly into A, and thus prevents a too rapid evolution of carbon dioxide. When the acid has all been added and evolution of gas ceases, the taps *a* and *c* are closed, the apparatus held in the fingers at *f*, and heated over a very small flame almost to boiling. The tap *b* is then closed, so that by cooling the lessened pressure will cause any carbon dioxide remaining in the solution to be liberated. As the vessel cools, the taps *c* and *a* are opened at intervals, to prevent accident, and, after 1–1½ hours' cooling, dry air

is passed through for about 20 minutes. The whole apparatus is now weighed with closed taps, and the loss in weight gives the amount of carbon dioxide evolved.

The author recommends this apparatus also for estimating manganese dioxide in manganese ores, pyrolusite, &c. The powdered mineral, together with water and potassium oxalate, is inserted into the flask A, and concentrated sulphuric acid is run slowly from the vessel B into the mixture, as in the Will-Fresenius method.—H. M.

*Ratio of Saccharimeter Degrees on the Ventzke Scale to Angular Degrees for Sodium Light.* E. Rimbaech. Ber. 1894, 27, 2282—2285.

ACCORDING to Schmitz (Ber. 10, 1420), 1° Ventzke (for saccharose) is equal to 0·3455 degree of arc (sodium light); whilst the observation of Tollens (*ibid.* 10, 1411; 17, 1757) indicates the ratio 1:0·3465. With the exception of some measurements by Landolt and Rathgen (Ber. 21, 194), no direct experiments have been made to ascertain this ratio; these chemists give the ratio 1:0·3465 for saccharose. The last-named experiments were made, however, by comparing the indications obtained with two polaristrometers by Laurent and Lippich respectively, with those obtained by a half-shadow saccharimeter. It is therefore not possible to judge the extent to which the results are vitiated by the unavoidable errors in construction peculiar to each instrument. To eliminate as far as possible this uncertainty, the author has repeated the observations, using a polarimeter recently constructed by Schmidt-Haensch. The instrument was at once a polaristrometer and a saccharimeter; as polariser, Lippich's combination was employed, whilst a Glan-Thompson prism, the rotation of which could be read to a minute, served as analyser. Besides this, the instrument was furnished with a quartz compensating arrangement and a Ventzke sugar scale.

The mean ratio S:K (S = Ventzke degrees; K = degrees of arc for sodium light) for the undermentioned compounds as observed by the author, are as follows:—

Saccharose,  $\frac{K}{S} = 0\cdot34394$ , which increases slightly with the concentration.

Glucose,  $\frac{K}{S} = 0\cdot3444$ , which decreases slightly with the concentration; the value is also affected according as an ordinary gas or petroleum lamp, or an Auer's lamp, is employed for the saccharimeter readings.

Santonin was examined in a chloroform solution, and camphor in an alcoholic solution. The ratio  $\frac{K}{S}$  is practically the same for these substances when either an Auer's lamp or sodium light is employed for the saccharimeter, but is somewhat higher when an ordinary gas or petroleum lamp is used.

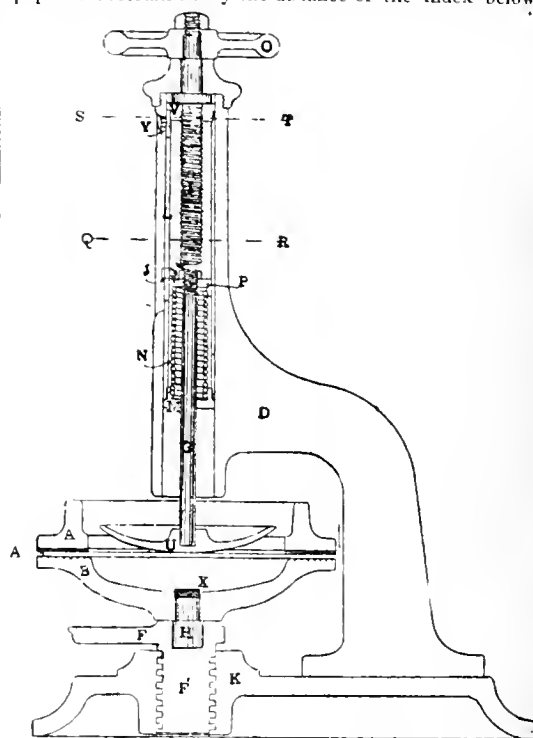
For ordinary purposes it is sufficiently accurate to take the ratio  $\frac{K}{S} = 0\cdot344$  in all cases.—A. R. L.

#### PATENT.

*An Improved Machine or Apparatus for Testing Paper and the like.* G. O. Woolley, Gloucester. Eng. Pat. 13,633, July 16, 1894.

THE machine described is for the purpose of testing the breaking strain of paper or similar material. Referring to the drawing, the paper to be tested is placed between the rings A and B, the surface of the upper one being covered by india-rubber; the lower ring is then raised by the screw F so as to firmly clamp the paper sheet. By use of the hand-wheel at the top of the apparatus the sliding cylinder L is forced down, carrying with it the piston G. By means of the spiral spring N the ram U is thus brought to bear upon the paper until the latter breaks. The index J, which slides in a groove in the cylinder L, carries a scale indicating

the pressure exerted. The amount of depression of the paper is ascertained by the distance of the index below a



zero mark on the standard, and indicates the amount of stretching before rupture.—J. C. C.

#### INORGANIC CHEMISTRY.— QUANTITATIVE.

*The Analysis of American Refined Copper.* H. F. Keller. J. Franklin Inst., July 1894, 54—57.

THE author has found Hampe's original method (Zeits. f. Berg. Hütt. u. Salinenwesen, 21, 218, and 22, 93), which consists in precipitating by electrolysis the greater part of the copper, separating the metallic impurities in the usual manner from the remaining solution, and determining the non-metallic elements, such as oxygen and sulphur, in separate portions of the sample, well adapted for "Lake copper."

For other kinds, and especially for electrolytically refined copper, he prefers Hampe's second method, already abstracted in this Journal, 1894, 421.—J. H. C.

*Attempts to Estimate Sulphur Compounds in the Atmosphere.* H. Oates. Proc. Chem. Soc. 1894 [143], 218.

THE paper contains an account of attempts made to estimate the total amount of sulphur compounds present in the atmosphere of Sheffield.

Various forms of apparatus and different oxidising agents, such as hydrogen peroxide, sodium peroxide, iodine, and potassium permanganate, were tried, but in none of the experiments was it conclusively proved that all the sulphur compounds present in the air were oxidised and retained by the apparatus used.

The results of the determinations tend to show that the amounts of sulphur compounds present in the air, which have been previously published, are considerably too low, and that none of the forms of apparatus used in the experiments described, are capable of yielding correct results.

*Quantitative Electrolytic Analysis.* A. Classen. Ber. 1894, 27, 2060—2081.

STRONG emphasis is laid upon the necessity for including the current-measuring instrument, be it ammeter or voltmeter, in the electrolytic circuit, and upon the worthlessness of many published results, in obtaining which, a voltmeter of unknown resistance has been substituted for the electrolytic cell at the moment of measuring. The method of measurement adopted by the author is to couple in the main circuit, the battery, the electrolytic cell, an ammeter, a variable resistance, and a switch, by which the current may be stopped during the measurement of resistance; this latter is effected by means of a Wheatstone's bridge, rheostat, and separate battery, which can be uncoupled when not in use; the voltage at the electrodes is determined by a voltmeter, which may be switched on or off at will, and is placed in a shunt circuit between the anode and cathode connections of the cell. Using always the same electrode-dimensions and interpolar space, he has made a number of observations, which are detailed in the paper, showing the

temperature, current intensity, interpolar voltage, polarisation, added resistance, and electrolyte resistance at different periods of the work; and from these it is seen that the cyanide solutions have resistances higher than those of double oxalates, and far higher than those of acid-baths. In practice, one ammeter may be made to serve any number of electrolytic cells, and a special switch-board is described, which is so arranged that the current in any given cell may be passed either through a separate ammeter circuit, or through a resistance equal to it, at pleasure; in this way a reading may be taken without any break of current or change of resistance. All the electrolysis cells are arranged parallel, but each has its own rheostat, capable of interposing a resistance of 60 ohms. Four secondary cells, with an aggregate interpolar E.M.F. of about 8.5 volts, are recommended for general use; they are more constant than primary batteries, and are preferable even in small laboratories. A separate circuit with special switch-boards is arranged so that the voltage at the poles of any electrolysis cell may be at once determined, one voltmeter being used for all. Illustrations of the author's laboratory and of the various connections with the battery and instruments are given. A long series of experiments is then described, from which the numbers in the following table have been collated, to show the wide range of experimental conditions under which good quantitative results are obtainable. Italicised figures are those which the author especially recommends:—

Metal.	Bath.	Free Acid, of any.	Good results obtained with				Remarks.
			Ampères per 100 Sq. Cm.	Volts.	Temperature.	Time.	
Cu	Double oxalate .....	Oxalic ...	0.3—1.0	2.5—3.2	° C. 58—60	Hours. 2—2½	Maintain slightly acid.
Cd	" " .....	" ....	0.5 1.0	2.7—3.4	68—76	3—3½	" "
Zn	" " .....	Tartaric .	0.5—1.0	3.5—4.8	55—60	1½—2	Wash before breaking current.
Fe <sup>II</sup>	" " .....	..	0.5—1.5	2.0—4.3	20—65	2½—4	
Fe <sup>III</sup>	" " .....	..	0.5—1.7	2.4—4.2	30—50	3—6½	
Ni	" " .....	..	0.5—1.0	2.7—4.3	15—70	3—5	
Co	" " .....	..	0.5—1.2	2.7—4.1	15—65	2½—6½	Conditions as for Ni.
Hg	" " .....	..	0.2—1.0	2.6—4.7	23—37	1½—5½	
Ag	" cyanide .....	..	0.2—0.5	3.7—4.8	20—30	1½—5	Using pure K <sup>+</sup> Cy.
Sn	" oxalate .....	Acetic ...	0.3—1.0	3.2—4.2	25—30	5½—6½	25 cc. of a 50 per cent. acetic acid.
Sb	" sulphide (Na) ..	..	0.3—1.5	0.8—1.8	21—60	2—17½	Wash before breaking current.
Pb	Nitrate .....	Nitric....	1.4—1.6	2.4—2.5	60—65	1	Anode deposit, weighed as PbO <sub>2</sub> .
Mn	Sulphate.....	Acetic ...	0.3	4.3—4.9	50—68	3	" " limited to Mn <sub>2</sub> O <sub>4</sub> .

In nearly all these tabulated experiments, material was taken for analysis sufficient to give from 0.2 to 0.1 gm. of deposit, and the lowest current intensity in any one series corresponded with the highest time requirement. The zinc is deposited in a platinum dish previously covered with a thin film of copper; the antimony in a platinum dish, the surface of which has been rendered matt by means of a sand blast, as it is found that the deposit on such a surface is more adherent, so that the current intensity may be increased and the duration of the analysis lessened. The methods used for preparing the solutions are sketched in the original paper; and further numbers are given to show that the known separations of metals may be satisfactorily made. In the electrolytic separation of antimony from tin it is now found that by raising the temperature of the solution from normal to 60° C., a stronger current (1.5 ampères) may be safely employed, and the usual period for the deposition of the antimony may thus be reduced from 15 to 2 hours. In separating copper from nickel by the acid oxalate process, the voltage must be maintained within the limits 1.1 and 1.3. In the iron-aluminium separation unduly high current intensity is to be avoided, as the solution thus tends to become heated, and the troublesome

deposit of alumina upon the iron is more likely to be formed.—W. G. M.

*Quantitative Analysis by Electrolysis.* F. Rüdorff. Zeits. angew. Chem. 1894, 388—389.

For the separation of mercury from other elements by electrolysis, the smallest possible current must be used, and more free acid than usual, as the tendency of mercury to dissolve co-precipitated metals, removes them in part from the influence of the liquid. Not more than 0.2 gm. should be used, or the deposited mercury may unite into globules, which will be mechanically carried away by the wash-water.

For the mercury-cadmium separation add from 2 to 5 cc. of nitric acid (sp. gr. = 1.2), and use two cells for eight hours.

For mercury and zinc use the chloride or nitrate solution with 1 cc. of nitric acid, or ten drops of strong sulphuric acid, and two or at most three cells for eight hours; with four cells, zinc also should be separated.

For mercury and iron add four to five drops of strong sulphuric acid, and employ two or three cells.

For mercury and nickel add six drops of sulphuric acid, and use two to three cells over-night; the same process

may be employed for cobalt, substituting 1 to 2 cc. of nitric for the sulphuric acid.

In the case of manganese and mercury, both may be precipitated simultaneously, but the manganese must not exceed 0.03 grm.; three drops of dilute sulphuric acid are used, and two cells, the manganese being collected as hydrated oxide on the platinum spiral anode (this Journal, 1892, 459). In all these cases the solution is diluted to 120 cc., and the battery used is that of Meidinger. In depositing nickel from the sulphate, no trace of nitric acid must be present, or precipitation by even six cells will be incomplete, and the operation must be finished with the aid of a stronger current, as, for example, that from a Gulcher thermopile.—W. G. M.

*The Separation of Metals by Hydrogen Peroxide in Alkaline Solutions.* P. Jannasch and E. Rose. Ber. 1894, 27, 2227—2231. (See this Journal, 1891, 659 and 1037; 1892, 270; and 1894, 67.)

*Separation of Bismuth and Zinc.*—The metals are dissolved in nitric acid (0.3—0.45 grm. in 5 cc. of strong acid), evaporated to dryness, redissolved in 50 cc. of water, and poured with constant stirring into a mixture of 25 cc. of a 3 or 4 per cent. hydrogen peroxide solution with 15 cc. of strong ammonia. The yellowish flocculent precipitate of hydrated bismuth peroxide settles readily and is poured upon a filter, where it is washed, first with dilute ammonia, and then with water; redissolved in dilute nitric acid (1:4), and reprecipitated as described; the dilute acid used for dissolving and washing the precipitate should not exceed 40 cc. and should be measured first. The precipitation must be effected by pouring the metallic salt solution into the alkaline peroxide, as the contrary order would involve the local acidification of the first drops of ammonia, with a consequent loss of oxygen, and it would cause a co-precipitation of a part of the zinc before the ammonia was added in excess, and a portion of this zinc would be permanently retained in the bismuth precipitate, even though the greater proportion ultimately passed into solution. For ordinary analyses a single precipitation will suffice. The bismuth precipitate is detached from the filter paper, and the latter is cut up and heated in a platinum crucible placed within a nickel air-bath covered with asbestos card, until charred; the cover is then removed, and the residue burned to ash; it is next moistened with strong nitric acid and dried. The bulk of the precipitate is now added, and the whole is first completely oxidised in the air-bath, and then ignited until constant in weight. The authors prefer this method to the oxygen ignition previously described by one of them (this Journal, 1894, 67). Since both the hydrogen peroxide and the ammonia generally contain traces of silica, it is safer to examine the bismuth precipitate, or to make a blank test with volumes of these substances equal to those that are used in the process. The filtrate from the bismuth is evaporated to dryness with constant stirring in a deep porcelain (not platinum) dish; the residue, after removal of the stirrer, is heated on an air-bath, and then on asbestos card, until all ammonia salts are expelled; it is then moistened with two drops of nitric acid, dissolved in water, filtered, and precipitated with sodium carbonate. The zinc carbonate is ignited in a porcelain crucible, and the oxide is examined for silica and foreign bases. The results are good.

*Separation of Bismuth and Nickel.*—This separation is conducted like the last; but the nickel may, if wished, be precipitated directly with soda, without previous heating.

*On metal separations by means of ammonium persulphate.*—The substitution of ammonium persulphate for the hydrogen peroxide in these separations, generally involves longer heating, and produces a less agreeable precipitate to work with, and moreover, owing to the formation of sulphuric acid by its decomposition, it is impossible to estimate sulphates in the same solution, whilst it produces interfering reactions in the presence of metals which form difficultly soluble sulphates. The authors are continuing their experiments in this direction.

—W. G. M.

*Separation and Estimation of Tin and of Antimony in Alloys.* Mengin. Comptes rend. 1894, 119, 224.

The author treats the alloy (for example, anti-friction metal) with nitric acid, collects the insoluble oxides of tin and antimony, washes, calcines and weighs them (M). The mixed oxides are next suspended in hydrochloric acid and water, and a ball or plate of pure tin added, whereupon the antimony is reduced to metal, and the tin converted into chloride; the reaction is best accelerated by heat; about 3 hours being then necessary, for 1.5 to 2 grms. of oxides. The precipitated antimony is washed by decantation with water which has been boiled and cooled out of contact with air; then collected and washed, first with water and then with alcohol; dried and weighed (A). There is no appreciable oxidation of the antimony and the method is very exact. The tin is estimated by difference.

$M - A \times 1.262 =$  weight of tin oxide: the latter multiplied by 0.78667, gives the weight of tin in the alloy.

An alternative method for the estimation of the tin is to precipitate the latter by zinc.

The following figures (indicating grms.) of an analysis show the accuracy of the method:—

Samples taken.	Oxides found.	Metals found.
Tin..... 1.162	3.131	Tin..... 1.154
Antimony..... 1.312		Antimony..... 1.309

The plate of tin, introduced to effect the reduction of the oxides, lost 6.918 grms.—E. R. B.

*The Valuation of Artificial Manures.* E. Henfeldt. Zeits. angew. Chem. 1894, 383—386.

This is a review of the principal phosphatic, nitrogenous and potassic manures, and of the methods of analysing them. Thomas slag from different works varies; for whilst the value of the tetra-basic phosphoric acid may be usually taken as half that of "soluble" phosphoric acid, it may in practice range from two-thirds to one-third, Bohemian slags being the less, and those from Rhenish, Westphalia the more valuable. The cause of this variation is not yet known, but high manurial value appears to be correlated with high silica percentage. The citrate solution method gives the most trustworthy indication of the value of Thomas slag, but is somewhat too lengthy for general use when many samples are being treated concurrently. In applying it, the basicity of the slag is first ascertained by shaking 5 grms. of the meal with a 1 per cent. citric acid solution, and determining the degree of neutralisation by acidimetry. 5 grms. of fresh slag are then weighed into a litre flask, and to this are added consecutively, sufficient 5 per cent. citric acid solution to satisfy the basicity, 200 cc. of an acid citrate solution, and enough water to bring the total volume to 500 cc. The whole is shaken for half an hour in a Stutzer apparatus, and is then ready for the molybdate determination. The acid citrate is prepared by dissolving 150 grm. of citric acid in water, neutralising with ammonia, adding 10 grms. of citric acid, and making the whole up to a litre. Among nitrogenous manures, nitrates are best, because the whole of their nitrogen is directly assimilable by the plant, whilst ammoniac nitrogen loses 10 per cent. during its conversion into nitric nitrogen, and organic nitrogen proportionately more during its successive conversion into ammonia and nitrate. In applying Kühn's modification of the zinc-iron method, the iron must be free from oxide, so that metal reduced by hydrogen should be avoided. As a means of applying potash and phosphoric acid conjointly to the soil, especially when mixed with ammonium nitrate, potassium phosphate should command a large market if it could be produced at a reasonable price.—W. G. M.

*Estimation of Phosphoric Acid by the Citrate Method.*  
F. Glaser. *Zeits. angew. Chem.* 1894, 543—545.

It is well known that when solutions of phosphates containing neither lime, iron, alumina, nor manganese are analysed by the citrate method variable results are obtained according to the quantity of magnesia mixture employed. Grupe and Tollens (*J. f. Landw.*, 30, 23) have already observed that a portion of the phosphoric acid remains in solution; on addition of a large excess of magnesia mixture, however, more magnesia is precipitated than corresponds with the phosphoric acid contained in the precipitate, and a compensation of errors is the result. But in the case of solutions of phosphates containing lime it is found that the analytical results are not affected to so great an extent by increasing the amount of magnesia mixture, consequently differences must in these cases be ascribed to the retention of phosphoric acid in solution. Neubauer has shown (*Zeits. anorg. Chem.* 2, 45; 4, 251) that when phosphoric acid is precipitated from an ammonium solution (such as a solution of the molybdic precipitate) by magnesia mixture and the precipitate is ignited, a loss of phosphoric acid by volatilisation occurs. This he believes to be explained by the formation of the salt  $\text{Mg}(\text{NH}_4)(\text{PO}_3)_2$ , which is first converted into  $\text{Mg}(\text{PO}_3)_2$ , and subsequently into  $\text{P}_2\text{O}_5$  and  $\text{Mg}_2\text{P}_2\text{O}_7$ .

The present author's experiments show that also with the citrate method a portion of the phosphoric acid is volatilised under certain circumstances. This was actually proved by igniting the precipitate in a crucible, the lid of which was covered with magnesia to absorb the  $\text{P}_2\text{O}_5$ ; the volatile phosphoric acid was then estimated by the molybdenum method. When a solution of alkali phosphate is mixed with 100 cc. of ammonium citrate solution and precipitated with only a slight excess of magnesia mixture, a portion of the phosphoric acid is volatilised on igniting the precipitate; this loss, is, however, quite annulled if a certain excess of magnesia mixture is made use of and the amount of magnesium pyrophosphate then obtained corresponds with that obtained by the molybdenum method. The author gives the following theoretical explanation:—Ammonium citrate enters into double decomposition with magnesium chloride, so that when but a slight excess of magnesia mixture is used there will be present, at a given period of the operation, relatively little magnesium chloride and much ammonium salt, so that the salt,  $\text{Mg}(\text{NH}_4)(\text{PO}_3)_2$ , will be formed as stated by Neubauer. The author worked with quantities of disodium and dipotassium phosphate corresponding with about 0.3—0.4 gram. of magnesium pyrophosphate. To ensure accuracy, it appears to be essential to employ about 40 cc. of magnesia mixture, and to stir well during the precipitation; the test should then be placed aside for several hours and stirred at intervals.

The results have no practical bearing on the estimation of phosphoric acid in manures, for the presence of lime and iron salts in these (using 25 cc. of magnesia mixture) reduces the volatility of phosphoric acid (even when large quantities are present) to such an extent that it need not be taken into account.—A. R. L.

*The Testing of Zinc Ores and Products of Manufacture.*  
F. Meyer. *Zeits. angew. Chem.* 1894, 391—395.

THE methods used in zinc-works are described with the hope of eliciting discussion, which may lead to the selection of an uniform standard process.

**Determination of Zinc.**—0.5 gram. of the finely-powdered ore (raw or roasted), zinc-dust or zinc-white, or 1 gram. of retort residue, is treated with 10 cc. of cold aqua regia (1  $\text{HNO}_3$ :2  $\text{HCl}$ ) in a small flask, which is then warmed. After evaporating off excess of acid, the residue is dissolved in 10 cc. of sulphuric acid (1 of 66° B + 211.0), which is next evaporated until fumes of sulphuric anhydride are given off. On cooling, 60 to 80 cc. of water and 10 cc. of sodium thio-sulphate solution (1:8) are added, and the mixture is boiled until the liquid portion has become clear, when it is filtered from the precipitated copper and cadmium sulphides

lead sulphate, and sulphur. It is now mixed with 6 cc. of nitric acid, and boiled until precipitated sulphur (from the thio-sulphate) has collected into globules, leaving the solution clear. The addition of 30 cc. of ammonia (22° B) precipitates iron and alumina, and a further addition of 20 cc. of bromine water when almost cool, brings down the manganese also. After standing, the liquid is boiled, to remove excess of bromine, and filtered; the mixed precipitate is redissolved, without thorough washing, in 5 cc. of aqua regia, and reprecipitated. The two filtrates, being united, are made up to 500 cc., and allowed to stand overnight. On the following day this liquid is titrated with sodium sulphide solution (1 cc. circa 0.01 gram. Zn), which must be re-standardised each time, using a quantity of zinc approximately equal to that in the test, dissolving it in 10 cc. of aqua regia, adding 20 cc. of ammonia, diluting to 500 cc., and allowing it to stand overnight. In titrating, the sulphide is run in with constant stirring, the thin glass tube used as a stirrer serving to withdraw a drop from time to time, for test with a strip of lead-paper. The formation of a just perceptible dark ring on the paper at the edge of the drop marks the end of the reaction. Tests are made in duplicate, and give closely-approximate results in different hands; out of 167 determinations worked simultaneously by two chemists, only five showed a variation exceeding 1 per cent.

**Sulphur Determination.**—0.5 gram. of the finely powdered raw blende, or 2 grams. of roasted ore or retort residue, are treated with 10 cc. of fuming nitric acid in a small flask; with rich blendes, the addition of a few potassium chlorate crystals prevents separation of sulphur. When all action in the cold has ceased, the liquid is evaporated to dryness twice with hydrochloric acid, to expel all nitric acid, and is then diluted to 80 or 100 cc. The iron is then precipitated by ammonia, and filtered off. After boiling to expel ammonia, 5 cc. of hydrochloric acid is added, and the sulphuric acid is precipitated boiling by barium chloride. The roasting-furnace manager judges of the completion of his work by the degree of discolouration produced by the reaction of 10 cc. hydrochloric acid with a measured quantity of the ore upon a neutral or weakly alkaline lead acetate paper, suspended in the neck of a flask tightly closed by the thumb. A long table is given for the conversion of grms.  $\text{PbSO}_4$  into percentages of sulphur.

In dealing with metallic zinc, 5 grams. of borings are dissolved in dilute sulphuric acid (1:4), filtered from the lead residue into a 500 cc. flask, from which, after filling to the mark, 50 cc. are withdrawn for each determination. The separation by means of thio-sulphate and the zinc titration are carried out as above described.—W. G. M.

*Quantitative Estimation of Nickel by Means of Ammoniacal Mercuric Cyanide.* F. W. Schmidt. *Ber.* 1894, 27, 1624—1627.

BEARING in mind the known fact that nickel may be precipitated as sulphide by means of normal (colourless) ammonium sulphide, and that the precipitation is assisted by the presence of ammonium salts, the author has devised the following method:—The nickel solution (10 cc. containing about 1 per cent. of metal) is mixed with an excess of ammonium nitrate and sufficient ammonia added until the liquid has a faint bluish colour; too great excess of ammonia must, however, be avoided as it leads to the formation of poly- (yellow) sulphide of ammonium during the treatment which follows. An excess of sulphuretted hydrogen water is now added to the boiling solution whereby, after continuing the boiling for a short period the whole of the nickel is thrown down as sulphide, and in a form which can easily be filtered from the colourless supernatant liquid; it is then washed with a hot solution containing sulphuretted hydrogen, and ammonium nitrate. The nickel sulphide so obtained resists the oxidising influence of the air to about the same extent as freshly-precipitated cupric sulphide. After washing the nickel sulphide it is converted into nickelous oxide by evaporating with mercuric cyanide and subsequent ignition

in the manner previously described (this Journal, 1894, 548) and weighed as such. The test analyses show that the method yields perfectly satisfactory results.

In considering the action of ammonium nitrate in promoting the precipitation of metallic sulphides, the author mentions that from a solution of zinc sulphide in ammonium chloride, the sulphide may be precipitated completely by adding calcium chloride, magnesium sulphate, and certain other neutral salts. In the same manner from the solution of oxythioarsenious acid, obtained by mixing solutions of sulphuretted hydrogen and arsenic trioxide, the whole of the arsenic may be precipitated as trisulphide on addition of sodium chloride, potassium iodide, ammonium carbonate, magnesium sulphate, &c., whilst such a solution undergoes decomposition on addition of hydrochloric acid. Also the hitherto unknown orange-red solution of thio- or oxythioantimonious acid, which the author obtained by mixing a very dilute solution of tartar emetic with sulphuretted hydrogen, is precipitated by addition of certain neutral salts thereto. All these exhibit, therefore, the behaviour of colloidal solutions, from which the fact that certain neutral salts favour the precipitation of the corresponding sulphides derives a simple explanation.

The author's previously expressed opinion that ammoniacal mercuric cyanide would effect the decomposition of many sulphides is therefore confirmed. Not only is powdered copper pyrites rendered black when evaporated with ammoniacal mercuric cyanide, but the residue, after ignition, is completely soluble in hot hydrochloric acid. This operation may be carried out in a short time, whereas the oxidation of copper pyrites with fuming nitric acid or potassium chlorate and hydrochloric acid takes several days.

The author mentions in a footnote that a good method of decomposing copper pyrites is to carefully roast the finely-pulverised mineral in a porcelain crucible; the residual copper and ferric oxides are then readily soluble in warm concentrated hydrochloric acid, and both metals may be estimated in the solution.—A. R. L.

#### ORGANIC CHEMISTRY.—QUALITATIVE.

*Maltol.* J. Brand. Ber. 27, 806.

See under XVII., page 1213.

*Picein: A Glucoside obtained from the Foliage of the Pine (Pinus Picea).* Tanret. Comptes rend. 119, 1894, 80.

See under XX., page 1217.

*The Influence of the Acid Molybdates of Soda and Ammonia on the Rotatory Power of Rhamnose (Isodulcitol).* D. Gernez. Comptes rend. 119, 1894, 63—65.

The following conclusions are drawn:—

1. A small addition of a molybdate to a solution of Rhamnose, determines a relatively large increase in the rotatory power; but further equal increments in the proportion of molybdate produce a less and less marked effect, until at length the rotatory power ceases to grow.

2. The maximum increase is obtained when the amount of added molybdate equals  $\frac{6.75}{24}$  of the molecular weight.

The rotatory power then becomes,  $(\alpha)_D^{18} = +22^\circ.95$  and  $+19^\circ.91$  in the case of acid sodium and acid ammonium molybdate respectively. (Rhamnose dissolved in water alone,  $(\alpha)_D = +9^\circ.7$ .)

3. The relative weight of molybdate required to produce the maximum effect, is identical with that which exerts a similar influence in the case of mannitol, sorbitol, and persistol.—H. T. P.

#### ORGANIC CHEMISTRY.—QUANTITATIVE.

*The Determination of the Nitrogen in Nitrates, Nitro- and Nitroso-Compounds in the Wet Way.* M. Krüger. Ber. 1894, 27, 1633.

KJELDAHL's original method cannot be applied to the determination of nitrogen in substances containing the nitrogen linked to oxygen. The author reduces such substances by means of a solution of stannous chloride in hydrochloric acid (containing 150 grms. of tin to the litre) and metallic tin. The latter should be made by precipitating a solution of stannous chloride with zinc. After reduction, the substance is treated with strong sulphuric acid and potassium bichromate, in the way previously described by the author (this Journal, 1894, 425).

The numbers obtained in this way are said to be very satisfactory.—T. E.

*Examination of Beeswax.* E. J. Parry. Chem. and Druggist, 1894, 692.

A CAREFUL application of Hüll's test of titrating with standard alkali, and then saponifying with alkali, will yield excellent results. If this test, combined with those of specific gravity and melting point (which should closely approximate  $63^\circ \text{C.}$ ) be applied, the deduction drawn will be such as to exclude adulteration with nearly all other substances. The figures for specific gravity of pure white wax are, with Parry and Estcourt, 0.962 and 0.963 respectively, whilst Schmidt obtained the number 0.961.

*Remarks on the Alcohol Extraction of Beetroot.* Wiskirchen. Zeits. Zuckerind. 1894, 44, 700.

TOLLENS and Hermann recommend that basic lead acetate shall be added to the beetroot sludge, instead of to the filtered extract. The author remarks that this is advantageous to the extraction as to the digestion; it not only hastens the operation considerably, but also lessens the dilation of the cell membrane.—A. R. L.

*The Gravimetric Method of Estimating Cupric Reducing Sugar with Fehling's Solution.* C. Killing. Zeits. angew. Chem. 1894, 431—433.

Of the numerous methods that have been devised for the gravimetric estimation of sugars the Märcker-Alilhn is the most generally adopted; it consists in collecting the cuprous oxide in a Soxhlet's asbestos filter, reducing it in a current of hydrogen, and weighing as metallic copper. The method leaves nothing to be desired in point of accuracy when the tubes are packed with asbestos, which will withstand the action of hot potash and nitric acid. At the present time, however, long-fibered asbestos is no longer obtainable on the market, and since this is so, the method cannot give such good results. Märcker (*Handbuch Spiritusfabrikation* 4 Aufl., 85) has already observed that certain kinds of asbestos are not adapted for this use. The author describes the following experiments made with the object of verifying this observation:—

A tube was packed with glass-wool and asbestos, which had been treated with soda and acid, and had already been used six times for the estimation of sugar; it was dried and weighed. Through it was filtered 60 cc. of boiling Fehling's solution diluted with an equal volume of boiling water, as in Alilhn's method. It was then successively washed with water, alcohol, and ether, dried and re-weighed, when it was found to have lost 0.0053 grm.; on treatment with nitric acid it sustained a further loss of 0.0032 grm.; when again treated with Fehling's solution it lost 0.0029 grm.; and under the influence of 2 cc. of nitric acid it lost 0.0076 grm. A tube was packed with new specially-selected long-fibre asbestos; it was treated successively with 20 per cent. soda, water, and nitric acid; on being subsequently washed with water, alcohol, and ether it was found to have lost 0.0086 grm. It was now



treated with 60 cc. of hot Fehling's solution and 60 cc. of water, when the loss amounted to 0.0020 gram. Treated with nitric acid it lost 0.0032 gram, and treated subsequently with Fehling's solution it lost 0.0011 gram; a further loss of 0.0031 gram. was sustained when it was again treated with nitric acid. Glass-wool, which is employed for packing the lower part of the filtering tubes (beneath the asbestos), also frequently sustains loss under similar treatment to that above described; thus:—0.8 gram. of glass-wool lost 0.0045 gram. after being used for the filtration of Fehling's solution; 0.0030 gram. on subsequent treatment with 2 cc. of nitric acid; and a further loss of 0.0062 gram. occurred when it was again used for filtering Fehling's solution.

The author then quotes the recent experiments of Nihoul (Chem. Zeit. 1893, 500; this Journal, 1894, 845) and of Grünhut (this Journal, 1894, 815); he then describes the following method, which is, he states, easily carried out and gives good results:—

The cuprous oxide is collected and washed on a paper filter, and the whole dried at 100°; the dry oxide is then detached as completely as possible from the filter, transferred to a tared crucible, weighed, and calculated to metallic copper by means of the factor 0.888. The filter-paper is then incinerated in a separate crucible; the residue, after deducting the ash of the paper and the copper retained by it (which is arrived at by a special experiment), is weighed as cupric oxide, and the weight, after being calculated to metallic copper by the factor 0.7986, added to that of the main quantity. The author ignores the presence of the organic substance, which is said to be retained by the cuprous oxide (see Nihoul, *loc. cit.*), as the quantity is too small to be taken into account; in one experiment, however, 0.4641 gram.  $\text{Cu}_2\text{O}$  gave, on reduction with hydrogen in a Rose's crucible, 0.4117 gram. Cu instead of 0.4120 gram.—A. R. L.

**Gravimetric Estimation of Cupric Reducing Sugars by Means of Fehling's Solution.** A. Prager. Zeits. angew. Chem. 1894, 520—521.

KILLING (Zeits. angew. Chem. 1894, 431; this Journal, 1894, 1226) has drawn attention to the inaccuracy of the Märcker-Allihn method of estimating cupric reducing sugars; Nihoul (Chem. Zeit. 17, 500, 18, 881; also this Journal, 1894, 845) and Grünhut (Chem. Zeit. 18, 447; this Journal, 1894, 815), in the course of a controversy on the respective merits and demerits of the asbestos filtration method and the paper filtration method, have pointed out several sources of error in both. The author states that the reason that low results are obtained, even after repeatedly evaporating the precipitated cuprous oxide with nitric acid previously to igniting it, is to be traced to the formation of small lumps, which occlude reduced copper and protect it from oxidation. The formation of these lumps is avoided by the modifications to be described, and the method then gives exact results:—The precipitated cuprous oxide is collected on a double filter. The filter and precipitate are then dried, which can be accomplished in 15 minutes; the precipitate is then detached and placed on a piece of glazed paper, the filter being meanwhile incinerated in a platinum crucible, and the ash reduced to powder by means of a piece of platinum wire. When cool, the cuprous oxide is transferred to the crucible, which is then ignited over a minute flame and the cuprous oxide stirred until it is converted into a fine powder. The crucible and its contents are now ignited for some minutes in a larger flame and then weighed. No increase in weight is obtained by treatment with nitric acid and subsequent ignition. If ash-free filter papers of equal size are employed, it is only necessary to make one blank experiment with pure Fehling's solution in order to arrive at the amount of copper which is retained by the filter. The results are generally in agreement with those obtained by the asbestos filtration method.—A. R. L.

**A New Gravimetric Method of Estimating Glucose.** F. Gaud. Comptes rend 119, 1894, 478—479.

The author states that Soxhlet's method yields too high results because small quantities of cuprous oxide escape reduction with hydrogen.

The method proposed by the author is as follows:—

Measure 50 cc. of freshly prepared Fehling's solution and 50 cc. of water into a porcelain dish; boil for two or three minutes, place the dish on a boiling water-bath, and add 25 cc. of the saccharine liquid to be estimated, which should be previously diluted to a strength of about 1 in 100. At the end of 10 minutes, reduction being complete, the supernatant liquid is decanted and the precipitate washed with boiled distilled water, care being taken to retain the precipitate. When the washings are no longer alkaline to phenolphthalein the precipitate is washed into a flask of 20 to 25 cc. capacity, of which the volume at 0 °C. is known. The volume is made up with boiled distilled water. Then, if  $P$  be the weight of the liquid, plus the precipitate contained in the flask at temperature  $t$ , the volume—

$$V_t = V_0[1 + 3\beta(t - t_0)]$$

Also, the density  $\Delta$  of dry cuprous oxide being 5.881, and  $d$  the density of water at  $t$  (deduced from tables), the weight of the precipitate  $p$  is obtained by the formula—

$$p = \frac{P - V_t d}{1 - \Delta}$$

The weight of cuprous oxide obtained does not vary proportionally with the weight of the sugar, and the author gives the following tables:—

$\text{Cu}_2\text{O}$ .	Glucose.	$\text{Cu}_2\text{O}$ .	Glucose.
Mgrms.	Mgrms.	Mgrms.	Mgrms.
10	5.413	100	46.221
20	9.761	200	91.047
30	14.197	300	135.842
50	23.036	400	180.928

The error of experiment is claimed to be only 1 in 1,000 when  $P$  is carefully corrected for temperature and pressure.

—T. A. G.

**The Estimation of the Sugar-Content of Tanning Materials, Tanning Extracts, Tanning Liquors, and Weighted Leather.** V. Schroeder, A. Bartel, and W. Schmitz-Dumont. Dingl. Polyt. J. 1894, 293, 234—237.

SEE this Journal, 1894, 1101, where it was stated that the authors have shown that for the estimation of sugar in tanning materials a longer heating than that recommended by Allihn is necessary, on account of the presence of unknown and slowly reducing sugars, 30 minutes having been found to give the best results.

The following table has been constructed showing the mgrms. of glucose corresponding to mgrms. of copper weighed, when the Fehling's solution is heated with the sugar solution for 30 minutes:—

TABLE for the DETERMINATION of the GLUCOSE in TANNING MATERIALS through the COPPER WEIGHED, after heating FEHLING'S SOLUTION with the GLUCOSE SOLUTION for HALF-AN-HOUR.—R. Koch and R. Ruhsam.

Cu	Glucose.	Cu	Glucose.	Cu	Glucose.
Mgrms.	Mgrms.	Mgrms.	Mgrms.	Mgrms.	Mgrms.
1	0.4	5	2.0	9	3.7
2	0.8	6	2.5	10	4.1
3	1.2	7	2.9	11	4.5
4	1.6	8	3.3	12	4.9



TABLE for the DETERMINATION of the GLUCOSE, &c.—*cont.*

Cu		Glucose.		Cu		Glucose.	
Mgms.	Mgms.	Mgms.	Mgms.	Mgms.	Mgms.	Mgms.	Mgms.
13	573	61	2679	109	5176		
14	577	62	2774	110	5211		
15	611	63	2870	111	5246		
16	615	64	2875	112	5311		
17	770	65	2970	113	5346		
18	774	66	2975	114	5411		
19	778	67	3070	115	5446		
20	822	68	3075	116	5511		
21	826	69	3170	117	5546		
22	970	70	3176	118	5611		
23	974	71	3211	119	5646		
24	979	72	3246	120	5711		
25	1073	73	3311	121	5746		
26	1077	74	3346	122	5811		
27	1111	75	3411	123	5846		
28	1116	76	3446	124	5911		
29	1270	77	3511	125	5946		
30	1274	78	3546	126	6011		
31	1279	79	3611	127	6046		
32	1373	80	3646	128	6111		
33	1377	81	3711	129	6146		
34	1411	82	3746	130	6211		
35	1416	83	3811	131	6246		
36	1570	84	3846	132	6311		
37	1574	85	3911	133	6346		
38	1579	86	3946	134	6411		
39	1673	87	4011	135	6446		
40	1677	88	4046	136	6511		
41	1771	89	4111	137	6546		
42	1776	90	4146	138	6611		
43	1870	91	4211	139	6646		
44	1874	92	4246	140	6711		
45	1879	93	4311	141	6746		
46	1973	94	4346	142	6811		
47	1977	95	4411	143	6846		
48	2071	96	4446	144	6911		
49	2076	97	4511	145	6946		
50	2170	98	4546	146	7011		
51	2174	99	4611	147	7046		
52	2278	100	4646	148	7111		
53	2279	101	4711	149	7146		
54	2373	102	4811	150	7211		
55	2377	103	4846	151	7246		
56	2471	104	4911	152	7311		
57	2476	105	4946	153	7346		
58	2570	106	5011	154	7411		
59	2574	107	5046	155	7446		
60	2678	108	5111	156	7511		

TABLE for the DETERMINATION of the GLUCOSE, &c.—*cont.*

Cu		Glucose.		Cu		Glucose.	
Mgms.	Mgms.	Mgms.	Mgms.	Mgms.	Mgms.	Mgms.	Mgms.
157	7575	205	9973	233	12172		
158	7670	206	9978	254	12178		
159	7675	207	10373	255	12573		
160	7770	208	10078	256	12578		
161	7775	209	10174	257	12673		
162	7870	210	10179	258	12679		
163	7875	211	10274	259	12775		
164	7970	212	10279	260	12870		
165	7975	213	10375	261	12875		
166	8070	214	10470	262	12970		
167	8075	215	10475	263	12975		
168	8170	216	10570	264	13071		
169	8174	217	10575	265	13076		
170	8179	218	10670	266	13171		
171	8274	219	10676	267	13176		
172	8279	220	10771	268	13272		
173	8374	221	10776	269	13277		
174	8379	222	10871	270	13372		
175	8474	223	10877	271	13377		
176	8479	224	10972	272	13472		
177	8574	225	10977	273	13477		
178	8579	226	11072	274	13573		
179	8674	227	11077	275	13578		
180	8679	228	11172	276	13673		
181	8774	229	11178	277	13678		
182	8779	230	11273	278	13774		
183	8874	231	11278	279	13779		
184	8879	232	11373	280	13874		
185	8974	233	11378	281	13970		
186	8979	234	11474	282	13975		
187	9074	235	11479	283	14070		
188	9079	236	11574	284	14075		
189	9173	237	11579	285	14171		
190	9178	238	11674	286	14176		
191	9273	239	11770	287	14271		
192	9278	240	11775	288	14276		
193	9373	241	11870	289	14372		
194	9378	242	11875	290	14377		
195	9473	243	11970	291	14472		
196	9478	244	11975	292	14477		
197	9573	245	12071	293	14573		
198	9578	246	12076	294	14578		
199	9673	247	12171	295	14673		
200	9678	248	12176	296	14679		
201	9773	249	12271	297	14774		
202	9778	250	12277	298	14779		
203	9873	251	12372	299	14874		
204	9878	252	12377	300	14970		

TABLE for the DETERMINATION of the GLUCOSE, &c.—*cont.*

Cu	Glucose.	Cu	Glucose.	Cu	Glucose.
Grms.	Grms.	Grms.	Grms.	Grms.	Grms.
301	149.5	349	175.6	397	203.3
302	150.1	350	176.2	398	203.8
303	150.6	351	176.8	399	204.4
304	151.1	352	177.3	400	205.0
305	151.7	353	177.9	401	205.6
306	152.2	354	178.5	402	206.2
307	152.8	355	179.1	403	206.8
308	153.3	356	179.6	404	207.3
309	153.9	357	180.2	405	207.9
310	154.4	358	180.8	406	208.5
311	155.0	359	181.4	407	209.1
312	155.5	360	181.9	408	209.7
313	156.0	361	182.5	409	210.3
314	156.5	362	183.1	410	210.8
315	157.1	363	183.7	411	211.4
316	157.6	364	184.2	412	212.0
317	158.1	365	184.8	413	212.6
318	158.7	366	185.4	414	213.2
319	159.2	367	186.0	415	213.8
320	159.8	368	186.5	416	214.4
321	160.3	369	187.1	417	214.9
322	160.9	370	187.7	418	215.5
323	161.4	371	188.3	419	216.1
324	162.0	372	188.8	420	216.7
325	162.5	373	189.4	421	217.3
326	163.0	374	190.0	422	217.9
327	163.6	375	190.6	423	218.4
328	164.1	376	191.1	424	219.0
329	164.7	377	191.7	425	219.6
330	165.2	378	192.3	426	220.2
331	165.8	379	192.8	427	220.8
332	166.3	380	193.4	428	221.4
333	166.9	381	194.0	429	221.9
334	167.4	382	194.6	430	222.5
335	167.9	383	195.2	431	223.1
336	168.4	384	195.7	432	223.7
337	169.0	385	196.3	433	224.3
338	169.5	386	196.9	434	225.1
339	170.1	387	197.5	435	225.8
340	170.6	388	198.0	436	226.4
341	171.2	389	198.6	437	227.1
342	171.7	390	199.2	438	227.8
343	172.2	391	199.8	439	228.5
344	172.8	392	200.3	440	229.1
345	173.3	393	200.9	441	229.8
346	173.9	394	201.5	442	230.5
347	174.5	395	202.1	443	231.2
348	175.0	396	202.7	444	231.8

TABLE for the DETERMINATION of the GLUCOSE, &c.—*cont.*

Cu	Glucose.	Cu	Glucose.	Cu	Glucose.
Grms.	Grms.	Grms.	Grms.	Grms.	Grms.
445	232.5	456	239.9	467	247.4
446	233.2	457	240.6	468	248.0
447	233.9	458	241.3	469	248.7
448	234.5	459	242.0	470	249.4
449	235.2	460	242.6	471	250.1
450	235.9	461	243.3	472	250.8
451	236.6	462	244.0	473	251.4
452	237.2	463	244.7	474	252.1
453	237.9	464	245.3	475	252.8
454	238.6	465	246.0	476	253.5
455	239.3	466	246.7		

*Determination of Glucose by Means of Alkaline Copper Solutions.* F. Gaud. *Comptes rend.* 1894, **119**, 650—652.

It is well known that when certain sugars are boiled with Fehling's solution various compounds are produced. The author has already shown (*Comptes rend.* **119**, 604) that in the case of glucose, tartaric acid, formic acid, and oxalic acid are formed by partial oxidation; whilst glucinic acid, catechol, gluconic acid, lactic acid and glyceric acid are among the products of the action of the alkali on this sugar. Thus a portion of the sugar escapes the oxidising action of the alkaline copper solution and an error is thereby introduced. In order that this error may have a constant value it is necessary to standardise the copper solution and perform the titrations under as nearly as possible the same conditions. To this end a preliminary titration is made and from its indications the sugar solution is adjusted to a concentration of 0.5–1 per cent. The author also finds that undiluted solutions, *i.e.*, those having a concentration up to 10 per cent., may be titrated direct if the following correction be applied which has been deduced from 400 experiments:— $y = -0.00004801x + 0.02876359x^2$  in which  $y$  denotes the error as a function of the exact titre  $x$ . To calculate analyses the experimented titre  $\theta$  must be introduced and the formula becomes  $0.02876x^2 - 1.00004801x + \theta = 0$  whence the value of  $x$  may be calculated. As an alternative it is suggested to employ ammonia instead of potash and soda in preparing alkaline copper solutions for sugar titration (compare, however, Allen and Gaud, next abstract).—A. R. L.

*New Method of Estimating Cupric Reducing Power.* Allen and Gaud. *J. Pharm. Chim.* 1894, [5], **30**, 305—307.

When glucose is heated at 100° with ammonia in a sealed tube for 50–40 hours only a small quantity is decomposed, corresponding with the production of 5–6 per cent. of formic acid; whereas when it is heated in the same manner with the fixed alkalis—soda and potash—the amount of decomposition corresponds with as much as 60 per cent. of lactic acid. These observations led the author to substitute ammonia for the fixed alkalis in preparing a solution for the volumetric estimation of sugars by the cupric reduction method.

The solution employed by the author is prepared as follows:—Pure electrolytic copper (8.7916 grms.) is dissolved in concentrated sulphuric acid (93 grms.) diluted with an equal volume of water; the solution is then made up to 1 litre with concentrated ammonia. 10 cc. of this solution is equivalent to 0.05 grms. of glucose, and since it contains no organic matter it keeps well.

In carrying out the titration, 10 cc. of the copper solution are measured into a flask, and 10 cc. of ammonia solution added thereto. The flask is closed by a cork having three

borings, the end of the burette being inserted into one whilst the two others permit of arrangements for passing a current of hydrogen through the liquid during the reaction. The flask is placed on a water bath heated at 90–95°, the temperature of the solution contained therein being maintained at 80–85°, during the addition of the sugar solution, which is run in drop by drop until the blue colour is destroyed; the cuprous oxide formed by the reducing action of the sugar is of course dissolved by the ammonia. It is important that the temperature of the solution should not rise above 80–85° or loss of ammonia will occur, which has the disadvantage of considerably slackening the reaction.

—A. R. L.

*Behaviour of Normal and Basic Lead Acetate towards Sodium Carbonate, Sulphate, and Phosphate in presence of Invert Sugar.* A. Borntraeger. Zeits. angew. Chem. 1894, 521–528.

THE author's results show that the presence of large quantities of disodium phosphate exert but a trifling effect on the titration of invert sugar. For the estimation of reducing sugar in sweet and non-sweet wine this influence can be completely ignored. Sodium phosphate is, however, the only convenient compound whereby the excess of lead can be precipitated from sweet wines after clarification with basic lead acetate (sodium carbonate, and sodium sulphate were also tried). Before using the phosphate (and the same applies to carbonate) the basic lead acetate precipitate must be filtered off. The removal of the lead from a wine containing more than 1 per cent. of sugar is unnecessary. In the analysis of non-sweet wines sodium phosphate has no advantage over sodium carbonate as a reagent for the removal of lead; the same may be said also of sodium sulphate, and the latter has the special advantage that the precipitate produced by basic lead acetate need not be removed before using it. The wine, after neutralisation and removal of the alcohol, is treated with one-tenth its volume of basic lead acetate solution, and subsequently with a similar volume of a cold saturated solution sodium sulphate; after remaining for an hour it is filtered and titrated.—A. R. L.

*Removal of the Lead from Musts and Wines which have been Clarified with Lead Acetate.* A. Borntraeger. Zeits. angew. Chem. 1894, 554–559.

THE author's results show that sodium sulphate is a better reagent than sodium carbonate for precipitating lead from wines which have been clarified for analytical purposes with lead acetate.—A. R. L.

*Influence of Normal and of Basic Lead Acetate on the Cupric Reducing Power of Invert Sugar.* A. Borntraeger. Zeits. angew. Chem. 1894, 579–582.

REFERRING to his observations on the influence of lead on the titration of sugar in wine analysis (see preceding two abstracts) the author shows that normal lead acetate exerts but little influence on the cupric reducing power of invert sugar even when solutions of the two have remained together for a day; if, however, a solution of invert sugar is evaporated with lead acetate a diminution in the reducing power is observed, this being the more pronounced the larger the relative proportion of lead acetate present. The author also finds that 2.5 cc. of basic lead acetate solution, prepared according to the German pharmacopœia, per 100 cc. of invert sugar solution has practically no influence on the cupric reducing power of the latter even when the mixture has been kept for two days. A far less relative amount of basic lead acetate diminishes the cupric reducing power of invert sugar on being evaporated with it.

—A. R. L.

*Influence of the Volume occupied by the Basic Lead Acetate Precipitate on the Titration of Sugar in Wine Analysis.* A. Borntraeger. Zeits. angew. Chem. 1894, 583–591.

THE author's experiments show that the volume occupied by the lead precipitate, together with that obtained on eliminating the excess of lead by addition of sodium sulphate, has for practical purposes no influence on the titration of sugar in wine analysis; hence he considers it quite superfluous to wash this precipitate; its volume may be ignored.—A. R. L.

## ANALYTICAL AND SCIENTIFIC NOTES.

*Contributions to the Chemistry of Cellulose. 1. Cellulose Sulphuric Acid and the Products of its Hydrolysis.* A. L. Stern. Proc. Chem. Soc. 1894 [142], 186–187.

WHEN cotton cellulose is added to strong sulphuric acid, it dissolves; if the solution be diluted, neutralised with baryta, and filtered, a solution of barium cellulose disulphate is obtained. The free acid,  $C_{12}H_{19}O_9(SO_3H)_2$ , may be obtained by removing the barium by sulphuric acid; it is very unstable, and almost impossible to isolate. The barium salt is a white or slightly-coloured glassy powder, very hygroscopic, soluble in all proportions in water, and precipitated from strong solutions by alcohol.

When an aqueous solution of cellulose disulphuric acid is boiled with dilute sulphuric acid, it is hydrolysed. This reaction takes place in two stages, which are, however, not sharply marked off from one another. In the first stage, sulphuric acid is eliminated, and the disulphuric acid is gradually converted into cellulose monosulphuric acid,  $C_{12}H_{19}O_9 \cdot SO_3H$ , no other product being formed. In the second stage, cellulose acids containing less sulphuric acid are formed, and also dextrose. The analytical results indicate the presence of only two of these acids, i.e.,  $C_{12}H_{19}O_9 \cdot SO_3H$  and  $H \cdot C_{12}H_{11}O_9 \cdot SO_3H$ .

*On the Composition of the Waters of Coal Mines.* J. König. Zeits. angew. Chem. 1894, 389–391.

THE occurrence of heavy spar in or with coal has long been known, and believed to be due to the action of sulphates upon waters containing soluble barium salts. The frequent presence of barium and strontium in the strata immediately above and below the coal-bearing strata of the Devonian, Permian, and Triassic formations, would account for the reaction; it is therefore desirable to look for these elements in waters which are free from sulphates, although the small proportion of barium chloride as compared with the total chlorides, negatives the possibility of poisoning by any water that is at all drinkable. The following analyses of mine-waters were made, using R. Fresenius' method for the separation of the alkaline earth metals (this Journal 1893, 627); the results are given in grms. per litre:—

	1.	2.	3.
Cl .....	43.6128	19.8240	61.5960
Total $CO_2$ .....	0.2448	0.2518	0.1506
$CO_2$ combined with alkalis..	0.0412	0.0297	0.0112
Ba .....	0.9562	0.0175	0.6915
Sr .....	0.5034	0.1629	0.4163
Ca .....	4.0886	0.8357	3.9474
Mg .....	0.4004	0.2494	0.7555
K .....	0.9427	0.4348	0.8827

—W. G. M.

*A White Form of Tin Sulphide soluble in Ammonium Carbonate.* F. W. Schmidt. Ber. 1891, 27, 2739—2743.

In course of preparing pure tin, the author treated the sulphide with a 10 per cent. ammonium carbonate solution to dissolve traces of arsenic, and found that the subsequent addition of dilute sulphuric acid to the filtered solution brought down an exceedingly voluminous white precipitate, easily soluble in ammonia and ammonium sulphide, from which it could be re-precipitated in like form on acidification. Heated to 105° C., it became dark brown, and on roasting yielded  $\text{SnO}_2$ . In large quantities this white sulphide may be prepared by precipitating the pure yellow sulphide from a solution of stannous chloride previously peroxidised with nitric acid, boiling the precipitate with strong hydrochloric acid, to separate arsenic, filtering, and re-precipitating the filtrate by hydrogen sulphide, and digesting the well washed purified sulphide with ammonia solution, in which it dissolves in the course of a few days, leaving only traces of lead and bismuth sulphides; the addition of dilute sulphuric acid throws down an almost white precipitate, which dissolves readily in ammonium carbonate solution when freshly precipitated, but is only soluble to the extent of two-thirds after washing upon the filter, or of half after drying; the addition finally of dilute sulphuric acid to this solution gives the precipitate required. The intermediate, nearly white, precipitate from the ammonia solution evolves an odour of freshly-precipitated sulphur on drying, and becomes greenish-yellow in colour. It is partly soluble in ammonium and sodium carbonate solutions, leaving a brown residue, dissolves readily in ammonia, and is apparently represented by the formula  $\text{Sn}_2\text{S}_3\text{O} \cdot 11 \cdot 5 \text{H}_2\text{O}$ . On air-drying, the final white sulphide (from the ammonium carbonate solution) gives an amber-coloured mass easily crushed to a yellowish powder. At first it may be completely dissolved in ammonium carbonate, but the longer it is dried the less soluble it becomes, although even after a year it dissolves readily in ammonium, from which the completely-soluble modification is again precipitated by acid. This change in solubility appears to be due to an increasing compactness in form accompanying a loss of water, which is indicated by the formula obtained for the substance after one, three, and six months standing viz.:  $\text{Sn}_2\text{S}_3\text{O}$  with  $11 \text{H}_2\text{O}$ ,  $10 \cdot 5 \text{H}_2\text{O}$  and  $5 \text{H}_2\text{O}$  respectively. In each case the sulphur percentage is slightly too high, owing apparently to the presence of a small proportion of that element uncombined. The tin obtained from the white precipitate had an atomic weight of 118.5, which indicates the purity of the precipitate. The fact that the ordinary yellow precipitate of tin sulphide dissolves to the extent of 10 per cent. in ammonium carbonate solution must not be lost sight of in separations of tin from arsenic by means of that reagent.—W. G. M.

## New Books.

**HANDBUCH DER ANORGANISCHEN CHEMIE**, unter Mitwirkung von Drs. Benedict, Gadebusch, Haitinger, Lorenz, Philipp, Stavenhagen, und Profs. Dr. Ernst, Schellbach, von Sommaruga und Zeisel. Herausgegeben von Dr. O. DAMMER. Drei Bände. 1894. Stuttgart: Verlag von Ferdinand Enke. London: H. Grevel and Co., 33, King Street, Covent Garden. Price 4l. 8s.

THIS great work on Inorganic Chemistry is just completed, and is comprised in four large 8vo volumes, Vol. II. actually appearing in two books, the First and Second Parts, so that, whilst nominally a work of three volumes, it is actually one of four volumes. Vol. I., issued in 1892,

contains a Preface, a Table of Contents, and subject-matter covering 745 pages, the volume ending with an alphabetical index. It treats of the following branches of the subject:—

THE GENERAL PART, Introduction, Principles of Modern Research. I. *The General Properties of Matter.* 1. Gaseous; 2. Liquid; and 3. Solid States; 4. Physical Mixtures (Theory of Solutions). II. *The Properties of the Molecules.* 1. Determining Atomic Weights; 2. Determination of Molecular Weights; 3. Further Construction of the Molecular Hypothesis; Chemical Composition and Physical Properties. III. *The Changes of Matter.* 1. Law of the Chemical Action of Mass; 2. Chemical Statics, Homogeneous System; 3. Chemical Statics, Non-homogeneous, Complete Equilibrium; 4. Chemical Statics, Non-homogeneous, Incomplete Equilibrium; 5. Chemical Kinetics. IV. *The Changes of Energy.* 1. Heat and Chemical Energy (Thermo Chemistry); 2. Electrical and Chemical Energy (Electro-Chemistry); 3. Radiant and Chemical Energy (Photo-Chemistry).

SPECIAL PART.—Hydrogen, Oxygen, Chlorine, Bromine, Iodine, Fluorine. The Halogens generally. Sulphur, Selenium, Tellurium. Vol. II, Part 1, issued in 1891, contains a Table of Contents, 702 pages of subject-matter, and an alphabetical index, the text treating of the following items:—Nitrogen, Phosphorus, Arsenic, Antimony, Bismuth, Carbon, Silicon, Titanium, Germanium, Zirconium, Tin, and Thorium. Vol. II, Part 2, also issued in 1891, with Table of Contents, 943 pages of subject-matter, and alphabetical index, is devoted to a detailed treatment of the following metals, &c., and their compounds:—Potassium, Sodium, Lithium, Rubidium, Cesium, Ammonium, Calcium, Strontium, Barium, Beryllium, Magnesium, Zinc, Cadmium, Lead, Thallium, Copper, Silver, and Mercury. Vol. III., issued in 1893, contains Table of Contents, 928 pages of subject-matter, and the alphabetical index, and treats in the text of the following metals, their compounds, and other matters:—Yttrium, Cerium, Lanthanum, Didymium (Neodymium and Praseodymium), Erbium (Holmium, Thulium, Dysprosium), Terbium, Gadolinium, Samarium, Decipium, Ytterbium, Boron, Aluminium, Glass, Mortar, Earthenware, Ultramarine, Scandium, Gallium, Indium, Manganese, Iron, Cobalt, Nickel, Chromium, Molybdenum, Tungsten, Uranium, Vanadium, Tantalum, Niobium, Gold, Platinum, Ruthenium, Rhodium, Palladium, Iridium, and Osmium.

**TABELLÄRISCHE UEBERSICHT ÜBER DIE KÜNSTLICHEN ORGANISCHEN FÄRBSTOFFE UND IHRE ANWENDUNG IN FÄRBEREI UND ZEUGDRUCK.** VON DR. ADOLF LEHNE, Herausgeber der Färber-Zeitung. Sechste (Schluss) Lieferung. Berlin: Verlag von Julius Springer. 1894. London: H. Grevel and Co., 33, King Street, Covent Garden.

IN this, the sixth number, the Tabular Review of the Artificial Organic Colouring Matters has reached its conclusion. All the requisite tests are given for the dyestuffs described, the methods of application, solubilities, powers of resistance, and one column devoted to patterns and samples of materials dyed, printed, or represented in lakes, &c. This part, No. 6, commences with Rose Bengal and after reaching Cœrulein S, and SW, gives examples of the Indophenols, and then passes on to the Oxazines and Thiazines. On page 86 the Azines are represented and illustrated with (a) Eurlhodines and (b) Safranines. Examples and descriptions are then given of Artificial Indigo and Indigo Carmine in paste, and then follow the Quinoline and Acridine Colours, with the Thiobenzoyl dyes. The work concludes with Group XVI., devoted to Dyestuffs of unknown constitution, and a Table of Contents, List of Errata, and a comprehensive Alphabetical Index.

This work is closely connected with the well-known Tables of Schultz and Julius, by corresponding numbers placed against the several dyestuffs in a corresponding column running through both works. Each work thus serves as an index to the other, and the chemistry and technology of the dyestuffs represented, are therefore complete in the two sets of Tables of Schultz and Julius, and of Lehne.

CHEMISCH TECHNISCHES REPERTORIUM. Uebersichtlich geordnete Mittheilungen der neuesten Erfindungen, Fortschritte und Verbesserungen auf dem Gebiete der technischen und industriellen Chemie, mit Hinweis auf Maschinen, Apparate und Literatur. Herausgegeben von Dr. EMIL JACOBSEN. 1893. Zweites Halbjahr, Zweite Hälfte. Mit in der Text gedruckten Illustrationen. 1894. Berlin, S.W.: R. Gaertner's Verlagsbuchhandlung. Herman Heyfelder, Schönebergerstr. 26. London: H. Grevel and Co., 33, King Street, Covent Garden.

THE second issue for the second half-year of Dr. Jacobsen's Repertory of Chemical Technology.

It contains Reports of the progress of the following branches of Chemical Industry:—Foodstuffs; Paper; Photography; Refuse Matters, Manures; Disinfectants and Sanitation; Soaps; Explosives and Matches; Preparation and Purification of Chemicals; Chemical Analysis; Apparatus, Plant, and Machinery; Electro-technology and Thermo-technology; Adulteration of Trade Products; New Books; and lastly the Alphabetical Index of the four quarterly issues of the Repertorium for 1893.

## Prizes.

CITY AND GUILDS OF LONDON INSTITUTE FOR THE  
ADVANCEMENT OF TECHNICAL EDUCATION.

### Foundation of Research Fellowships.

The Court of the Salter's Company have placed at the disposal of this Institute a grant of 150*l.* a year for founding one or more fellowships for the encouragement of higher research in chemistry in its relation to manufactures, applications for which are invited.

The grant will be applied to founding one or more fellowships entitled "Salters' Company's Research Fellowships," for the encouragement of higher research in chemistry in its relation to manufactures.

The Research Fellowships will be awarded by the executive committee of the Institute, with the consent and during the pleasure of the Court of the Salter's Company, in accordance with the present scheme, or with such modifications as the Court of the Company may from time to time approve.

The amount of the grant attached to each Research Fellowship will be determined by the executive committee, with the approval of the Court of the Salter's Company, with reference to the nature of the research, the time required to complete it, and the merits of the candidate.

The executive committee will appoint a special committee to report on the selection of candidates and the progress of researches, and such committee will include the representative or representatives for the time being of the Salter's Company on the executive committee.

The executive committee will each year apply the sum provided by the Salter's Company to the award of fellowships to British-born subjects, of a value not exceeding 150*l.* (a) to students of the Institute who have completed a full three-years' course of instruction in the chemical department of the Central Technical College, or (b) to candidates duly qualified in the methods of chemical research in its relation to manufactures, without restriction as to age or place of previous study.

The executive committee will report to the Salter's Company the award of each Research Fellowship, and at the close of each session the results or progress of the research or researches undertaken during the session.

A Research Fellowship may be renewed for a second and third year, but shall not be held by anyone for more than three years.

The holders of Research Fellowships must devote their whole time to the prosecution of research, unless otherwise sanctioned by the executive committee; and must report as required on their work.

The researches are to be carried out at the Institute's Central Technical College, and the holders of Research Fellowships will be subject to the regulations of the college and the supervision of the board of studies.

Applications for Salter's Company's Research Fellowships must be made in writing addressed to the Honorary Secretary of the Institute, at Gresham College, Basinghall Street, E.C., and must state the nature of the research proposed to be undertaken, and the qualifications of the candidate.

The first award will be made early in the new year.

## Trade Report.

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

(From the Board of Trade Journal.)

#### RUSSIA.

##### Customs Decision.

Pound = 36 lb. airdupois; Rouble (gold) = 3*s.* 2*d.*

The following decisions affecting the classification of articles in the Russian Customs tariff have recently been given by the Russian Customs authorities:—

As articles liable to duty with toilet soap are to be considered all receptacles, wrappers, cardboard boxes, &c., which are intended for sale together with such soap.

Enamel paints, prepared with oil with an admixture of drying substances, such as turpentine, &c., to be cleared under section 137. Duty, 3 roubles gold per pound.

#### JAMAICA.

##### Tariff Changes.

The following changes in the rates of duty leviable in Jamaica on the importation of the undermentioned articles have been made by the Governor and Legislative Council of that colony, as set forth in Ordinance No. 13 of 1894:—

Ale, beer, and porter, 9*d.* per gallon.

Spirits:—brandy, 12*s.* 6*d.* per gall.; gin, 12*s.* 6*d.* per gall.; rum, the produce of and imported from British Possessions, 12*s.* 6*d.* per gall.; whiskey, 12*s.* 6*d.* per gall.

Spirits of wine, alcohol, and all other spirits, cordials, or spirituous compounds, except methylated spirits, 12*s.* 6*d.* per gall.

Spirits, methylated, 12½ per cent. *ad val.*

#### UNITED STATES.

##### Customs Decisions.

The following decisions respecting the construction to be given to Acts of Congress relating to the classification of articles in the Customs tariff, and the application of the Customs laws of the United States, have recently been given by the Customs authorities in that country:—

Collodion, a fine spun yarn or thread, used as a substitute for silk, to which it bears a striking resemblance, is dutiable at 60 cents a pound and 25 per cent. under paragraph 21, Act of October 1st, 1890.

Saké, a liquor made from rice by a process of fermentation and pressing, and not distilled liquor or spirits, containing from 11 to 14 per cent. of alcohol developed by fermentation, is dutiable at 50 cents per gallon under paragraph 336 as still wine.

Symphorol, or sodium caffeine sulphonate, a chemical salt and a chemical compound, and a medicinal proprietary preparation of which alcohol is not a component part, and in the preparation of which alcohol is not used, is dutiable at 25 per cent. *ad val.* under paragraph 76.

The residuum obtained in the distillation of petroleum, and partaking of the nature of pitch or bitumen, is dutiable under section 4 at 20 per cent. *ad val.*

Orthotoluidine, a coal-tar preparation, a chemical compound, and commercially known as aniline oil, is free of duty under paragraph 661 as aniline oil.

## NEW LAWS AFFECTING CHEMICAL INDUSTRIES.

### THE MARGARINE TRADE IN FRANCE.

In its sitting of the 20th July last, a bill was presented to the French Chamber of Representatives for the repression of fraud in the butter trade and in the manufacture of margarine. The French *Journal Officiel* for the first of November, publishes the text of the bill together with the *exposé des motifs* to the same. In the *exposé* it is pointed out that it is 25 years since Mège-Mouriez discovered a way to utilise tallow so that it could be used as a substitute for butter in cooking. This discovery was favourably received, and the Academy of Science granted a prize to the discoverer.

At first the invention of Mège-Mouriez was put to unscrupulous uses. Real butter was very dear, and some makers were addicted to palming off the margarine as such. As a result commercial credit suffered and demands for legislation to put a stop to the abuses came in from all sides. But, as the *Journal Officiel* observes, the evil from which agriculture is suffering is not confined to France, and France was not the country in which the adulteration originated.

In 1893 the exports of Mouriez margarine from France amounted to 7,456,647 kilos.; it has varied very little since 1887.

In Scandinavia the exports in 1893 exceeded 2,000,000 kilos.; in Belgium they were 3,437,325 kilos.; in the Netherlands they were not less than 74,000,000 kilos. England received from foreign countries in the same year 65,000,000 kilos. of margarine. The Netherlands was accountable for 60,000,000 kilos., and France for scarcely 2,000,000 kilos.

The complaints as to the unfair competition of margarine with butter are heard on every side. This has a detrimental effect on production, and brings about a reduction in the price of the butter.

The following is a précis of the French bill:—

Art. 1 prohibits the sale, import, and export of margarine as butter.

Art. 2 is to the effect that all food substances resembling butter are to be designated as "margarine."

Art. 3 proposes to prohibit the manufacture and sale of butter and margarine in the same building.

Art. 4 provides that the manufacturer of margarine should make a declaration to the communal authorities in the district where he intends to establish his factory; and Art. 5 requires the places where it is made or sold to bear the inscription "fabrique, dépôt ou débit de margarine ou d'oleo-margarine."

Art. 6 provides for the factories being subject to the supervision of government inspectors, and by Arts. 7 and 8 the duties and pay of these inspectors are fixed.

Art. 9 stipulates that all the necessary tubs, boxes, and papers used in the sale of margarine or oleo-margarine must bear distinct inscriptions of the names "margarine" and "oleo-margarine."

Art. 10 provides for the same process to be carried out as regards imports, exports, or consignments.

Art. 11 prohibits any other course from being followed than that indicated in the two preceding articles.

According to Art. 12, all invoices, way-bills, &c., must mention only margarine or oleo-margarine. The absence of such reference will be taken to indicate that the merchandise is butter.

Art. 13 provides that the inspectors mentioned in Art. 6, and, if necessary, special experts, may visit margarine factories, warehouses, Customs houses, &c., and take samples for analysis; and if fraud be discovered may take steps for criminal prosecution. By Art. 14 it is the duty of the Minister of Agriculture to supervise the proceedings to be taken as to analysis under the preceding section.

Art. 15 provides for the disposal of fees for analysis and fines for fraud.—*Board of Trade Journal.*

### NEW UNITED STATES LEGISLATION AS TO DISCHARGE OF REFUSE INTO STREAMS AND HARBOURS.

The River and Harbour Act of August 17th, 1894, contains the following section:—

Section 6. That it shall not be lawful to place, discharge, or deposit, by any process or in any manner, ballast, refuse, dirt, ashes, cinders, mud, sand, dredgings, sludge, acid, or any other matter of any kind other than that flowing from streets, sewers, and passing therefrom in a liquid state, in the waters of any harbour or river of the United States, for the improvement of which money has been appropriated by Congress, elsewhere than within the limits defined and permitted by the Secretary of War; neither shall it be lawful for any person or persons to move, destroy, or injure in any manner whatever any sea-wall, bulkhead, jetty, dike-levée, wharf, pier, or other work built by the United States, in whole or in part, for the preservation and improvement of any of its navigable rivers, or to prevent floods, or as boundary-marks, tide-gauges, surveying-stations, buoys, or other established marks; any and every such act is made a misdemeanour, and every person knowingly engaged in, or who shall knowingly aid, abet, authorise, or instigate a violation of this section shall, upon conviction, be punishable by fine or imprisonment, or both, such fine to be not less than two hundred and fifty dollars nor more than twenty-five hundred dollars, and the imprisonment to be not less than thirty days nor more than one year, either or both united, as the judge before whom conviction is obtained shall decide, one-half of said fine to be paid to the person or persons giving information which shall lead to conviction of this misdemeanour.

Sections 7 and 8 refer exclusively to boats or vessels dumping prohibited materials in any harbour contemplated in section 6. That section, however, as will be observed, has a much wider range. It is reported to have been introduced by Mr. Alderson, a representative from West Virginia, and to have been specially intended to apply to New River, in that State. But it may operate against many operations in coal-washing, ore-washing, stamp-milling, &c. elsewhere. Apparently, every such operation, if it discharges mud or other injurious refuse into the streams or harbours contemplated, is absolutely prohibited until the Secretary of War shall have granted specific and defined permission for it. And the waters thus protected are those of "any harbour or river of the United States for the improvement of which money has been appropriated by Congress." This clearly includes all past appropriations, as well as those contained in the act of which this section is a part. Congressmen who have heretofore secured public money for the improvement of insignificant streams, with the idea that they were making themselves popular among their constituents, may turn out to have subjected local industries to considerable vexation, through the jurisdiction thus conferred upon the general government. There is, however, no reason to anticipate more than a temporary inconvenience to such industries as are not in fact injuring navigable waters. The Secretary of War may be relied upon to use his discretionary power with common-sense. But it is evident that the practice of producing shoals for the government to dredge out, if it has anywhere obtained, will have to cease; and that the waters into which solid matter shall be hereafter permitted to be discharged, for the benefit of private enterprises, will not figure in the

river and harbour bills of the future. If they are really "navigable," individuals must let them alone; if they are not "navigable," the United States ought to spend no money on them.—*Engineering and Mining Journal*.

#### THE BRASS TRADE.

The Home Secretary (Mr. Asquith) has appointed a special committee to make inquiries into the condition of the brass trade, with the view, if necessary, to the construction of special rules for its conduct as an unhealthy occupation. The committee consists of Mr. W. Dawkins Cramp, one of the Superintending Inspectors of Factories and Workshops; Dr. R. M. Simon, of Birmingham; Mr. W. R. Lane, manager to Messrs. Winfield, Birmingham; Mr. W. J. Davis, secretary to the Brassworkers' Association; and Mr. Seymour Henry Knivett, one of H.M. Inspectors of Workshops in Birmingham. Dr. Simon, I understand, is appointed as a medical expert, who has given special attention to the conditions of the brass trades, and the health of those engaged in them. The committee is instructed to make "inquiry into and report on the conditions of work as they affect the health of the operatives in the processes of brass casting and mixing; brass dipping, polishing, and finishing, and in all other processes in brass manufacture; and also in the casting and mixing of gun-metal, bell-metal, and other kindred amalgams, and in particular to report what, if any, special rules should be made under section 8 of the Factory and Workshops Act, 1891, for the protection of the persons employed in such processes—whether the special rules so made should be applicable to all works in which such processes are carried on, or whether any exception should be made in cases where brass-casting, &c., is only a subsidiary operation." The committee will sit principally in Birmingham, but will visit other places where the brass trade is carried on. Mr. Knivett will be glad to receive communications from individual manufacturers and individual workmen, or associated bodies of either, who may wish to give evidence before the committee.—*Ironmonger*.

#### CHEMICAL WORKS AND THE SUNDAY-REST ACT.

A conference of chemical manufacturers met in Berlin last month to discuss the question of Sunday labour in chemical works (a stringent Sunday-rest Act being now in operation in Germany), and to frame representations to Government on the subject. It was decided to urge upon the authorities the absolute necessity of exempting from the Sunday Act all processes of reaction in the manufacture of organic dyes requiring more than 24 hours, as well as drying-room operations in the same industry. The permission to work on Sunday was further declared to be necessary in the sublimation of sal ammoniac, the feeding of the drying room ovens in mineral-paint works, in English-umber works, saltpetre works, camphor refineries, ceresin, cyanide of potassium, and sulphate of ammonia works. Many of the manufacturers present also demanded the exemption of the Easter, Whitsun, and Christmas holidays from the Act.—*Chemist and Druggist*.

#### PROCEEDINGS OF CHAMBERS OF COMMERCE.

##### MANCHESTER.

A meeting of the Board of Directors of the Manchester Chamber of Commerce was held on the 14th November, Sir F. Forbes Adam, C.I.E., in the chair.

In the minutes of the Chemical Sectional Committee a resolution appeared, suggesting that the preparation of evidence to be laid before the Parliamentary Committee on the Food and Drugs Act should be undertaken by a joint sub-committee selected from the members of the Produce and the Chemical Sectional Committees. The suggestion was made on the ground that in some cases the knowledge of chemical experts would be of essential service. With

this view the chairman of the former committee entirely concurred, and the Board accordingly agreed to the appointment of the joint committee.

On the proposition of the Chemical Sectional Committee the Board sanctioned the appointment of a deputation of chemical manufacturers to seek an interview with the Health Committee of the Manchester City Council, for the purpose of showing what steps have been taken by chemical manufacturers for the abatement of smoke, and to request that a distinction may be made, as already partly promised, between intermittent fires and ordinary steam-boiler fires. The resolution was approved by the Board.—*Board of Trade Journal*.

#### EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

##### THE PRODUCTION OF TARTARIC MATERIALS IN ITALY.

In a report to the Foreign Office, dated the 7th November, Mr. E. H. B. Hartwell, Her Majesty's Consul at Naples, submits a statement of the production of tartaric materials in Italy for the 12 months, 1st October 1893 to 30th September 1894. From this statement it appears that of half-refined tartar there were produced 1,266½ tons, valued at 70,955*l.*; of Vinaccia argols, 2,396 tons, valued at 109,070*l.*; of crude argols, 2,255½ tons, valued at 81,220*l.*; and of winelees, 2,676 tons, valued at 37,470*l.*; showing a total production of 8,594 tons, valued at 298,715*l.* The exports to the United States reached 3,949 tons, to Great Britain 994½ tons, to Germany 1,580½ tons, to France 419½ tons, and to other countries 1,650½ tons.—*Board of Trade Journal*.

#### GENERAL TRADE NOTES.

##### THE PETROLEUM SUPPLY OF THE CAUCASUS.

According to the *Moniteur Officiel du Commerce*, the petroleum springs existing around Baku do not appear to have lost their prolific character, at all events in the districts of Balakhani-Salrundschi, Bibi Kibat, and Romany, where the petroleum industry is concentrated. In 1893 the production of mineral oil was 337,051,834 pounds; of this quantity, 324,763,197 pounds (13·6 per cent. more than in 1892) underwent treatment in the local refineries, the remainder having been used for purposes fuel or lost. The increase arises chiefly from the fact that the springs, without the aid of pumps, supplied about one third of the aforesaid total. The price of the unrefined petroleum has been affected by this abundant supply; it has varied from 2·75 copecks per pound in winter to about 0·75 copeck in summer.

In 1893 there were 38 springs and 458 wells with pumps. A set of pipes, from 75 to 150 mm. in diameter and of a total length of 260 kilom., connecting the wells and the works, has permitted of the refining of about 1,440,000 pounds daily. There are only 85 refineries in work, and several have had but little to do, because in Russia, as everywhere else, the larger establishments are crushing out the smaller.

The refineries have produced and exported the following quantities:—Refined petroleum, 85,633,791 pounds; lubricating oils, 6,232,740 pounds; benzine, 252,493 pounds; tar and gasoline, 437,620 pounds; and petroleum residues, 145,538,399 pounds. To these figures there should be added those for local consumption.

According to the *Chemiker Zeitung*, the tools and processes employed by the refineries are often incomplete and defective; there is no longer pure water in sufficient quantity.

Sulphuric acid and caustic soda used for the treatment of petroleum are now made on the spot; four establishments supply about 9,000 tons of sulphuric acid, and three firms produce 2,700 tons of black acids, which are used for refining purposes. Two factories prepare soda, the one by means of Glauber salts (sulphate of soda), and the other according to the Leblanc method. As the residues have



increased in price and are generally sold at a higher price than the oil, 30 per cent. of the raw product only is distilled, instead of 36·5 per cent. as formerly.—*Ibid.*

#### THE RUSSIAN PETROLEUM (NAPHTHA) INDUSTRY, AND THE PETROLEUM OUTPUT IN 1893.

*F. Thiess. Journ. f. Gasbeleuchtung u. Wasserversorgung. 1894. 37, 704—706.*

In the petroleum industry of the Baku district the demand for the heavy residuals (masut, astatki, &c.), which

were formerly by-products of little value, has so increased during the last few years that these residuals have now become the most important products, and the kerosene really the by-product. The residuals are now very largely used (especially in Russia) for fuel on railways, steamers, and in factories; and quite recently an Astrakhan engineer is said to have successfully introduced their use for domestic fuel. For steamers this liquid fuel has the great advantage that it occupies little more than half as much storage space as the equivalent amount of coal.

The following table gives an idea of the growth of the liquid fuel (petroleum residuals) industry—

	1881.	1884.	1885.	1886.	1890.
Number of steamers using liquid fuel.....	..	126	..	1,507	1,824
Horse-power represented thereby.....	..	79,105	..	86,404	103,177
Tons of petroleum residuals consumed on these steamers.....	..	217,502	..	279,549	575,169
" " " " on Russian railways .....	1,900	..	82,607	..	280,138

The author calculates that in the last 13 years about 8,200,000 tons of residuals have been used on railways and steamers, &c. This would correspond to about 105,000,000 cub. yds. of wood (the former principal fuel), and would represent the saving from destruction of about 1,136,000 acres of forest.

The quantity of crude petroleum obtained in 1891 was 4,500,000 tons, in 1892 4,690,000 tons, and in 1893 5,320,000 tons. Of the latter quantity 1,790,000 tons were obtained from 38 fountain-springs, the remainder being pumped from wells. The present storage tanks in the Baku district number 250 and have a storage capacity of 452,920 tons, but it is proposed to erect further storage for about 820,000 tons. Of the petroleum collected in 1893 4,775,440 tons were worked up, with the production of 1,402,728 tons kerosene, 102,095 tons lubricating oil, 4,136 tons benzine, 7,758 tons asphalt, gasoline, &c., and 2,351,241 tons residuals. Thus 3·41 tons of crude petroleum were used for the production of one ton of kerosene, or burning oil. The sale of residuals increased from 1,013,068 tons in 1888 to 1,699,509 tons in 1891 and to 2,351,241 in 1893. The large increase of residuals is in part due to the fact that with the increasing demand for the latter and the falling demand for illuminating oils the distillation of the latter is not now carried so far as formerly. Thus formerly an average of 30—35 per cent. of the crude oil was distilled off for illuminating oil, whilst now only about 20—25 per cent. is so distilled off. In fact, the distillation is sometimes so reduced that the dangerously light and inflammable oils are left in the so-called heavy residuals and becomes virtually a process of adulteration. The annual increase in residuals exported (mainly by the Caspian) averaged in recent years 196,566 tons, whilst it was 425,893 tons in the first ten months of 1893.

Until recently the Russian petroleum industry was almost exclusively confined to the rich petroleum fields south of the Caucasus, but lately petroleum wells have been successfully sunk and worked in the Terek (or Tersk) district north of the Caucasus. In one boring of about 400 ft. deep a petroleum fountain was produced throwing out about 4,900 tons in 24 hours, with such force as to destroy much of the boring apparatus. New pipe lines and railways bring this district in connection with the Caspian. Very extensive new pipe lines are also planned both in this and the Baku districts for facilitating the petroleum transport.

—L. T. T.

#### THE HUNGARIAN COMMERCIAL MUSEUM AT BUDAPEST.

The primary aim of this State-supported institution is to encourage the sale of native manufactures. It consists at present of an enquiry office, library, museum, and a sale department, and it runs a trade newspaper and occasional special exhibitions. It is under the control of a society with a membership of about 800, strictly limited to Hungarian manufacturers, no traders being admitted.

The income of the society consists of:—(1) annual subscriptions of members at 10 florins; (2) rent of space in the exhibitions, at 2 florins per square metre in the open, and 5 florins per square metre for glass cases; and (3) a commission of 2½ per cent. on all sales. Any deficit is met by the State.

The staff consists of 5 officers and 1 servant (enquiry office), 1 officer and 1 servant (library), 12 officers and 15 servants (museum), and 42 officers and 12 servants in the sales department. The enquiry office (Academic Strasse 12), open from 9 a.m. to midday and again from 3 to 6 p.m., affords general information as to customs and freights. Members also may obtain information as to foreign credits. The library contains works on law, political economy, and trade, together with directories and newspapers. The trade newspaper of the museum (*Kereskedelmi Közlemények*), issued once a week to members only, contains:—consular, trade, market and exhibition reports, statistics, and home and foreign commercial and technical notices. The museum is devoted to foreign samples and models for native imitation, and native goods and samples. It offers special facilities to the small craftsman, who possesses no showroom, to exhibit and sell his work. The large producer also finds it to his advantage, for the museum sells his products from the exhibited samples and models. Six inspectors or store-keepers are entrusted with the safe custody of the exhibits, each one of whom is under guarantee to the small extent of 300 florins, and sells the exhibits in person.

The museum is situated in the central pavilion or "Industrial Hall" built for the Exhibition of 1885. The ground space occupies 10,000 square metres, while the vertical show space can be increased at will by means of movable screens (écrans). The museum supplies glass cases, but private cases are also admitted. Only members may exhibit; each one is entitled to free ground space to the extent of one square metre.

The visitors in 1893 numbered 80,000. No charge for admission is made, except on Sunday afternoons. The exhibits consist of Agricultural and Horticultural implements; Farm products, such as milk, cheese, and butter; silk, honey, wool, feathers, and bristles; Products of woods, forests, and mines; Chemicals; Foods and Beverages; Ceramics; Metals; Clothes; Furniture; Gold and Silver work; Millinery; Printing; Musical and Scientific Instruments, Carriages; Machines; Road, Canal, and Railway Constructions; Posts and Telegraphs; Naval and Fishery appliances; Equipments; Hospital and other Sanitary appliances; Home Industries, and Educational appliances.

The annual special exhibitions are not exclusively Hungarian. Up to the present the following have been held—Railways and Railway appliances; Leather; Pottery and Earthenware; and Electrical appliances.

*Sale Department.*—The permanent museum at Budapest does the largest business. Twelve branches are also now open, viz.: Serajevo, Banialuka, Mostar, Bukarest, Saloniki, Bricksa, Fiume, Belgrade, Sofia, Philippolis, Rust.



chuk, and Constantinople. This number will shortly be increased.

Prior to the opening of the first foreign branch at Sarajevo in 1891, Hungarian goods to the value of four million florins had been sold by the museum. Each member can correspond direct with the foreign sale branches and receive therefrom any required information free of cost.

The central museum has only at present a temporary home pending the completion of the great Millennial Exhibition of 1896 to commemorate the foundation of the Hungarian State by Arpad. During this Exhibition the museum officers will be transferred to the Stephanie Weg, Stadtwaldchen.

The President of the museum is the Hungarian Minister of Trade, and the Vice-President is the Secretary of State for the same department. The Committee consists of seven and the Council of 28 members, all nominated by the Minister of Trade.—*J. Lazarus.*

#### THE INDIAN SHELLAC INDUSTRY.

Dr. George Watt, Economic Reporter to the Government of India, observes, in a report recently drawn up by him, that within recent years the demands for shell and button lac have far exceeded the supply, so that it may be said that a considerable revival of interest in this peculiarly Indian article of trade has taken place. During the past five years the exports have increased 95 per cent. in value, and only 21 per cent. in quantity, and the returns would appear to manifest a steady improvement. But it would be unwise to advance too emphatic assurances, since the history of the trade has often manifested the wildest eccentricities. During the past 18 years the lowest exports were in 1879-80, viz., 57,335 cwt., valued at 33,490,600 rs., but, as showing the erratic nature of the trade, it may be mentioned the figures for the immediately preceding year were 83,722 cwt., valued at only 2,895,430 rs. Still more puzzling are the returns for 1875-76, viz., 92,915 cwt., valued at 7,291,760 rs.; and of 1876-77, 109,665 cwt., at 4,991,230 rs. It will thus be seen that few articles of Indian export trade manifest more sudden changes than that of lac. To the people of India it might almost be said the interest in lac lies in the fact that it is collected from wild sources, and that the industry is a positive boon to people in tracts of country that stand greatly in need of such aid. Its encouragement is, therefore, of considerable consequence, though, as compared with other specially Indian products, it is not of much value. There are 21 native lac factories, and one European at Mirzapore, in the North-West Provinces. In Bengal there are 35 factories, but, excepting the Cossipore Factory near Calcutta, they are all small native works. There are, for example, 25 native shellac factories in Manbhoom, one in Beerbhoom, one in Palamow, and seven in Lohardugga. These various factories give employment to 2,614 permanent hands, and 1,665 additional ones during the working season.—*Chemist and Druggist.*

#### NITRATES IN EGYPT.

##### *Report of the Commission.*

This report to the Egyptian Council of Ministers deals with the nitrate-bearing clay in Upper Egypt, which commonly contains nitrates equivalent to 15 per cent. of sodium nitrate. The supply is stated to be practically inexhaustible. At a distance of 760 kilometres south of Cairo, on the east bank of the Nile, are low ranges of hills at the foot of the principal range of limestone cliffs. These "foothills," which were examined for a length of 15 miles, are formed of nitrate-bearing clays with a thin cap or covering of limestone. The clay is termed "Marog," and is a blue or grey foliated marl. Large numbers of cultivators are engaged at the appropriate season in removing the clay for use in the neighbourhood as a fertiliser. Rains appear to remove the

nitrate rapidly, but it was the opinion of the workers on the spot that under favourable conditions nitrification goes on equally rapidly. They rely upon the taste in judging whether the clay is in a good, or nitrated, condition, when it is said to be "hami" or bitter. With regard to the mode of nitrification the authors point out that "in these hot valleys there is no trace of organic life nor of vegetable matter . . . and . . . microscopical organisms cannot here be the active agent, for the sun's heat is too intense."

The nitrate-bearing clay is supposed to occur over an area stretching from 24° to 27° N. lat., and from 28° to 34° E. long. Without the use of this "marog," agriculture, it is said, would be impossible between Assuan and Armut. Subjoined are analyses of "marog" made by Dr. Mackenzie at the Tewfikia College of Agriculture.

	No. 1.	No. 2.	No. 3.	No. 4.
Moisture (dried at 100° C.).....	2'44	1'83	3'42	2'68
Silica.....	19'34	10'37	22'56	22'81
Carbon dioxide.....	14'77	8'43	7'02	5'92
Phosphoric acid.....	0'57	1'72	0'29	0'27
Sulphuric acid.....	3'82	0'87	7'13	6'87
Nitric acid (N <sub>2</sub> O <sub>5</sub> ).....	9'40	9'94	8'82	11'77
Chlorine.....	4'90	12'62	6'31	6'31
Iron oxide.....	4'37	4'37	5'65	5'27
Alumina.....	11'03	6'88	12'94	12'99
Lime.....	18'90	16'31	13'36	11'51
Magnesia.....	0'67	1'13	1'12	0'90
Potash.....	0'10	3'45	0'18	0'21
Soda and combined water, by diff.	10'74	15'77	12'55	13'84
	101'05	102'69	101'35	101'35

—Cairo National Printing Office.

#### ADULTERATION OR IMPROVEMENT OF CEMENT?

We are indebted to our contemporaries the *Ironmonger, Journal of the London Chamber of Commerce*, and the *Chemical Trade Journal* for the following particulars:—

A meeting of English Portland cement manufacturers was held recently in London, called on the initiative of Knight, Bevan, and Sturge, "for the purpose of establishing an association of English cement manufacturers," and "of dealing with, and, if possible, of putting a stop to, the unprincipled and disreputable practice" of "adulterating cement by the mixture of Kentish rag-stone, other stone, furnace, or oven ashes, disused or exhausted firebricks, and other inert material." It was attended by representatives of 29 manufacturers of Portland cement, Mr. Bevan occupying the chair. There were considerable differences of opinion on various points. Some of those present approved of the proposed association; others decidedly objected to it; and others preferred leaving the question of adulteration in the hands of the London Chamber of Commerce. One gentleman, Mr. Holt (of Macevoy, Holt, and Co.), boldly denied that the addition of Kentish rag-stone was an adulteration. He frankly admitted having used it, and after two years' experience declared it improved the cement in point of colour and in its tensile strain: the sand tests were higher, and in every respect it was a better article. It did not follow, he said, that because Portland cement had been made for 30 or 40 years by the admixture of chalk and clay that there was no other article by which it could be improved. Mr. Holt quoted figures in support of his views, and indignantly repudiated the charge of "adulteration." Mr. Leedham White (J. Bazley White and Brothers) said English cement manufacturers were making a better article than they did some years ago, but he admitted they had been distanced by

German skill. He thought they ought not to be debarred from imitating that German skill, from taking advantage of scientific research, and from making all the progress they could. He held, therefore, that the matter should be sifted by a qualified body in a scientific manner, and then fully reported upon. Ultimately the meeting broke up, and several firms signed a statutory declaration, joining the association, and avowing that they never had "adulterated" their cement the manner indicated.

In this connection it is instructive to note what the Germans have done in the same direction. According to the *Chamber of Commerce Journal*, early in the year 1880, when adulteration threatened to become very prevalent, the Union of German Cement Manufacturers found itself compelled to set its face in the strongest manner possible against the admixture of foreign materials with cement, both in order to protect the good name of German Portland cement, and at the same time to protect the building trade from disadvantage and danger. There have not been wanting, on the other hand, attempts to defend the admixture of foreign materials with the cement. It has been asserted that the strength of Portland cement was more than sufficient for many purposes, and that it was in the interest of the building trade to produce for them a cement which should be cheaper, and yet sufficiently strong for their requirements. It was even argued that the admixtures employed were an improvement to the cement, and in support of this assertion the defenders of the system pointed to the investigations of Professor Tetmeyer and Dr. Michaelis, who by certain admixtures obtained an increase in the strength of the cement.

With regard to the first point (cheapness), the Union insisted that it must be left to the consumer, if he wished to cheapen cement, to make the desired admixtures himself. If either the manufacturer or the dealer were permitted to do this, the door would be opened wide to fraud, and the confidence of the public in Portland cement would be shattered, for the consumer would not be in a position to know or to test either the degree of the adulteration or the characteristics of the added material.

With regard to the second point (*i.e.*, the alleged improvement), it was certainly known that by the addition of ultramarine, precipitated silicic acid, preparations of potters' clay, and such like, the strength of Portland cement could be increased, but it is still doubtful whether the other valuable qualities of Portland cement do not suffer, and, moreover, these admixtures are excluded from us, if only by reason of their high price. If the defenders of the system point to the admixtures of "compositions" made by Michaelis and Tetmeyer in their laboratory experiments, and the constitution of which was not made public, there is no evidence that the materials used in the works where this adulteration is practised are identical with the materials used by these gentlemen.

It is quite certain that the quality of cement is not in the least improved by the granulated blast furnace slag, which is used for the production of Puzzuolan cement, and which hardens well when mixed with lime. On the other hand, the ordinary ground or disintegrated slag (and it goes without saying that the same remark applies to ground limestone) deteriorates the quality of the cement, and is to be regarded simply as "ballast" in cement.

It has been found that adulteration is chiefly practised with these two materials, and it is agreed that the mixed cements hitherto put upon the market were all of little value, and they have been so considered to this day.

For all these reasons, the Union of German Cement Makers, which since 1888 has borne the title of the "Union of German Portland Cement Manufacturers," has continuously exerted itself to root out, as far as possible, this system of adulteration.

With this view the Union has drawn up certain information as to what may legitimately be added to cement and what may not. The following were the conclusions arrived at:—

**Admixture of Gypsum.**—The addition of unburnt gypsum, made at the time of grinding, has for its advantageous object

the retarding of the setting of such Portland cements as are by their nature quick setting, and thereby rendering them more fit for use. The action of the gypsum is probably owing to the fact that when the cement is mixed with water into mortar, the gypsum is first dissolved, and then precipitated in extremely fine particles on the grains of the cement, in exactly the same way as it is recommended for lime by F. Schott. (*See Dingler's Journal*, 207, 52, and 209, 30.) If the particles of cement are enveloped in the thinnest possible film of gypsum, the chemical incorporation of the water is notably delayed. In consequence of this the cement becomes slower setting, and by this means (possibly also by the simultaneous occurrence of some chemical action) cement, mixed with a little gypsum, gives a higher degree of strength than the unmixed quick-setting cement. Inasmuch as the addition of from one-half to two per cent. of gypsum is sufficient for the attainment of this object; and as, moreover, the addition of a larger percentage would cause the cement to "blow," there can be no possible ground for asserting that the admixture of gypsum is made with a fraudulent intention; nay, more, such an addition, within the prescribed limits, must be regarded as an improvement. In consequence of this the united declaration of the German Portland cement manufacturers specially permits additions, not exceeding 2 per cent., if made with the object of regulating the time of setting.

**Fraudulent Admixtures.**—Of materials which may from time to time be mixed with Portland cement with the intention of adding to the profits, and without open acknowledgment of such admixture, it is evident that only those can be employed which are cheap, and which resemble Portland cement both in weight and colour. Chief amongst such materials are slag, principally blast furnace slag, grey limestone, slate, hydraulic lime, black or grey limestone, and trass. The most popular adulterant is slag, because, when pulverised, it is very similar in appearance to Portland cement, and is therefore less easily detected, even when added in large proportions.

All the foregoing admixtures make the cement inferior, not only in respect of strength, but also in respect of those other valuable qualities (or characteristics) of Portland cement, such as resistance to frost, durability, cohesiveness, &c., as has been proved by the most exhaustive researches.

The subsequent proceedings of the English movement have been as follows:—

At a meeting of the cement trade, held at the London Chamber of Commerce, it was resolved—"That a committee be appointed, with power to engage experts to investigate into the question of the admixture of Kentish rag-stone, other stone or other material with Portland cement, so far as carried on at home and on the Continent, and to report and advise thereon." Representatives of 11 firms were appointed upon the committee. The formation of a Cement Manufacturers' Association, with the object of stopping adulteration, is also well on the way towards completion.

The above-mentioned committee appears now to have decided that Kentish rag-stone is not a desirable constituent in Portland cement, for at another meeting it is reported by *Industries and Iron* to have resolved with only one dissentient "that it is advisable that all manufacturers of Portland cement who have used any outside material in their manufacture should at once discontinue the practice." The use, however, of not more than 2 per cent. of gypsum, as practised by the German manufacturers for the purpose of regulating the setting of the cement, is considered optional.

#### THE GERMAN CEMENT TRADE.

There are upwards of 63 cement works in Germany. The year's output is about 10,600,000 barrels, and in the production of this quantity of cement 18,000 workmen were employed in 1893, occupying 5,500,000 working days, and earning 14,250,000 marks in wages.

Near Hamburg there are three or four factories for the production of this article, but the centre of the production is on the Rhine. The largest customers for cement are the United States, Brazil, Chile, and Venezuela, and in Europe,

Norway, and Russia, as follows, quantity in cwt., and value in pounds :—

Country.	Quantity.	Value.
United States.....	1,386,872	168,023
Brazil.....	446,338	40,280
Chile.....	131,067	13,102
Venezuela.....	102,904	9,736
Norway.....	58,356	6,709
Russia.....	34,353	2,941

The importation to Great Britain was insignificant, but British possessions, such as Australia, the Cape, and British East India, took 123,666 cwt., valued at 12,247*l.*--*Chemical Trade Journal.*

#### THE COMPARATIVE WORKING OF COKE OVENS.

According to the report of Mr. Farrell, an American furnace engineer, who spent three months in Europe last year investigating the different coking systems, and who had access to the records of all leading oven builders, the following is a comparison of results in by-products, as accomplished by ovens of the different models working on similar grades of Westphalian coal :—

Names.	Period.	Tar.	Sulphate of Ammonia.
		Per Cent.	Per Cent.
Semet-Solvay.....	Oct. 1892 to Mar. 1893	1·6	0·61
Carvès-Hussener..	Oct. 1892 to Feb. 1893	1·81	1·0
Otto Hoffman....	Oct. 1892 to Mar. 1893	3·53	1·18

—*Ibid.*

#### GERMAN ELECTRO-CHEMICAL SOCIETY.

This new society held its first meeting at Berlin recently. The object of the society is the development of electro-chemistry in its scientific, technical, and economic aspects. Amongst the first communications to the society is one by Professor Ostwald on the work done by J. W. Ritter, a contemporary of Galvani and Volta, who was the first to perceive that Volta's electro-chemical arrangement of the metals corresponded to the order of their affinity for oxygen. Dr. O. Frölich has communicated a paper on "The Practical Application of Ozone," which has been introduced into many different branches of manufacture, the most important of these being bleaching, the ageing of spirits, tobacco, and coffee, and also of wood for the sounding-boards of pianos, for which latter purpose it has been applied by a piano manufacturer, Mr. René, of Stettin, since 1881. In this case the chemical change due to action of ozone on the resins contained in the wood is probably an important factor. The thickening of linseed oil, which is effected in linoleum manufactories by allowing it to trickle over strips of gauze for several months, can in like manner be done with ozonised air in a few days. The conversion of crude potato starch into the soluble variety, as well as into dextrin and other gummy substances by ozone, has been effected by Dr. Frölich, and the method is to be introduced practically into a manufactory now in process of construction. Dr. Frölich also hopes to accomplish the production of nitric acid from the air by means of ozone.—*Chemist and Druggist.*

#### BOARD OF TRADE RETURNS.

##### SUMMARY OF IMPORTS.

Articles.	Month ending 30th November	
	1893.	1894.
	£	£
Metals.....	1,670,249	1,481,266
Chemicals and dyestuffs.....	474,816	421,196
Oils.....	552,965	642,351
Raw materials for non-textile industries.	3,299,044	3,683,795
Total value of all imports....	35,801,961	35,236,790

##### SUMMARY OF EXPORTS.

Articles.	Month ending 30th November	
	1893.	1894.
	£	£
Metals (other than machinery) ....	2,162,042	2,428,450
Chemicals and medicines .....	674,972	708,077
Miscellaneous articles.....	2,357,699	2,444,388
Total value of all exports.....	17,653,759	18,083,087

##### IMPORTS OF METALS FOR MONTH ENDING 30TH NOVEMBER.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
Copper :—			£	£
Ore..... Tons	9,892	6,764	62,373	35,024
Regulus .....	8,106	5,915	188,192	112,584
Unwrought ....	5,042	5,029	220,008	203,353
Iron :—				
Ore..... "	221,392	303,128	164,296	195,375
Bolt, bar, &c.... "	7,172	7,378	66,576	59,615
Steel, unwrought.. "	708	1,548	6,046	10,975
Lead, pig and sheet "	13,101	11,553	131,550	113,293
Pyrites .....	35,586	30,298	63,491	67,513
Quicksilver..... Lb.	8,000	82,392	600	7,050
Silver ore..... Value £	..	..	260,585	178,642
Tin..... Cwt.	77,835	82,510	305,368	281,672
Zinc..... Tons	5,569	4,369	99,303	66,182
Other articles ... Value £	..	..	101,861	149,988
Total value of metals	..	..	1,670,249	1,481,266

## IMPORTS OF OILS FOR MONTH ENDING 30TH NOVEMBER.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Cocoa-nut..... Cwt.	11,152	22,292	11,188	22,189
Olive ..... Tuns	556	398	21,053	33,574
Palm ..... Cwt.	93,184	88,385	113,820	93,146
Petroleum ..... Gall.	13,018,560	16,879,555	208,085	244,573
Seed ..... Tons	2,668	2,711	63,947	59,004
Train, &c..... Tuns	2,359	1,172	14,848	26,913
Turpentine ..... Cwt.	19,940	68,591	21,782	67,311
Other articles .. Value £	..	..	63,033	84,244
Total value of oils ...	..	..	552,965	612,351

## IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDING 30TH NOVEMBER.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Bark, Peruvian .. Cwt.	2,634	3,285	5,956	7,538
Bristles..... Lb.	161,883	107,765	20,114	52,360
Caoutchouc..... Cwt.	29,141	24,195	331,527	254,360
Gum:—				
Arabic..... "	5,401	5,186	21,102	14,922
Lac, &c..... "	10,594	12,444	50,294	65,026
Gutta-percha .... "	3,618	2,175	29,601	16,919
Hides, raw:—				
Dry..... "	20,463	22,964	50,332	52,188
Wet..... "	10,672	58,116	87,860	123,066
Ivory..... "	190	1,053	10,200	45,861
Manure:—				
Guano..... Tons	1,502	3,350	7,184	17,214
Bones..... "	3,566	9,785	17,081	41,298
Nitrate of soda... "	673	8,813	6,061	79,628
Phosphate of lime "	15,767	34,290	27,959	72,242
Paraffin..... Cwt.	75,897	69,119	79,314	70,933
Linen rags..... Tons	1,096	1,803	10,691	17,484
Esparto..... "	8,095	10,239	36,180	46,456
Pulp of wood .... "	19,396	27,592	99,126	159,274
Rosin..... Cwt.	43,551	61,460	9,544	11,616
Tallow and stearin "	62,605	115,307	83,172	141,449
Tar ..... Barrels	3,390	5,609	3,539	5,004
Wood:—				
Hewn ..... Loads	162,170	174,220	303,989	359,987
Sawn ..... "	466,292	479,439	986,193	1,000,259
Staves ..... "	19,047	10,713	72,041	48,082
Mahogany ..... Tons	6,847	4,450	57,337	34,312
Other articles.... Value £	..	..	892,290	957,137
Total value .....	..	..	3,299,014	3,683,795

Besides the above, drugs to the value of 65,767*l.* were imported as against 62,198*l.* in November 1893.

## IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH ENDING 30TH NOVEMBER.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Alkali..... Cwt.	9,078	14,078	9,174	11,414
Bark (tanners', &c.) "	209,57	24,739	6,747	9,943
Bluestone ..... "	14,984	19,536	3,485	4,742
Chemicals..... Value £	..	..	128,429	125,697
Coal-nut ..... Cwt.	484	384	3,015	2,324
Cutch and gambur Tons	4,748	2,242	70,880	45,142
Dyes:—				
Aniline..... Value £	..	..	19,277	31,204
Alizarin ..... "	..	..	29,417	24,435
Other ..... "	..	..	2,496	4,444
Indigo ..... Cwt.	3,865	784	74,369	11,696
Nitrate of potash . "	18,888	24,827	18,247	22,291
Valonia..... Tons	1,344	2,270	16,973	27,262
Other articles... Value £	..	..	56,581	109,347
Total value of chemicals	..	..	474,840	421,196

## EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDING 30TH NOVEMBER.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Gunpowder..... Lb.	395,400	643,000	16,194	18,636
Military stores.. Value £	..	..	87,288	100,057
Candles..... Lb.	1,550,200	2,057,100	28,948	34,225
Caoutchouc..... Value £	..	..	97,092	106,451
Cement..... Tons	32,898	24,284	55,210	42,863
Products of coal Value £	..	..	106,879	117,910
Earthenware ... "	..	..	146,799	161,544
Stoneware ..... "	..	..	11,652	13,429
Glass:—				
Plate..... Sq. Ft.	118,549	121,640	6,524	6,729
Flint..... Cwt.	7,673	6,945	19,206	18,503
Bottles..... "	48,559	54,894	22,420	24,492
Other kinds.... "	13,537	18,894	11,497	14,476
Leather:—				
Unwrought .... "	12,385	11,351	111,118	102,412
Wrought ..... Value £	..	..	29,768	24,695
Seed oil..... Tons	4,743	4,415	108,844	91,779
Floorcloth ..... Sq. Yds.	1,126,800	1,060,900	65,986	59,193
Painters' materials Val. £	..	..	198,094	116,631
Paper ..... Cwt.	71,949	75,940	117,485	121,549
Rags..... Tons	3,730	4,642	24,547	26,706
Soap..... Cwt.	58,246	48,841	60,593	51,884
Total value .....	..	..	2,557,809	2,444,388

EXPORTS OF METALS (OTHER THAN MACHINERY) FOR  
MONTH ENDING 30TH NOVEMBER.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Brass..... Cwt.	9,781	8,349	56,585	32,055
Copper:—				
Unwrought..... „	24,290	31,157	55,993	67,240
Wrought..... „	26,010	24,343	72,513	65,274
Mixed metal..... „	23,777	27,518	51,985	59,395
Hardware..... Value £	..	..	163,242	172,593
Implements..... „	..	..	102,045	101,987
Iron and steel..... Tons	184,835	235,042	1,490,913	1,681,259
Lead..... „	3,355	3,231	38,161	38,580
Plated wares... Value £	..	..	34,359	44,673
Telegraph wires „	..	..	19,520	49,468
Tin..... Cwt.	10,336	11,419	43,294	49,550
Zinc..... „	16,443	18,263	12,469	11,887
Other articles.. Value £	..	..	71,863	63,210
Total value.....	..	..	2,162,042	2,428,450

EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING  
30TH NOVEMBER.

Articles.	Quantities.		Values.	
	1893.	1894.	1893.	1894.
			£	£
Alkali..... Cwt.	477,014	721,025	134,640	171,228
Bleaching materials „	68,747	130,450	28,575	49,654
Chemical manures. Tons	20,240	16,547	162,587	141,522
Medicines..... Value £	..	..	82,715	86,270
Other articles... „	..	..	266,455	258,703
Total value.....	..	..	674,972	708,077

## Monthly Patent List.

\* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### APPLICATIONS.

22,524. J. H. Bowden. Improvements in apparatus for drying china-clay, stoneware, or other suitable materials. November 21.

23,031. H. J. Haddan.—From the Ferry Manufacturing Co., United States. Improvements in apparatus for carbonating liquids. Complete Specification. November 27.

23,032. H. J. Haddan.—From the Ferry Manufacturing Co., United States. Improvements in the method of and means for carbonating liquids. November 27.

23,220. J. A. Yeadon and W. Adgie. Improvements in apparatus for producing or revivifying animal charcoal and other analogous materials. November 30.

23,235. J. Foster. Improvements in and relating to evaporating apparatus. November 30.

23,956. S. J. Robinson and S. Pope. Improvements in open hearth steel-melting furnaces and regenerative furnaces generally. December 10.

23,961. J. Weir. Improvements in and connected with steam-heated evaporators. December 10.

24,199. J. A. Fisher. Improvements in non-conducting compositions for coating boilers and for like uses. December 12.

24,447. M. Otto. Improvements in apparatus for distillation. December 15.

24,457. E. G. Scott. Improvements in evaporating brine and other liquors, and apparatus therefor. December 15.

#### COMPLETE SPECIFICATIONS ACCEPTED.\*

##### 1893.

22,736. A. F. Beyer and A. G. Beyer. Improvements in and connected with apparatus for cooling, solidifying, and drying substances in a liquid or viscous state. November 28.

23,069. R. Cunliffe. See Class XVII.

##### 1894.

1790. D. Stewart. See Class XVI.

1866. O. Hamilton. A new or improved drying apparatus. December 5.

3665. H. Evers-Swindell. Improved manufacture of gas retorts, also applicable to other tubular objects of fire-clay or earthenware, and apparatus therefor. December 19.

3702. W. Chorley. Improvements in machinery for the compression of air or other elastic fluids. December 19.

18,992. H. L. Doulton and A. W. Manger. Improvements in the process of and apparatus for softening waters and purifying waste liquors. November 28.

19,085. A. Müller. Improvements in apparatus for effecting the absorption of gases by liquids and for cooling gases. November 28.

20,202. E. Entwistle.—From W. L. Wallis. Improvements in means and apparatus for treating smoke and fumes. November 28.

21,326. W. P. Thompson. — From J. Fathrieger. Improvements in muffle furnaces. December 12.

21,778. I. S. McDougall. Improvements in evaporating pans for the concentration of sulphuric acid and other liquids. December 19.

## II.—FUEL, GAS, AND LIGHT.

### APPLICATIONS.

17,71A. W. Felton. Improvements in apparatus for producing and burning gas to heat furnaces. Filed November 1, date claimed September 18, 1894.

22,452. H. A. Allport and A. Crossley. Improvements in coke ovens and in the method of removing and utilising the products of combustion from such coke ovens. November 20.

22,504. S. Banner. Improvements in deodorising and bleaching hydrocarbons and volatile oils or spirits. November 21.

22,615. L. Denayrouze. Method of and apparatus for illumination by means of combustible gas with forced supply produced by electrical energy. November 22.

23,230. R. V. Horsfall. Improvements in retorts for distilling, cracking, or gasifying oils, tars, or any liquid hydrocarbons. November 30.

23,278. J. M. Bosselaar and J. F. M. van den Elshout. Improvements relating to the manufacture of gas and to apparatus therefor. November 30.

23,297. N. van der Sleen and A. Schnellen. Improvements in and relating to an apparatus and a method for causing chemical changes of gases and mixtures of gases by dark electric discharges. December 1.

23,559. F. J. Koopmann. Improvements relating to the manufacture of artificial fuel. December 4.

23,631. A. Greg. The purification of and utilisation of wood-gas for illuminating purposes. December 5.

23,662. J. Henderson. Improvements in or relating to gas retorts and such like apparatus. December 5.

23,839. W. S. Tailleur. Improvements in the utilisation of natural and artificial mineral or hydrocarbon oils and their various products and by-products in the production of heat and making and enriching gas. December 7.

23,996. C. Weygang and The Petroleum Products Syndicate, Ltd. Manufacture of artificial fuel from petroleum. Complete Specification. December 10.

24,005. A. G. Glasgow. Improvements in the method and apparatus for carburetting water-gas. Complete Specification. December 10.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

22,638. E. de Pass. — From L. J. Baptiste. Improvements in machines for the manufacture of hollow candles. November 28.

23,433. W. R. Herring. Improvements in and in apparatus for manufacturing oil-gas. December 5.

23,950. J. H. R. Dinsmore. Improvements in or connected with apparatus for making gas from coal or analogous material. December 12.

1894.

1231. F. B. Hill and R. D. Brett. Improvements relating to the burning of hydrocarbons for heating and lighting purposes, and to apparatus therefor. November 28.

1238. D. Hancock, J. B. Craig, and A. H. Hancock. Improvements in or relating to the employment of air for lighting and heating purposes. December 19.

1485. T. H. Jones. Improvements relating to the carbonising of wool for the production of charcoal, and in the utilisation of the products given off in the process of carbonisation. November 28.

1490. J. Galletly and H. Armour. Improvements in the manufacture of gas from mineral oils and in the apparatus employed therefor. November 28.

1650. C. C. Walker. Improvements in apparatus to be used in the purification of coal gas or for analogous operations. December 5.

2488. C. J. Yarnold. Improvements in the method and apparatus employed in the production of ozon. December 12.

2702. T. de Zebrowski. A new or improved manufacture of briquettes or artificial fuel blocks. December 12.

1189. C. W. Pinkney. Improvements in and apparatus for the manufacture of gas suitable for use in gas engines or for heating purposes. December 12.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, ETC.

### APPLICATION.

23,373. A. Weil and A. Goutierre. New or improved system of and apparatus for the simultaneous carbonisation and distillation of animal and vegetable matters, and the recovery therefrom of useful products. December 1.

## IV.—COLOURING MATTERS AND DYES.

### APPLICATIONS.

22,154. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of dyestuffs and materials therefor. November 20.

22,533. O. Imray. — From W. J. Matheson and Co., Ltd., United States. Manufacture of colouring matter from logwood. November 21.

22,603. J. Y. Johnson. — From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of new black colouring matter, and the production of black colour on vegetable fibre. November 22.

22,743. L. G. Williams and A. Turner. Improvements in dyes. November 23.

22,904. J. Imray. — From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, France. Manufacture of new blue colouring matters which resist alkaline carbonates. November 26.

22,914. S. Pitt. — From L. Cassella and Co., Germany. The production of fast yellow dyestuffs from the sulpho acids of dehydrothiotoluidine or analogous bases. November 26.

23,435. C. D. Abel. — From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of dichlorotolidine and of colouring matters therefrom. December 3.

23,584. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of colouring matters and materials therefor. December 4.

23,927. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of derivatives of hydroxyanthraquinone. December 8.

24,005. E. B. Aspinall. Improvements in obtaining colouring matters from cotton-seed oil and other like oils. December 11.

## COMPLETE SPECIFICATIONS ACCEPTED.

1894.

1227. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. Improvements in the manufacture of  $\alpha_1$ - $\alpha_2$ -dihydroxynaphthalene- $\alpha$ -monosulpho acid. November 28.

1229. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of dyestuffs by means of trihydroxynaphthalenemonosulpho acid. November 28.

1555. H. Loesner. Manufacture of azoxy compounds of the aromatic series. November 28.

1641. G. W. Johnson. From Kalle and Co. Improvements in the manufacture of new azo-dyestuffs. November 28.

1920. S. Pitt.—From L. Cassella and Co. The production of perichloronaphtholdisulpho acid and disazo dyestuffs therefrom. November 5.

2206. H. Loesner. Improvements in the manufacture of ortho-nitro-toluene, meta- and para-azoxytoluene, meta- and para-toluidin. December 5.

2325. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture of hydroxyanthraquinone quinolines. December 12.

2503. Read, Holliday, and Sons, Lim., and K. B. Elbel. The manufacture of a bluish-red dye belonging to the rosinduline series. December 12.

2591. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture of dyestuffs. December 12.

2592. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The production of azo colouring matters upon fibres. December 12.

2758. J. Altschul. Manufacture of a new diazo compound for use in dyeing and printing. December 12.

3026. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation. Manufacture of new bases containing sulphur of their sulpho acids and of colouring matters derived therefrom. December 12.

3032. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture of azo dyestuffs. December 19.

3036. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik. The manufacture of new mordant-dyeing colouring matters and intermediate products relating thereto. December 19.

3113. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture of colouring matters. December 19.

3301. S. Pitt.—From L. Cassella and Co. Trisazo dyestuffs. December 19.

3397. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik. The manufacture of new nitrosamine compounds suited for use in the preparation of diazo compounds. December 12.

3474. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik. The manufacture of new mordant-dyeing colouring matters. December 5.

3843. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation. Manufacture of colouring matters. December 19.

6198. B. Willcox.—From F. von Heyden, Nachfolger. Improvements in the treatment or purification of commercial toluol-sulpho-amide. December 5.

## V.—TEXTILES, COTTON, WOOL, SILK, Etc.

## APPLICATIONS.

22,366. E. Esser and P. Scheider. Machine for treating textile fabrics. Complete Specification. November 19.

22,416. R. J. Maclean. An improved method or process for producing mixed coloured silk yarn. November 20.

22,595. W. P. Thompson.—From J. F. Kerr, United States. A new or improved method of softening vegetable fibres. Complete Specification. November 22.

22,785. J. B. Fearnley and J. S. Fearnley. Improvements in the waterproofing of textile fabrics and in apparatus therefor. November 24.

22,969. R. J. Rastrick. Improvements in the treatment of rhea and other fibrous plants. November 27.

23,118. P. P. Faure. Improvements in the method of and machinery for decorticating ramie and other textile plants and leaves. November 28.

23,872. W. Warburton. Improved method of and apparatus for rendering textile fabrics or paper waterproof and porous. December 8.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

24,769. J. H. Openshaw. Improvements in the manufacture of cloths known as cotton cords. December 19.

24,904. P. P. Craven. Improved process and machinery for glazing, polishing, and finishing textile fabrics. December 5.

1894.

943. E. B. Manby. See Class XIV.

3554. J. V. Eves. Improvements in the treatment of wet spun flax yarn. December 19.

20,341. G. C. Franklin. Improvements in broad silk pieces, and in the manufacture of the same. December 12.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

## APPLICATIONS.

22,603. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. See Class IV.

23,119. J. Arnold. Improvements in or relating to dyeing. November 28.

26,741. S. H. Sharp. Improved method and machinery or apparatus for producing designs on textile and felted fabrics and the like. Complete Specification. December 6.

## COMPLETE SPECIFICATIONS ACCEPTED.

1894.

1656. J. C. Mewburn.—From La Société Leblais Piceni et Co. Improvements in preparing for bleaching and dyeing, and in bleaching, dyeing, and washing vegetable textile material, and in apparatus therefor. November 28.

18,218. J. Bolet. An improved machine for printing on textile fabrics and the like. November 28.

## VII.—ACIDS, ALKALIS, AND SALTS.

## APPLICATIONS.

22,316. R. Pearson. Improvements in the method of evaporation of brine. November 19.

22,511. G. T. Boilby. The conversion of sulpho-cyanides into ferrocyanides. November 21.

22,569. B. Peitzsch. Improvements in the manufacture of sulphides of the alkalis. November 22.

22,826. J. Brock, A. E. Wareing, and F. Hurter. Improvements in the treatment of solutions of zinc chloride for the purification thereof for electrolytic purposes. November 24.

22,827. J. Brock, A. E. Wareing, and F. Hurter. Improvements in and relating to the treatment of solutions of chloride of zinc for the purification thereof for electrolytic purposes. November 24.

22,828. J. Brock, A. E. Wareing, and F. Hurter. Improvements in the utilisation of zinc blende for obtaining metallic zinc and sulphur, and in effecting the decomposition of common salt for the production of soda and chlorine. November 24.

22,844. I. Levinstein. Improvements in the method of and apparatus for concentrating sulphuric acid. November 26.

22,853. J. W. Walker. Improvements in obtaining a continuous supply of hydrochloric acid or nitric acid gas, and in utilising acid sulphates. November 26.

22,862. W. Garroway. Improvements in the manufacture of chromates and of nitric acid. November 26.

22,917. J. Brock, A. E. Wareing, and F. Hurter. Improvements in the treatment of the residual solutions of the ammonia soda process, and in the recovery therefrom of ammonia and chlorine. November 26.

23,262. C. F. Claus. Improvements in the roasting of sulphide ores. November 30.

23,274. L. P. Hulín. Process and apparatus for the conjoint manufacture of metallic peroxides and caustic alkalis. Filed November 30. Date applied for May 8, 1894, being date of application in France.

23,370. L. P. Hulín. Process and apparatus for the manufacture of caustic alkalis from alloys of the alkali-metals. Filed December 1. Date applied for May 9, 1894, being date of application in France.

24,113. J. Holt and J. G. Lorrain. See Class XVIII. A

24,313. A. G. von Gratz. Improvements in the manufacture or production of aluminates of alkalis. December 14.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

24,563. M. N. D'Audria. Improvements in and connected with the manufacture of magnesium hydrate. December 19.

1894.

2618. A. Crossley. Improvements in apparatus for producing ferro-ferric and ferric oxides. December 12.

2696. A. R. Davis. Improvements in the production of sulphate of manganese and oxide of iron. December 12.

3037. J. Brock, and others. Improvements in and apparatus for the treatment of "caustic salts" obtained as a by-product in the manufacture of caustic soda. December 19.

6621. J. B. Readman. Improvements in obtaining cyanides and ferrocyanides. December 5.

21,778. I. S. McDougall. See Class I.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

## APPLICATIONS.

22,440. H. Gurnard. Improved process of making hollow glass ware. Complete Specification. November 20.

22,834. La Société Anonyme des Matières Manufacturées des Glaces et Produits Chimiques de St. Gobain, Chauxy et C<sup>ie</sup>. The manufacture of a new vitrified material. Complete Specification. Filed November 24. Date applied for June 14, 1894, being date of application in France.

23,708. D. Moore. Improved beer or light for annealing glass. December 6.

24,974. T. Arrow-smith, H. T. Arrow-smith, and J. Arrow-smith. Improved construction of thimble pins for use in supporting pottery-ware whilst being fired. Complete Specification. December 10.

## COMPLETE SPECIFICATIONS ACCEPTED.

1894.

20,964. F. von Hardtmuth. Improvements in presses for the production of tiles and other ceramic ware. December 5.

21,091. F. H. Stubbs. Improved method or methods of decorating tiles or the like. December 12.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

## APPLICATIONS.

22,308. C. G. Picking. Improvements in bricks or blocks to be employed in the construction of the floors of fireproof and other buildings. Complete Specification. November 19.

22,449. M. Thury. Improvements in presses for manufacturing compressed bricks, such as briquettes from granular or pulverulent materials. November 20.

22,633. J. Leuba. An improved paving stone. November 22.

22,793. W. Orr. Improvements in concrete and composite buildings or structures. November 24.

23,123. W. P. Campbell-Everden. An improved method of and means for mixing clays and other materials of a similar nature, more especially suitable for use in the manufacture of cement. November 28.

23,905. J. E. Stephenson. Improvements in the manufacture of bricks or building blocks and the like. December 8.

23,918. T. Potter. Improvements in the construction of fireproof floors and ceilings. December 8.

24,139. J. Bromilow. Utilising slag and coal ashes to produce substitutes for bricks, stone, and concrete. December 12.

## COMPLETE SPECIFICATIONS ACCEPTED.

1894.

1157. H. H. Lake.—From B. Gran. Improvements relating to the manufacture of artificial marble. December 18.

1427. A. McAr. Improvements in the manufacture of Portland cement. November 28.

15,753. J. Elliott. A stone composition for repairing any natural stone and refacing cement floors, &c., also for making imitation stone and bricks of any colour. December 12.



19,437. E. Tortora. Improvements relating to the treatment of terra-cotta, wood, clay, gypsum, limestone, and other materials for preserving and other purposes. December 5.

20,650. L. Küpper. Improvements in walls and wall plates or slabs. December 5.

## X.—METALLURGY, MINING, Etc.

### APPLICATIONS.

19,510A. H. G. Partin. Solder for aluminium. Filed December 6. Date claimed October 13, 1894.

22,415. P. Inch. An improved composition of metals. Complete Specification. November 20.

22,514. A. J. Hobson. An improved metallic alloy for use in the manufacture of pocket cutlery. Complete Specification. November 21.

22,657. G. Hookham. A new metallic alloy for cartridge cases and articles subjected to high temperatures. November 23.

22,740. L. Oliven. Improvements in or relating to the soldering of aluminium and its alloys. November 23.

22,797. W. J. Langham. An improved method of utilising bosh dirt in the manufacture of iron and steel. November 24.

22,903. J. J. Atkinson. An improvement in the manufacture of composite sheet metal. November 26.

23,006. L. C. Daumas. An improved process for the extraction of gold from its ores and apparatus employed therefor. November 27.

23,065. A. J. Maskrey. An improvement relating to the manufacture of tin andterne plates. November 28.

23,385. A. Carter and E. Purser. Improvements in the manufacture of iron from iron sand. December 3.

23,518. W. Fairweather. From G. W. Webber, United States. Improvements in the cyanide process of recovering gold from its ores. December 4.

23,557. J. Pflieger. Improvements in or relating to processes for the extraction of gold and silver from ores. December 4.

23,739. J. Pflieger. Improvements relating to the extraction of gold or silver from their ores. December 6.

23,855. P. H. Bertrand. An improved method of producing magnetic oxide on wrought or cast iron. December 7.

24,092. E. Meyer. Improved process for disintegrating Thomas slag. Complete Specification. December 11.

24,278. C. D. Abel. From Electricitäts-Gesellschaft Geinhausen mit beschränkter Haftung, Germany. Method and apparatus for the production of metals and other fusible materials in pulverulent form. December 13.

24,360. The Cowper-Coles Galvanising Syndicate, Lim., and S. O. Cowper-Coles. Improvements in or connected with the coating of iron or steel articles with nickel, cobalt, silver, platinum, and like metals. December 14.

### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

23,492. E. T. Zohrab. An improved method of hardening or converting into steel the surfaces of iron plates and other objects made from iron. December 12.

24,704. D. W. Sagg. Improvements in the manufacture of alloys. November 28.

24,767. W. Jenks. Improvements in furnaces for annealing iron and steel sheets, and in other furnaces employed in the manufacture or treatment of iron and steel. December 5.

24,939. H. L. Sulman. Improvements in the treatment of gold ores. December 12.

20,247. D. W. Sagg. An improved metallic alloy. November 28.

20,422. W. P. Thompson.—From E. Bertrand and O. Thiel. Improvements in the manufacture of steel, steely iron, or homogeneous metal. December 5.

20,615. J. C. Fell.—From F. A. Gooch and L. Waldo. Improvements in processes for reducing aluminium. December 5.

20,616. J. C. Fell.—From F. A. Gooch and L. Waldo. Improvements in processes for reducing aluminium. December 5.

20,872. P. de Wilde. A method for the extraction of gold. December 5.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### APPLICATIONS.

22,613. J. C. Richardson. Improvements in or relating to the electro-chemical decomposition of solutions of salts. Complete Specification. November 22.

22,965. W. P. Thompson.—From C. L. Coffin, United States. Improvements in or relating to electrically heating metal or other conducting bodies, or welding metals electrically. Complete Specification. November 27.

22,973. C. S. W. Brown. Improvements in and relating to storage batteries. November 27.

23,117. L. P. Hulin. An improved electrolytic process and apparatus for obtaining rich alloys of the metals of the alkalis or alkali earths and the production of halogen elements. Filed November 28. Date applied for May 5, 1894, being date of application in France.

23,160. E. L. Levetus. Improvements in and relating to primary batteries and such like. November 29.

23,198. L. P. Hulin. Process for the treatment of certain metallic alloys for the manufacture of elements for voltaic batteries and caustic alkalis or their derivatives. Filed November 29. Date applied for May 7, 1894, being date of application in France.

23,441. J. C. Fuller and G. Fuller. Improvements in and connected with galvanic batteries. Complete Specification. December 3.

23,486. F. L. Berners and F. Griffin. Improvements in or relating to secondary batteries or accumulators. December 4.

23,553. L. Wagner and J. Marr. Improvements in apparatus for the electrical treatment of beer or other liquid or material. Complete Specification. December 4.

23,590. H. T. Barnett. Improvements in gas batteries. December 5.

23,669. The Cowper-Coles Galvanising Syndicate, Lim., and S. O. Cowper-Coles. Improvements in or connected with the electro-deposition of zinc upon iron. December 5.

23,679. La Société des Cuivres de France. Process for the manufacture of metallic articles by electro-deposition. Filed December 5. Date applied for May 22, 1894, being date of application in France.

23,680. A. M. Clark.—From La Société des Chèvres de France, France. A process of and apparatus for making compressed electrolytic copper, applicable also to other metals capable of being deposited by electrolysis. December 5.

23,688. G. Platner. An improved charging material for galvanic elements. Complete Specification. December 5.

24,297. W. McChleary. An improved porous cell for an electric generator or battery. December 14.

24,364. P. de Wilde. Improved method of treating battery slimes from gold mines. December 14.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1893.

22,674. R. Haddan.—From H. Rignelle. Improvements in or relating to electric regenerative batteries or accumulators and in porous cells therefor. November 28.

23,669. P. Schoop. Improvements in electrolytic vessels for primary and secondary batteries or accumulators, and other processes of electrolytic decomposition and electrolytic action, such as for the extraction of metals from their ores, electro-depositions, and the like. December 12.

1894.

494. W. P. Thompson.—From C. L. Coffin. Improvements in apparatus for heating or working metals electrically. December 5.

835. J. Hargreaves and T. Bird. Improvements in the construction of electrolytic apparatus, and in the application thereof in oxidising, chlorinating, and analogous processes. December 19.

2487. C. J. Hall. Improvements in or relating to secondary batteries. November 28.

13,358. H. H. Lake.—From I. L. Roberts. Improvements relating to electrolysis and to apparatus therefor. November 28.

#### XII.—FATS, OILS, AND SOAP MANUFACTURE.

##### APPLICATIONS.

22,504. S. Banner. See Class II.

23,054. H. Heaton. Improvements in dry soap. Complete Specification. November 28.

23,228. S. Crowder. Improvements in the methods of recovery of oils or fatty substances from clays, sands, ores, mine slimes, or finely-divided substances. November 30.

23,995. C. Weygang and The Petroleum Products Syndicate, Ltd. Manufacture of saponaceous products from petroleum. Complete Specification. December 10.

24,080. D. H. McClelland and R. D. Jackson. Improvements in and connected with oil purifiers. Complete Specification. December 11.

24,095. F. B. Aspinall. See Class IV.

24,103. S. Banner. Improvements in the treatment or oxidation of fixed oils, fats, and the like, and the application thereof as a menstruum for paint, varnish, oil-cloth, and the like. December 11.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1894.

264. F. Burton. A process for the purification of oils, fats, and the like. November 25.

2132. F. Petit. Improvements in the acid saponification of fatty bodies and in apparatus employed therein. December 5.

3497. H. Lambert. Improvements in the method of and apparatus for extracting oil from seeds and the like, and for forming cakes from such or any suitable substances. December 5.

#### XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, ETC.

##### APPLICATIONS.

22,363. A. Gutensohn. An improved process, and means used therein, for the manufacture of oxidised lead. November 19.

22,905. C. E. Bergeon. Improved method of and apparatus for effecting the vulcanisation of caoutchouc articles. November 26.

22,966. W. P. Thompson.—From W. Grisco, jun., United States. Improvements in or relating to the production of compounds applicable for such uses as for paints for electric insulating and waterproofing, as substitutes for vulcanisable rubber and gum compounds, and for other purposes. Complete Specification. November 27.

23,849. J. Holliday and A. Cameron. An improved anti-fouling paint. Complete Specification. December 7.

24,049. J. Moore, J. Sampson, and E. T. Whitelow. Improved substitute for india-rubber, and improvements in the process of producing the same. December 11.

24,103. S. Banner. See Class XII.

24,199. J. A. Fisher. See Class I.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1894.

2428. D. J. Harris. A composition or solution for making waterproof, hardening, and preserving the soles of boots and shoes. December 12.

19,878. A. J. Boulton. From G. Philippe. Impermeable paint for mirrors. December 19.

#### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

##### APPLICATIONS.

22,968. W. R. Earp. Improvements in or relating to the treatment of hides for the manufacture of leather. November 27.

23,585. A. Zimmermann.—From The Chemische Fabrik auf Actien vormals E. Schering, Germany. A process for rendering insoluble albumen and albumen-like matters, casein, albumos, and the products of conversion of glue and gelatine. December 4.

24,404. J. Marshall. Improvements in the method of separating hair, wool, and fur from skin or hide. December 15.

24,442. F. Blenkins and F. G. Bate. Improvements in coloured gelatine sheets, and in apparatus for the production of the same. December 15.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

24,098. H. Neil. Improvements in the treatment of leather. December 12.

1894.

943. E. B. Manby. An improvement in the sizing, dressing, and finishing of textile products, and material for that purpose. December 12.

## XV.—AGRICULTURE AND MANURES, Etc.

## APPLICATIONS.

22,756. T. Twynam and F. T. Barnett. Improvements in the manufacture of fertilisers. November 24.

24,013. A. J. Thoroye. A new composition for improving the soil. December 10.

24,063. W. P. Thompson.—From Wirth and Co., Germany. An improved process for obtaining dry manure and ammonia from faeces or faecal matter, and apparatus therefor. December 11.

24,092. E. Meyer. See Class X.

24,365. A. L. Radford and M. F. Hutton. Improved binding composition for use with fertilising and other substances. December 14.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

24,465. A. R. O. Pieper. Improvements relating to the manufacture of artificial manure. December 19.

1894.

792. M. C. Ginster. Improvements in the manufacture of manure and fertiliser. December 5.

20,971. C. H. Thompson. The manufacture of improved fertilised and fertilising materials for promoting the growth of plants. December 5.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

## APPLICATIONS.

22,568. G. Gin. Improvements in the method of and apparatus for purification of saccharine syrups or liquids. November 21.

22,726. J. Y. Jobuson.—From T. von Heyden, Nachfolger, Germany. A new crystallised sweet compound called erystallöse, and process for manufacturing the same. November 23.

## COMPLETE SPECIFICATION ACCEPTED.

1894.

1790. D. Stewart. Improvements in centrifugal apparatus for drying and purifying sugar or other crystalline or granular matters. December 5.

## XVII.—BREWING, WINES, SPIRITS, Etc.

## APPLICATIONS.

22,504. S. Banner. See Class H.

23,330. W. H. Walker. Improvements in or relating to the treatment or manufacture of extracts of malted grain or the like. December 1.

23,553. L. Wagner and J. Marr. Improvements in apparatus for the electrical treatment of beer or other liquid or material. Complete Specification. December 4.

23,069. R. Cunliffe. Improvements in machines for drying brewers' refuse and other substances or materials. December 5.

23,224. A. Collette. See Class XVIII. A.

## COMPLETE SPECIFICATIONS ACCEPTED.

1893.

23,069. R. Cunliffe. Improvements in machines for drying brewer's refuse and other substances or materials. December 5.

25,052. S. H. Sheldrake. An improved process for the preparation of ale and stout. December 12.

1894.

2966. W. R. Reffell. An external mashing apparatus for the use of brewers and others in mashing malt with water or raw grain liquor, in brewing or distilling. December 19.

3076. D. A. Blair. Improvements in and relating to continuous working spirit stills. December 19.

14,919. L. Prochazka. An improved process and appliances for mashing and brewing in breweries. December 5.

19,662. T. R. Timby. Mechanism for ageing liquors. November 28.

20,796. P. Kropf and A. Gruell. Improvements in and connected with the fermentation of beer. December 19.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

## APPLICATIONS.

## A.—Chemistry of Foods.

22,437. J. H. Payne. A preparation of herbal solutions, sauces, and similar dietetic materials, in tablet or block form. November 20.

23,194. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, France. Improvements in the preservation of alimentary substances. November 29.

23,270. G. A. Clowes. A new or improved medicinal and nutritive compound or preparation. November 30.

23,853. G. Gaertner. Process and appliance for sterilising or pasteurising liquids without any contact with the atmosphere, and for preserving these liquids in sterilised condition and preventing their motion within the vessels in which they are kept. December 7.

24,113. J. Holt and J. G. Lorrain. Improvements in the manufacture of vinegar. December 11.

**B.—Sanitary Chemistry.**

23,415. J. S. Pickering. Improvements in or relating to precipitation tanks for the clarification of sewage. December 3.

23,847. Bacillite Sewage Purification Syndicate, Ltd., and A. P. Hope. Improvements in and apparatus for treating sewage, sewage effluent, sewage sludge, or other like waters or matters. December 7.

**C.—Disinfectants.**

22,491. F. M. Spence. Improvements in disinfecting powders. November 21.

22,725. G. F. Strawson. Improvements in copper fungicide. November 23.

23,101. A. A. Beeheraz. A new or improved composition or preparation for the destruction of insects. November 28.

23,135. C. T. Gardner. An improved sterilising, disinfecting, and deodorising fluid. Complete Specification. November 29.

23,580. C. Stern. Improvements in preservative compounds for fresh meat and other articles of food. December 4.

24,310. E. Andreoli. Producing disinfectants and antiseptics by ozone. Complete Specification. Filed December 14. Date applied for May 16, 1894, being date of application in Belgium.

**COMPLETE SPECIFICATIONS ACCEPTED.****A.—Chemistry of Foods.**

1893.

23,224. A. Collette. Improvements relating to the preservation of yeast and other vegetable and animal substances. December 12.

1894.

1405. F. T. Bond. Improvements in the preparation of cheese. November 28.

2445. C. Kidson. A new method of preserving eggs. December 5.

**B.—Sanitary Chemistry.**

1894.

5819. J. Schmerser and A. Billig. An improved process and apparatus for impregnating air with the essence of fir-wood. December 12.

**C.—Disinfectants.**

1893.

24,531. S. Pitt.—From The Chemische Fabrik auf Actien vormals E. Schering. An improved antiseptic, disinfectant, or deodorant. November 28.

1894.

21,815. A. B. F. Wegemund. Process for the manufacture of a solution for destroying vermin. December 19.

**XIX.—PAPER, PASTEBOARD, ETC.****APPLICATIONS.**

22,566. A. Grandjean. Improvements in or relating to the manufacture of paper or paper pulp. Filed November 21. Date applied for June 1, 1894, being date of application in Belgium.

22,929. F. Wehlers and R. Crosse. An improved manufacture of artificial ivory. November 26.

23,405. H. G. Forbes and E. M. Sommerville. An improved apparatus for producing water marks in paper. December 3.

23,872. W. Warburton. See Class V.

**COMPLETE SPECIFICATIONS ACCEPTED.**

1894.

2923. B. Cawthorn and J. P. Cornet. Improvements in the process of bleaching fibrous materials in the manufacture of paper. December 19.

20,051. W. P. Thompson. From M. C. Stone. Improvements in or relating to the treatment of paper or other tubes. November 28.

**XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.****APPLICATIONS.**

22,617. H. E. Newton. — From the Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of pharmaceutical compounds. November 22.

22,902. F. Ludy. Manufacture of bis-muthoxyiodide-gallate. Complete Specification. November 26.

23,438. R. Jurgensen and A. Kauschlicher. Improvements in the manufacture of acetone. December 3.

24,308. T. Chamings. Germ extract. December 14.

24,146. M. Otto and A. Verley. Improvements in the manufacture of perfumes. December 15.

24,147. M. Otto. Improvements in apparatus for distillation. December 15.

**COMPLETE SPECIFICATIONS ACCEPTED.**

1893.

24,459. S. Pitt.—From The Chemische Fabrik auf Actien vormals Schering. Improvements in the manufacture of levulose. November 28.

1894.

1287. F. W. Warwick. An improved pharmaceutical preparation of ferrous salts. December 5.

**XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.****APPLICATION.**

22,952. A. G. Adamson. Improvements in and connected with the developing of photographs. Complete Specification. November 27.

## XXII.—EXPLOSIVES, MATCHES, Etc.

## APPLICATIONS.

23,260. C. P. Da Bait. Improvements relating to the preparation of smokeless powders for use in firearms and for other purposes. November 30.

24,235. A. F. Hargreaves. Improvements in the manufacture of nitro-cellulose ammunition. December 13.

## COMPLETE SPECIFICATIONS ACCEPTED.

1894.

2144. F. J. Maubeuge. Improvements in combined time and percussion fuses. December 19.

20,281. B. Thieme. An improved process for producing nitropentaerythrite and a smokeless explosive therewith. December 5.

21,331. R. C. Schüpphaus. Improvements in pyroxylin compounds and in the processes of manufacturing the same. December 19.

## XXIII.—ANALYTICAL CHEMISTRY.

## APPLICATION.

22,472. E. Natanson. An improved apparatus for analysing gases. November 20.

3







## ENGINE STORAGE



